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ลิขสิทธิ์ของจุฬาลงกรณ์มหาวิทยาลัย

PRODUCTION OF ALKYL ESTERS AS LUBRICATING BASE OIL
FROM PALM STEARIN



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สถาบันวิทยบริการ
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Department of Chemical Engineering

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งานวิจัยนี้ได้ทำการศึกษาการผลิตแอลคิลเอสเทอร์จากไขปาล์มสเตอรินเพื่อใช้เป็นน้ำมัน
 หล่อลื่นพื้นฐาน โดยการนำไขปาล์มสเตอรินมาผ่านกระบวนการทรานส์เอสเทอริฟิเคชันกับ
 แอลกอฮอล์ที่มีชนิดแตกต่างกัน ดังนี้คือ 1-โพรพานอล ไอโซโพรพานอล 1-บิวทานอล 1-เฮกซานอล
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 ชั่วโมงและมีสัดส่วนโดยโมลระหว่างปาล์มสเตอรินต่อแอลกอฮอล์เท่ากับ 1 ต่อ 6 ซึ่งใช้กรดซัลฟิวริก
 เป็นตัวเร่งปฏิกิริยาภายใต้การปั่นกวนคงที่ และมีอุณหภูมิในการทำปฏิกิริยาอยู่ในช่วงอุณหภูมิ 82 ถึง
 130 องศาเซลเซียส เมื่อปฏิกิริยาเกิดสมบูรณ์จึงนำแอลคิลเอสเทอร์ที่ผลิตได้มาทดสอบคุณสมบัติทาง
 กายภาพและเคมี รวมถึงทำการทดสอบความสามารถในการใช้เป็นสารเพิ่มความหนืดของแอลคิล
 เอสเทอร์ที่ผลิตได้ โดยการนำแอลคิลเอสเทอร์ผสมกับน้ำมันหล่อลื่นพื้นฐานจากปิโตรเลียมชนิด 150
 SN ในปริมาณที่แตกต่างกัน

จากการศึกษาพบว่า ชนิดของแอลกอฮอล์มีผลต่อคุณสมบัติทางกายภาพและเคมีของแอลคิล
 เอสเทอร์ที่ผลิตได้ ทั้งนี้เป็นเพราะแอลกอฮอล์แต่ละชนิดมีขนาด โมเลกุลและ โครงสร้าง โมเลกุล
 แตกต่างกัน โดยเมื่อแอลกอฮอล์มีขนาด โมเลกุลใหญ่ขึ้นหรือมี โครงสร้าง โมเลกุลเป็นแบบ ไซ่ตรง
 ส่งผลให้แอลคิลเอสเทอร์ที่ผลิตได้มีค่าความหนืดสูง จุดวาบไฟสูง และจุดไหลเทต่ำ ซึ่งเป็นคุณสมบัติ
 ที่ดีและเหมาะสมในการนำมาใช้เป็นน้ำมันหล่อลื่นพื้นฐาน นอกจากนี้จากผลการทดสอบยังชี้ว่า
 2-เอทิล-1-เฮกซิลเอสเทอร์มีคุณสมบัติทางกายภาพและทางเคมีใกล้เคียงกับค่ามาตรฐานของน้ำมัน
 หล่อลื่นพื้นฐานชนิดไฮดรอลิก นอกจากนั้นพบว่าแอลคิลเอสเทอร์ที่ผลิตได้จากแอลกอฮอล์ชนิดอื่นๆ
 นั้นมีค่าความหนืดค่อนข้างสูงและเหมาะที่จะนำมาใช้เป็นสารเพิ่มความหนืด ดังนั้นจากคุณสมบัติ
 ดังกล่าว เมื่อนำแอลคิลเอสเทอร์ที่ผลิตได้แต่ละชนิดมาผสมกับน้ำมันหล่อลื่นพื้นฐานชนิด 150 SN
 พบว่าเมื่อเพิ่มสัดส่วนของแอลคิลเอสเทอร์ในปริมาณที่มากขึ้น ส่งผลให้ค่าดัชนีความหนืดของ
 น้ำมันหล่อลื่นพื้นฐานชนิด 150 SN มีค่าเพิ่มสูงขึ้น แสดงให้เห็นว่าแอลคิลเอสเทอร์ที่ผลิตได้แต่ละ
 ชนิดสามารถใช้เป็นสารเพิ่มความหนืดในอุตสาหกรรมการผลิตน้ำมันหล่อลื่นได้

ภาควิชา.....วิศวกรรมเคมี..... ลายมือชื่อนิสิต.....ฉันทุกรานต์ กันยาประสานกิจ
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NATTAKARN KANYAPRASARNKIT: PRODUCTION OF ALKYL ESTERS AS LUBRICATING BASE OIL FROM PALM STEARIN.

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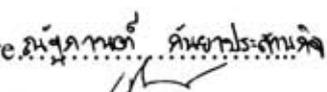
In this study, the production of alkyl esters from palm stearin for use as lubricating base oil via transesterification reaction with various alcohols as 1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol were investigated. The experiments were conducted in a batch system using sulfuric acid as a catalyst at reaction temperature between 82°C to 130°C. The reaction was carried out at a molar ratio of alcohol to palm stearin of 6:1. The reaction time is 3 hours in each experiment under a continuous stirring. After each experiment, alkyl esters were characterized for their physical and chemical properties. Use of alkyl esters as viscosity index improver was investigated by blending alkyl esters with mineral base oil 150 SN different amounts of alkyl ester.

Experimental results show that different types of alcohol affected physical and chemical properties of alkyl esters. Because of different molecular size and molecular structure of each alcohol, the higher molecular size and straight-chain structure provide advantage to alkyl ester properties to have higher kinematic viscosity, higher flash point and lower pour point which are suitable to be used as lubricating base oil. The experimental results also show that 2-ethyl-1-hexyl ester has its properties similar to hydraulic oil. It is also found that viscosity index of each alkyl esters is high enough to be used as viscosity index improver. Blending of mineral base oil 150 SN with alkyl ester shows higher viscosity index of lubricating base oil 150 SN.

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Student's signature 
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CHAPTER I

INTRODUCTION

1.1 Introduction

Lubricant is an important component for mechanical device. It is used as a key to drive every machines, such as bearing, gears, screws, sliding surface, pistons and cams, because it can help the machines to reduce and overcome the friction. Moreover, it also gets rid of power loss and provides a long life of the machines.

In generally, lubricant consists of two main components, additive and base oil. The additive is used for adjustment the base oil's properties (increase viscosity index, decrease pour point, etc) in order to have higher performance. Another important component is base oil that can be divided into three types. The first type is vegetable and animal oils which are not popular because of its low stability and easy to get worse in quality. The second one is petroleum base stock (mineral oil) which has high quality, good stability and low price. Even though there are many great properties, it is still not favor for using because of its disadvantages; For example, the high pollution from the production process and the lack of petroleum oil that use as raw material. The last type is synthetic base stock (synthetic base oil) which is the combination between low molecular compound to produce high molecular compound via chemical reaction. This kind of lubricant is synthesized under controlled conditions (pressure, temperature and the percentage of elements) to get the purified substance and has greater suitable performance than others. For these results, it make synthetic base oil has its own distinctive characteristics that provide to the desired properties. Although, the synthetic base oil has relatively high price, it is always advised to use because it has a positive effect on machine capability, oil life, energy consumption, safety and environmental friendly.

There are many kinds of synthetic base oil that produce from different compounds such as polyalphaolefins, alkylated aromatics, polybutenes, aliphatic diesters, esters, polyolesters, polyalkylenelycols and phosphate esters. From these kinds of synthetic base oil, esters are widely used as the main base oil because of its high viscosity index, low pour point, low volatility and good thermal stability. Moreover, it also can put together with other base oil easily. When it is blended with others base oil, it can increase viscosity index of base oil. To be responsible for environment, the chemistry of esters is being modified to produce compound which have high biodegradability, low toxicity and clean engine emission.

Raw materials that have significant attention for producing the synthetic esters are natural fats and oil of vegetables such as palm stearin. Actually, palm stearin has been defined as the ester of fatty acid with trihydric alcohol glycerol (triglyceride) which has two main components, saturated palmitic acid and mono-unsaturated, oleic acid. Nowadays, palm stearin is widely used in many industries such as shortening, pastry margarines, soap, candles and oleo-chemical industry.

Owing to the higher requirement of lubricating base oil quality is still needed to develop its specific properties such as higher viscosity index, low pour point and lower volatility. So, it is interesting to find the way to improve those lubricating base oil properties. As this reason, this research has been set up to investigate the production of alkyl esters which are used as lubricating base oil from palm stearin via transesterification reaction with various alcohols using sulfuric acid as a catalyst. Because the various types of alcohol will affect the physical and chemical properties of alkyl esters. Therefore, this study concerns about the selection of alcohol types to mix up with palm stearin during the transesterification reaction. Then, the various amounts of produced alkyl esters will be blended with mineral base oil 150 SN (solvent neutral) and characterized using various analytical techniques to investigate the alkyl esters and mixture of alkyl esters with mineral base oil 150 SN properties.

The objectives of this research:

1. To investigate the production of alkyl esters from palm stearin via transesterification with various alcohols by using sulfuric acid as a catalyst.
2. To investigate the physical and chemical properties of the alkyl esters.
3. To prove that the alkyl esters can increase viscosity index of the mineral base oil 150 SN.
4. To get the benefit from palm stearin and make it has more valuable.

This research has been scoped as follow:

1. Produce the alkyl esters from palm stearin via transesterification reaction.

The studies parameters are:

- a. The various types of alcohol such as 1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol
 - b. The various amounts of produced alkyl esters which are blended with mineral base oil 150 SN are 3, 5, 8, 13, 18 and 20 % wt. in order to use as viscosity index improver.
2. Characterization of produced alkyl esters and mixture of alkyl esters with mineral base oil 150 SN are required to determine the physical and chemical properties such as kinematic viscosity, viscosity index, API gravity, pour point, flash point, copper strip corrosion and weld load.

CHAPTER II

THEORY AND LITERATURE REVIEW

2.1 Lubricant

A lubricant is a substance used to reduce the coefficient of friction between the rubbing surfaces in machinery in order to reduce frictional energy losses. The lubricant also prevents direct contact of the rubbing surface since under proper conditions of lubrication film of the lubricant is maintained between these surfaces. This prevents failure due to seizure and also reduces wear. The frictional heat generated by the rubbing surfaces is removed by the lubricant acting as a coolant or heat transfer fluid. In internal combustion engines, the lubricant also seals the piston and cylinder wall at the compression rings so that the high pressure gas in the combustion chamber will not leak pass the rings and cause power losses. Briefly, the lubricant reduces energy losses from friction, reduces wear, serves as a coolant and may also seal.

2.1.1 The basic function of lubricant

The basic functions of lubricant are: (1) friction reduction (2) heat removal and (3) containment of contaminants

2.1.1.1 The reduction of friction

Simple stated friction reduction is accomplished by maintaining a film of lubricant between surfaces which are moving with respect to each other, thereby preventing these surfaces from coming in contact and subsequently causing surface damage.

One of the most important properties of lubricating oil is its viscosity. It forms lubricating films under both thick and thin film conditions. Viscosity affects heat generation in bearing, cylinders and gears related to fluid internal friction. It governs the sealing effect of oils and the rate of oil consumption. It determines that machines may be started under varying temperature conditions, particularly at cold temperature. For many given piece of equipment, satisfactory results are obtained only with the use of an oil of proper viscosity under the operated condition.

The basic concept of viscosity is shown in Figure 2.1, where a plate is being drawn at uniform speed over a film of oil. The oil adheres to both the moving surface and the stationary surface. Oil in contact with the moving surface travels at the same velocity (U) as that on surface, while oil in contact with the stationary surface is at zero velocity.

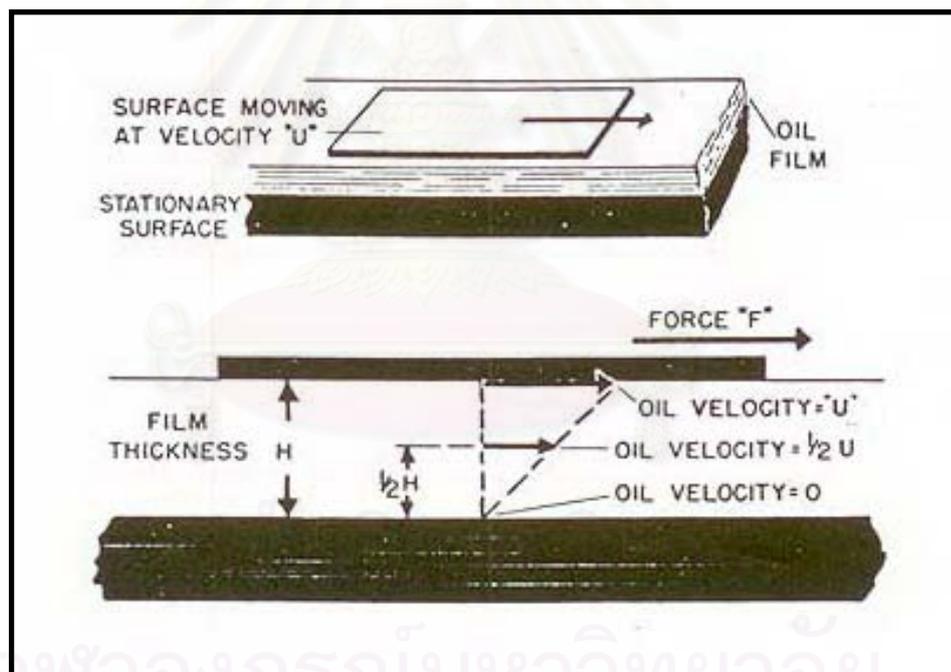


Figure 2.1 Concept of dynamic viscosity (George, W.L., 1980)

In between the oil film may be visualized as made up of many longer, each being drawn by the layer above it is a friction of velocity that is proportional to its distance above the stationary plate. A force (F) must be applied to the moving plate to overcome the friction between the fluid layers. Since the friction is the results of viscosity, the force is proportional to viscosity.

2.1.1.2 Heat Removal

Another important function of a lubricant is to act as a coolant, removing heat generated either by friction or other sources such as via combustion process or transfer by contacting with substances at higher temperatures. In performing this function, it is important that the lubricant remain in a relatively unchanged condition. The variation in thermal and oxidation stability which affect its ability to reach the areas involved will materially decrease its efficiency in this respect.

2.1.1.3 Containment of contaminants

The ability of a lubricant to remain effective in the presence of outside contaminants is quite important. Among these contaminants are water, acidic combustion products, particulate matter, etc., which generally find their way into lubricants employed in various applications. Here again additives are generally the answer in accomplishing these objectives.

2.1.2 The constituent of lubricating oil

Typically lubricants contain 90 % base oil and less than 10% additives. The additive is a substance used to improve the base oil into a better performing lubricant to suit for application. The main families of additive are antioxidants, detergents, anti-wear, anti-foaming agent, friction modifier, viscosity index improver, corrosion inhibitors, rust inhibitors, pour point depressants, etc.

The base oil has several functions but primarily it is the lubricant. Many of the properties of the lubricant are enhanced or created by the addition of special chemical additives to the base oil. The base oil also functions as the carrier for these additives and must therefore be able to keep the additives in solution under all normal working conditions.

The majority of lubricant base oil is produced by the refining of crude oil. Crude oil is the end result of physical and chemical process on the buried remains of plants and animal. Each accumulation or oilfield contains different types of crude oil, varying chemical composition and physical properties. Obviously only some of these crude oil constituents are desirable in lubricant base oil. Other process steps involving chemical reactions may also be used to enhance properties of the oil. Different types of base oil are produced at refineries; oil which have different viscosities or chemical properties are needed for different application. The base oil can be separated into 3 types as follows:

2.1.2.1 Vegetable or animal oil

There is less usage of this type of oil due to its low stability, easy decomposition during use and the fact that it has to pass the quality improvement processes before it can be used, which makes its costs very expensive. Thus, it is currently used as only an additive to improve the lubricity of some lubricating tasks that require specific qualities.

2.1.2.2 Mineral oil

Mineral oil is the most favorable because of its high quality and low price. It is the product from crude oil distillation, in which the heavy fraction at the bottom of the tower will be used as raw material in base oil manufacturing through vacuum distillation process. The type and quality of mineral oil that is separated depends on type of crude oil that is distilled. Some crude oils have to pass other processes to eliminate undesirable things and to obtain good stability.

2.1.2.3 Synthetic oil

This type of base oil is synthesized by chemical processed and is mainly used as base oil that requires specific properties i.e., high viscosity index, low pour point and low volatility. The favorable synthesizes base oils are polyglycol, polyalphaolefins (PAO), esters, halogenated hydrocarbon, etc.

2.2 Synthetic base oil

Synthetic lubricants have been used for many years but they were not commercialized due to the inherent cost of the new synthetic base fluids. In general, the improved properties of lubricant, achieved with the early synthetic base stocks, could be obtained more cost effectively by improved formulations based on mineral oil. However, the requirement for lubricants has simulated the continuing development of synthetic lubricant technology.

A requirement exists for a lubricant; one must select an appropriate lubricant in order to provide trouble-free service over a long period of time. There are number of factors to consider during the selection process, but four considerations that found to be the most predominant are:

2.2.1 Performance requirement or operation environment

This is the most common factor in the selection of a synthetic lubricant over a non-synthetic. There are synthetic-based lubricants which have unique properties that make them the lubricant of choice over non-synthetics, For example, viscosity versus temperature properties, oxidation stability, etc. Other synthetic fluids still exhibit improved stability at elevated temperatures resulting in longer life lubricants requiring less maintenance in equipment using them.

2.2.2 Lubricant availability

The reduced availability of high quality, specialty grade mineral oil base stocks has led to the development of synthetic base stocks with similar chemical composition and behavior as mineral oil for incorporation into lubricants for existing or future applications. The reason for reduced availability of these qualities non-synthetic base fluids have led to the development of a number of synthetic-based lubricants.

2.2.3 Safety

The most often used factor involved in the selection of synthetic lubricants is probably the aspect of enhanced fire resistance, resulting in improved safety. A number of synthetic lubricant development programs have addressed this issue and a variety of fire-resistant and/or non-flammable synthetic-based lubricants/hydraulic fluids have been developed. This is an area of increasing importance.

2.2.4 Cost

This is usually the last often used factor in selecting a synthetic lubricant over a non-synthetic. The exception to this is generally when a high quality, limited volume non-synthetic base fluid is required and either the need for special processing of a high degree of segregation of crude oil results in a very high price for non-synthetic base oil. It is necessary to bring in factors like longer lubricant life, reduced maintenance costs, etc.

2.3 Selection of synthetic base oil

Many compounds have been investigated as possible base stocks for synthetic lubricants. The major types are polyalphaolefins, alkylated aromatics, polybutenes, aliphatic diesters, polyolesters, polyalkyleneglycols and phosphate ester. Other materials such as silicones, borate esters, perfluoroethers and polyphenylene ethers are also of importance, but their applications are restricted due either to high cost or to performance limitation. Some of the primary applications for synthetic lubricants are listed in Table 2.1.

The primary performance advantage of synthetic lubricants is the extended range of service temperatures. Their outstanding flow characteristics at extremely low temperatures and their stability at extremely high temperatures mark the preferred use of these lubricants. The comparisons of some important physical and chemical properties of the most important synthetic lubricating base oil are shown in Table 2.2.

Table 2.1 Primary applications for synthetic lubricants (George, W.L., 1980)

Field of service	Synthetic lubricants
<p><u>Industrial</u></p> <p>Circulating oils</p> <p>Gear lubricants</p> <p>Hydraulic fluids(fire resistant)</p> <p>Compressor oils</p> <p>Gas turbine oils</p> <p>Greases</p> <p><u>Automotive</u></p> <p>Passenger car engine oils</p> <p>Commercial engine oils</p> <p>Gear lubricant</p> <p>Brake fluids</p> <p><u>Aviation</u></p> <p>Gas turbine oils</p> <p>Hydraulic fluids</p> <p>Greases</p>	<p>Polyglycol, SHF, Synthetic ester</p> <p>Polyglycol, SHF</p> <p>Phosphate ester, Polyglycol</p> <p>Polyglycol, Synthetic ester, SHF</p> <p>SHF, Synthetic ester</p> <p>SHF</p> <p>SHF, Synthetic ester</p> <p>SHF, Synthetic ester</p> <p>SHF</p> <p>Polyglycol</p> <p>Synthetic ester</p> <p>Phosphate ester, Silicones, SHF</p> <p>Silicones, Synthetic ester, SHF</p>

SHF: Synthesized Hydrocarbon Fluids such as polyalphaolefins, alkylated aromatics and polybutenes

Table 2.2 Properties of synthetic base fluids compared to mineral oil (Leslie et al., 1999)

Properties	Types of lubricating base oil							
	Mineral oil	Poly i-butenes	Polyalpha olefins	Alkylated aromatics	Polyalkylene glycols	Dicarboxylic acid esters	Polyol esters	Phosphate esters
Viscosity index	F	P	VG	F	VG	VG	VG	G
Pour point	P	F	E	G	G	E	VG	G
Liquid range	F	P	VG	G	G	VG	VG	VG
Oxidation stability	F	F	VG	F	G	VG	VG	G
Thermal stability	F	F	F	F	G	G	VG	VG
Volatility	F	F	VG	G	G	E	E	VG
Flash temperature	P	P	P	P	F	F	F	VG
Hydrolytic stability	E	E	E	E	G	F	F	G
Corrosion protection	E	E	E	E	G	F	F	F
Miscibility with mineral oils	-	E	E	E	P	VG	VG	F
Solubility of additive	E	E	VG	E	F	VG	VG	E
Toxicity	G	E	E	P	G	G	G	F
Biodegradability	F	P	P	P	VG	E	E	VG

*Letter signifies performance level: P = Poor, F = Fair, G = Good, VG = Very Good and E = Excellent

2.4 Synthetic ester lubricating base oil

In the past, the main lubricating base oil were natural ester contained in animal fat or vegetable oil, but due to the reason of low stability and easy to decompose during use. Esters of long chain alcohols and acids were proved to be excellent for low temperature lubricating base oil. The ester is an organic, oxygen-containing material resulting from the reaction of an alcohol and an organic acid. Selected esters for lubricant application have excellent physical properties such as low volatility, low pour point and good low temperature fluidity. They also excel in certain chemical characteristics, such as oxidative and thermal stability. The high solvency of esters also is an advantage. They readily dissolve most additive and help retain deposit precursors in solution. Esters are now uses in many applications including automotive and marine engine oil, compressor oil, hydraulic fluids, gear oil and grease. The inherent biodegradability of ester molecules offers added benefits to those of performance.

The superiority of ester fluids is shown very clearly by comparing the physical properties of an ester with mineral base oil 150 SN as shown in Table 2.3. The ester and the mineral oil have similar viscosities at 100 °C. However, the esters have very low pour point, retains its fluidity to much lower temperature and higher viscosity index than mineral base oil, which is indicative of its excellent viscosity temperature characteristics. The ester also has very low volatility, apparent from high flash point and low percentage distilled at 400 °C.

Table 2.3 Properties of synthetic esters lubricating base oil and mineral base oil (Leslie et al., 1999)

Properties	Synthetic Ester	Mineral base oil 150 SN
Kinematic viscosity, cSt		
at 100 °C	4.6	5.25
at 40 °C	21.0	30.30
at -17.8 °C	474	wax
at -40 °C	5465	wax
Viscosity index	140	104
Pour point, °C	-57	-9
Flash point, °C	243	218
Distillation		
% overhead at 400 °C	3	20

The direct effect of the ester group on the physical property of a lubricant is to lower the volatility and raise the flash point. The presence of the ester group also affects other properties such as:

- Thermal stability
- Hydrolytic stability
- Solvency
- Lubricity
- Biodegradability

The major types of esters with their feedstock and physical property of these esters are reviewed in Figure 2.2 and Table 2.4, respectively.

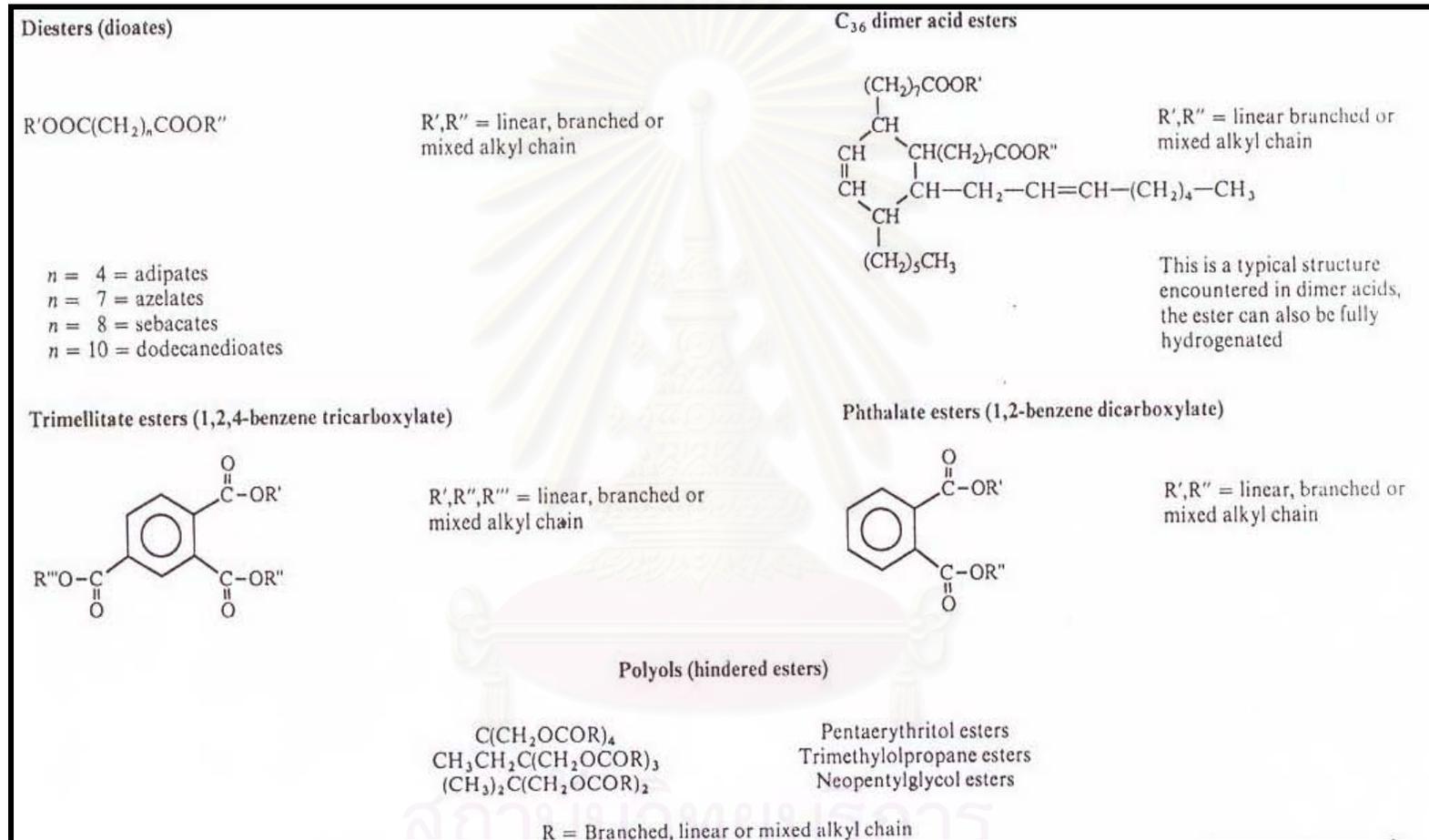


Figure 2.2 The major types of synthetic esters lubricating base oil (Leslie et al., 1999)

Table 2.4 The properties of synthetic esters lubricating base oil (Leslie et al., 1999)

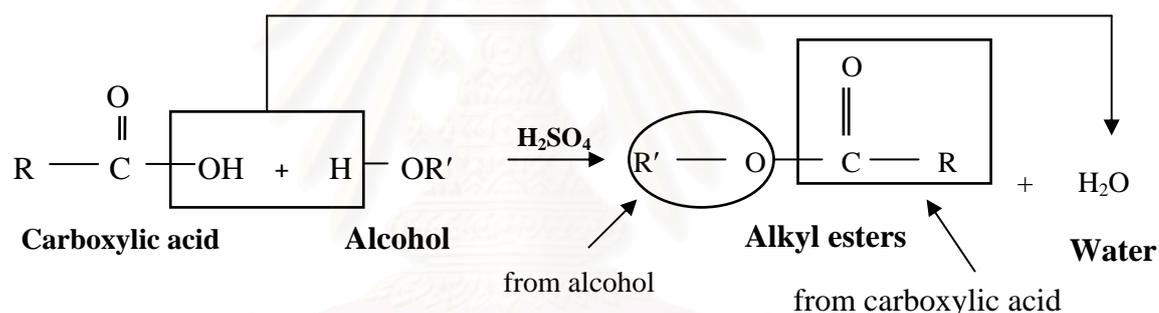
Properties	Diester	Phthalates	Trimellitates	C ₃₆ dimer ester	Polyols	Polyoleates
Kinematic viscosity, cSt						
at 40 °C	6 to 46	29 to 94	47 to 366	13 to 20	14 to 35	8 to 95
at 100 °C	2 to 8	4 to 9	7 to 22	90 to 185	3 to 6	10 to 15
Viscosity index	90 to 170	40 to 90	60 to 120	120 to 150	120 to 130	130 to 180
Pour point, °C	-70 to -40	-50 to -30	-55 to -25	-50 to -15	-60 to -9	-40 to -5
Flash point, °C	200 to 260	200 to 270	270 to 300	240 to 310	250 to 310	220 to 280
Thermal stability	Good	Very good	Very good	Very good	Excellent	Fair
Conradson carbon	0.01 to 0.06	0.01 to 0.03	0.01 to 0.40	0.20 to 0.70	0.01 to 0.10	-
% Biodegradability	75 to 100	46 to 88	0 to 69	18 to 78	90 to 100	80 to 100
Costs (PAO = 1)	0.9 to 2.5	0.5 to 1.0	1.5 to 2.0	1.2 to 2.8	2.0 to 2.5	0.6 to 1.5

2.5 Production of esters

The manufacturing process of esters consists of two processes which are (a) esterification and (b) transesterification.

2.5.1 Esterification

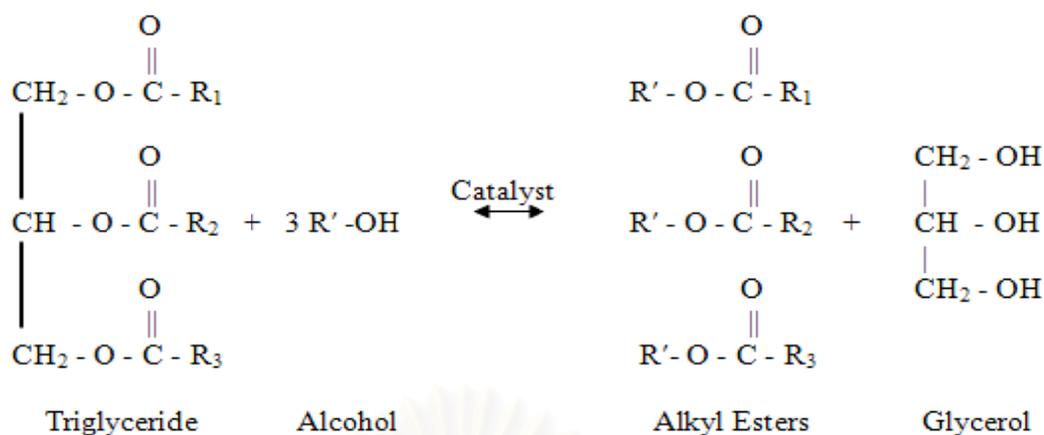
The formations of esters occur through a condensation reaction known as esterification. The esterification requires two reactants, carboxylic acids (fatty acids) and alcohols. Esterification reactions are acid catalyzed and proceed slowly in the absence of strong acids such as sulfuric acid, phosphoric acid, sulfonic acid and hydrochloric acid. The equation of esterification reaction is shown in Equation 2.1.



Equation 2.1 Esterification of carboxylic acid with an alcohol

2.5.2 Transesterification (Alcoholysis)

Transesterification (also called alcoholysis) is the reaction of triglyceride with an alcohol in the presence of catalyst (such as acid, basic and enzyme) to producing a mixture of fatty acids alkyl esters and glycerol. The reaction of transesterification is shown in Equation 2.2.



Equation 2.2 Transesterification of triglyceride with an alcohol (Fukuda et al., 2001)

The overall process is a sequence of three consecutive and reversible reactions, in which di- and mono-glycerides are formed as intermediates as shown in Equation 2.3. The stoichiometric reaction requires 1 mol of a triglyceride and 3 mol of the alcohol. However, an excess alcohol is used to increase the yield of the alkyl esters and to allow its phase separation from the glycerol formed.



Equation 2.3 Transesterification of triglyceride with alcohol: three consecutive and reversible reactions. R', R₁, R₂ and R₃ represent alkyl groups (Fukuda et al., 2001)

From Equation 2.3, the first step is the conversion of triglyceride to diglyceride, followed by the conversion of diglyceride to monoglyceride, and then monoglyceride to glycerol. For each step, one molecule of alkyl esters is liberated (Schwab et al., 1987). Several aspects, including the types of catalyst, alcohol to triglyceride molar ratio, temperature, purity of the reactants (mainly water content) and free fatty acid content have an influence on the cause of the transesterification and will be discussed below, based on the type of catalyst used.

2.5.2.1 Acid-catalyzed transesterification

The transesterification process is catalyzed by Bronsted- Acids, preferably by sulfonic and sulfuric acid. These catalysts give very high yields in alkyl esters, but the reactions are slow, requiring typically temperatures above 100 °C and more than 3 hours to reach complete conversion. However, acid-catalyzed transesterification hold an important advantage with respect to basic-catalyzed ones. The performance of the acid-catalyst is not strongly affected by the presence of FFAs in the feedstock. Thus, a great advantage with acid-catalysts is that can directly produce alkyl esters from low-cost feedstock, generally associated with high FFA concentrations (low-cost feedstock, such as used cooking oil and greases, commonly have FFAs levels of $\geq 6\%$). Freedman et al. (1984) showed that the methanolysis of soybean oil, in the presence of 1 mol% of sulfuric acid, with an alcohol to triglyceride molar ratio of 30:1 at 65 °C, takes 50 hours to reach complete conversion of the vegetable oil (>99%), while the butanolysis (at 117 °C) and ethanolysis (at 78 °C) using the same quantities of catalyst and alcohol, take 3 and 18 hours, respectively.

The alcohol to triglyceride molar ratio is one of the main factors that influence the transesterification. An excess amount of the alcohol favors the formation of the products. On the other hand, an excessive amount of alcohol makes the recovery of the glycerol difficult, so that the ideal alcohol to triglyceride molar ratio has to be established empirically, considering each individual process.

The mechanism of the acid-catalyzed transesterification of triglyceride is shown in Figure 2.2, for a monoglyceride. However, it can be extended to di- and tri-glyceride. The protonation of the carbonyl group of the ester leads to the carbocation II, which after a nucleophilic attack of the alcohol, produces the tetrahedral intermediate III, which eliminates glycerol to form the new ester IV, and to regenerate the catalyst H^+ .

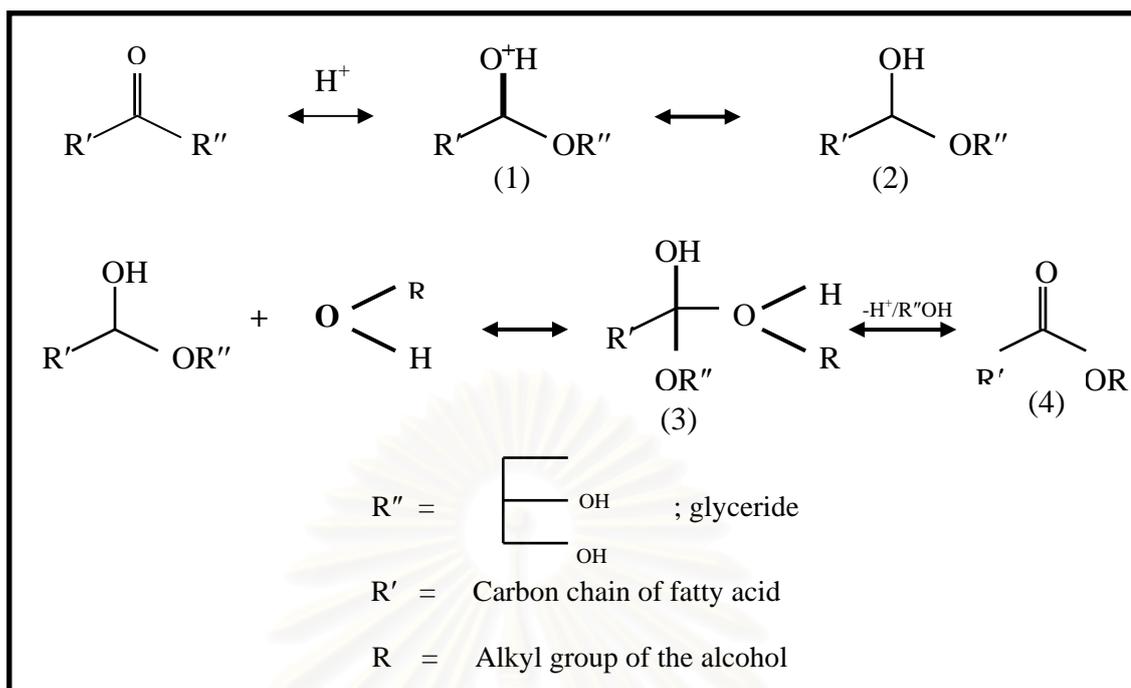


Figure 2.3 The mechanism of the acid-catalyzed transesterification of triglyceride (Goodwin et al., 2005)

2.5.2.2 Basic-catalyzed Transesterification

The basic-catalyzed transesterification of triglyceride proceeds faster than the acid-catalyzed reaction. Due to this reason, together with the fact that the basic-catalysts are less corrosive than acidic compounds, industrial processes usually favor basic-catalysts, such as alkaline metal alkoxides and hydroxides as well as sodium or potassium hydroxide (NaOH or KOH).

The mechanism of basic-catalyzed transesterification of triglyceride is shown in Figure 2.3. The pre-step is the reaction of the base with the alcohol, producing an alkoxide and the protonated catalyst. The nucleophilic attack of the alkoxide at the carbonyl group of the triglyceride generates a tetrahedral intermediate (Step 1), from which the alkyl ester and the corresponding anion of the diglyceride are formed (Step 2). The latter de-protonates the catalyst, thus regenerating the active species (Step 3), which is now able to react with a second molecule of the alcohol, starting another catalytic cycle. Diglycerides and monoglycerides are converted by the same mechanism to a mixture of alkyl ester and glycerol.

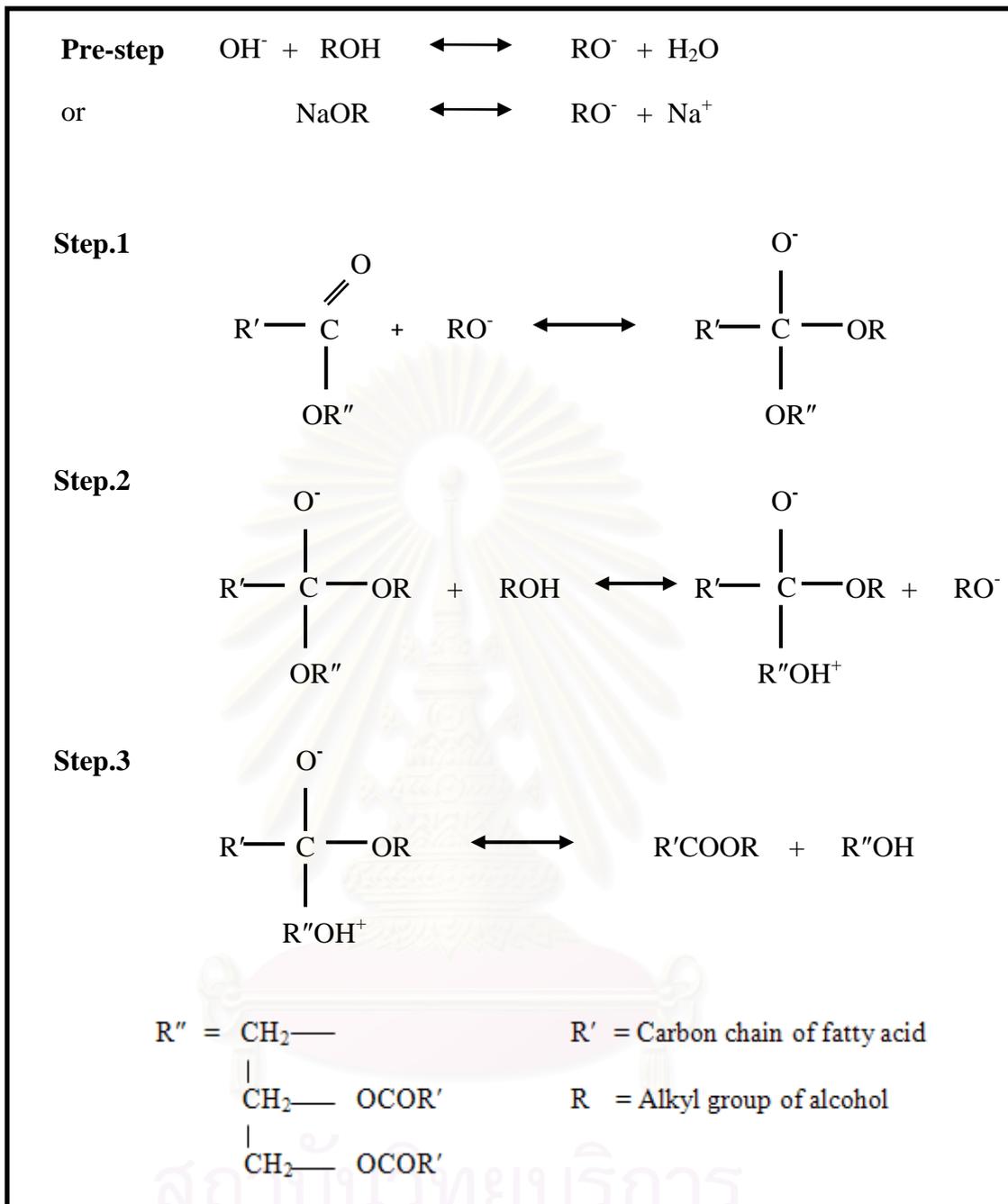


Figure 2.4 The mechanism of the basic-catalyzed transesterification of triglyceride.
(Goodwin et al., 2005)

2.6 Palm stearin

Palm stearin, which has a triglyceride structure, was the more solid fraction obtained by fractionation of palm oil after crystallization at controlled temperatures. At the room temperature (25 °C), palm stearin behaves as a solid state, which contains the more saturated fatty acids and triglycerides. So, it is not used directly for edible purposes due to its high melting point ranging from 44 to 56 °C. The physical characteristics of palm stearin differ significantly from those of palm oil and it is available in a wider range of melting points and iodine values. The components of palm stearin are palmitic acid, oleic acid, stearic acid and linoleic acid. Palm stearin is a very useful source of fully natural hard fat component for products such as bakery margarines, shortening and pastry.

2.7 The principle properties of lubricating base oil

Some of the most commonly used test for physical and chemical properties of lubricating base oil are outlined in the following sections, with brief explanations of the significance of the test from the standard point of the refiner and consumer. For detailed information on the methods of test, the reader is referred to the American Society for Testing and Materials handbooks of “Annual Standards for Petroleum Products and Lubricants”, The British Institute of Petroleum handbook of “Standard Methods for Testing Petroleum and its Products”, The United States Federal Test Method Standard No. 791 and similar types of publications used in a number of other countries.

There are multitudes of physical and chemical properties tests which yield useful information on the characteristics of lubricating base oil. However, the quality of the performance features of lubricating base oil cannot be adequately described on the basis of physical and chemical properties tests alone. Thus, major consumers of lubricating base oil, such as military purchasing agencies and many commercial consumers, include performance tests as well as physical and chemical properties tests in their purchase specifications. Physical and chemical properties tests are of considerable value in maintaining uniformity of products during manufacture. Also,

they may be applied to a used oil to determine changes that have occurred in service and to indicate possible causes for those changes.

2.7.1 Physical properties

2.7.1.1 Specific Gravity, ASTM D1298

Specific gravity is defined as the ratio of the mass of a volume of substance to the mass of the same volume of water. As it ratio there are no units. It is dependent on the temperature of both the substance and water. The standard temperatures for specific gravity are 15.6°/15.6 °C (60°/60°F). In the petroleum industry, the API gravity scale is almost universally used as the measure of the density of a petroleum product. API gravity is an arbitrary scale, calibrated in degrees and related to the specific gravity by

$$\text{API gravity} = \frac{141.5}{\text{Sp. Gr. } 15.6/15.6 \text{ } ^\circ\text{C}} - 131.5$$

The API gravity value increases as the specific gravity decrease. Since both density and gravity change with temperature, determinations are made at a controlled temperature and then corrected to the standard temperature by using special table.

2.7.1.2 Viscosity, ASTM D445

Viscosity is the most significant property of lubricating oil. To meet a particular application, viscosity is generally the most important controlled the properties for manufacture and selection. Viscosity is an index for measuring of internal resistance to the motion of the fluid by reason of the cohesion forces between molecules. It decreases with increasing temperature and increases considerably with large increasing pressure. The extent of the viscosity change depends upon the crude oil source and molecular weight of the constitute components.

Kinematic viscosity is the more common and measured by timing the flow rate of the fixed amount of oil through a capillary tube under gravitational force

at a standard temperature. The test temperatures are usually at 40 and 100 °C. The unit of viscosity used in conjunction with this method is the centistokes (cSt, mm²/s). This unit may be converted to the other viscosity systems (Saybolt, Redwood, Engler) by using a suitable table.

The main objective of lubrication is to provide a film between load-bearing surfaces, the selection of the correct viscosity for the oil is aimed at the balance between a viscosity high enough to prevent the lubricated surfaces from contacting and low enough to minimize energy losses caused by having too viscous a lubricant.

2.7.1.3 Viscosity index, ASTM D2270

The viscosity index (VI) is a method of applying a numerical value to this rate of change, based on comparison with relative rates of change of two arbitrarily selected types of lubricating base oil that differ widely in this characteristic. When the temperature increases, all lubricating oil films “thin out” or have lower viscosity. Likewise, oils become thicker or more viscous as the temperature decreases. Oil having a higher VI is less sensitive to temperature than does the oil with the lower VI. The VI of oil is calculated from viscosities determined at two temperatures by means of tables published by ASTM. The table based on the viscosities which are determined at both 40 °C (100 °F) and 100 °C (212 °F), are available as shown in Appendix E.

The lubricating base oils are made by conventional methods range in VI from somewhat below 0 to slightly above 100. Some synthetic lubricating base oils have VI both below and above this range. Therefore, the additives called Viscosity Index Improvers can be blended into lubricating base oils to adjust VI well above 100. The relationship of kinematic viscosity at 40 and 100 °C are shown in Figure 2.5.

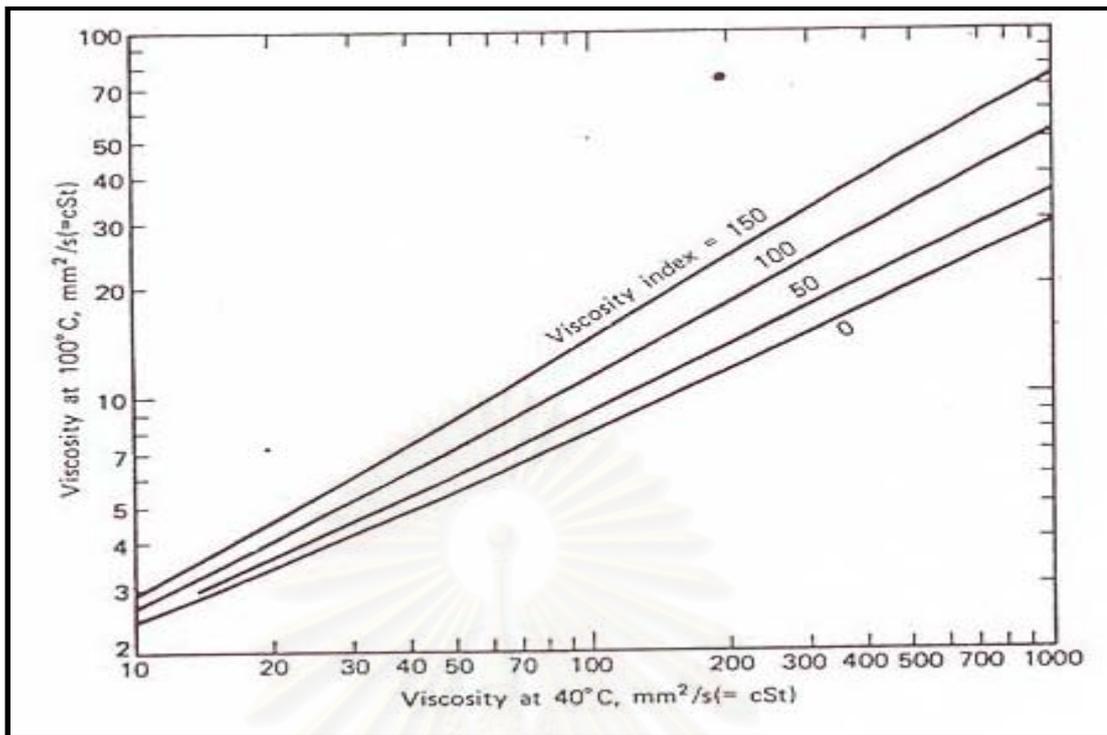


Figure 2.5 The relationship of kinematic viscosity at 40 and 100 °C (George, W.L., 1980)

2.7.1.4 Pour point, ASTM D97

The pour point of lubricating oils is the lowest temperature at which it will pour or flow when it is chilled without disturbance under prescribed conditions. Definition of pour point from ASTM is the temperature at which a measured volume of fluid does not flow for 5 seconds when tilted horizontally. Most oil contains some dissolved wax and, as oil is chilled, this wax begins to separate as crystals that interlock to form a rigid structure which traps the oil in small pockets in the structure. When this wax crystal structure becomes sufficiently complete, the oil will no longer flow under the condition of test. The pour point of the oil will just flow under specified test conditions and is roughly equivalent to the tendency of oil to cease flowing from a gravity-fed system or container. Since the size and shape of the containers, the head of oil, and the physical structure of the solidified oil all influence the tendency of the oil to flow, the pour point of the oil is not an exact measure of the temperature at which flow ceases under the service condition of a specific system.

From the consumer's viewpoint, the importance of the pour point is limited to applications where low temperature is likely to influence oil flow. For example, the pour point of winter grade engine oil must be low enough so that the oil can be dispensed readily, and will flow to the pump suction in the engine at the lowest anticipated ambient temperatures.

2.7.1.5 Flash point, ASTM D92

The flash point of lubricating oil is the temperature at which the lubricating oil releases enough vapor at its surface to ignite when an open flame is applied. For example, if lubricating base oils are heated in an open container; ignitable vapors are released in increasing quantities as the temperature rises. When the concentration of vapors at the surface becomes great enough, exposure to an open flame will result in a brief flash as the vapors ignite. When a test of this type is conducted under certain specified conditions, as in the Cleaveland Open Cup (COC) method, the bulk oil temperature at which this happens is reported as the flash point. The releases of vapors at this temperature are not sufficiently rapid to sustain combustion, so the flame immediately dies out. However, if heating is continued, a temperature will be reached at which vapors are released rapidly enough to support combustion. This temperature is called the fire point. For any specific product, both flash and fire points will vary depending on the apparatus and the heating rate.

Flash and fire tests are of value to refiners for control purposes and significant to consumers under certain circumstances for safety considerations. Also, in certain high temperature applications, use of oils with a low flash point, indicating higher volatility, may result in higher oil consumption rates.

2.7.1.6 Color, ASTM D1500

The color of lubricating oil as observed by light transmitted through them varies from practically clear or transparent to opaque or black. Usually, the various methods of measuring color are based on a visual comparison of the amount of light transmitted through a specified depth of oil with the amount of light transmitted through one of a series of colored glasses. The color is then given as a

number corresponding to the number of the colored glass. In finished, the color of lubricants oil has little significance except in the case of medicinal and industrial white oil, which are often compounded into or applied to products where staining or discoloration would be undesirable.

2.7.1.7 Four-ball test, ASTM 2266

The four ball test is used to test the Wear Preventive (WP) and Extreme Pressure (EP) characteristics of lubricating oils in sliding steel-on-steel applications. The point contact interface is obtained by rotating a 12.7 mm diameter steel ball under load against three stationary steel balls immersed in the lubricant. The normal load, frictional force, and temperature can be monitored using trop. The rotating speed, normal load, and temperature can be adjusted in accordance with published ASTM and IP test standards. For evaluating, the wear-preventive characteristics of lubricants, the subsequent wear scar diameters on the balls can be measured using a graduated-scale microscope. The wear scar can also be captured using an optional digital camera and measured using our Wear Scar Analysis Software. For evaluating, the Extreme-Pressure (load-carrying) capacity of lubricant, the normal load at which welding occurs at the contact interface can be recorded.

2.7.2 Chemical properties

2.7.2.1 Copper Strip Corrosion, ASTM D130

The copper strip corrosion test was measured to study the relative degree of corrosives of a sample according to ASTM D130. A polish copper strip is immersed in a given quantity of sample and heated at a temperature and for a time characteristic of the material being tested. At the end of this period the copper strip is removed, washed and compared with the ASTM Copper Strip Corrosion Standards.

2.8 Literature reviews

Phattanaphakdee (1995) produced synthetic ester lubricating base oil from palm oil. Palm oil was transesterification with an alcohol such as 1-butanol, 1-hexanol, cyclohexanol and 2-ethyl-1-hexanol using sulfuric as a catalyst. The alkyl esters product was then hydrogenated using 3% wt. of platinum supported on alumina as a catalyst. The synthetic ester had physical properties in the range that can be used as lubricating base oil. The synthetic 1-butyl ester was obtained from transesterification with 1-butanol and after hydrogenation was blended with petroleum lubricating base oil 150 SN. The viscosity index of the blended oil was increased from 103.86 to 130.12 when it contained the hydrogenated 1-butyl ester 24% wt. This result indicated that the synthetic esters obtained from the hydrogenation can be used as viscosity index improvers.

Tungwongcharoen (1995) produced synthetic ester lubricating base oil from castor oil via transesterification with 2-ethyl-1-hexanol at 80 °C for 3 hours by using sulfuric acid as a catalyst and then hydrogenation. The catalyst for hydrogenation contained 3% wt. of platinum supported on alumina. The optimum operating condition included 4% catalyst concentration by weight of oil at 100 °C under hydrogen pressure of 100 psi for 3 hours. The monoester product was expected that synthetic ester could be used as lubricating base oil because its high viscosity index, low pour point and good thermal and oxidation stability.

Suwanprasert (1996) produced synthetic ester lubricating base oil from soybean oil via transesterification with an alcohol such as isopropanol, 1-butanol, 1-hexanol, 4-methyl-2-pentanol and 2-ethyl-1-hexanol at the reaction temperature of 80 °C for 3 hours by using sulfuric acid as a catalyst. Then each monoester product was subjected in hydrogenation process under hydrogen gas pressure of 150 psi and reaction temperature of 150 °C for 3 hours by using 1% platinum supported on alumina as a catalyst. The hydrogenated 2-ethyl-1-hexyl ester had tendency to use as synthetic lubricant such as hydraulic lubricant in industries because it has good physical and chemical properties. Furthermore, each monoester was blended with petroleum lubricating base oil 150 SN to give higher viscosity index value than pure

petroleum lubricating base oil. This result showed that it could also be used as viscosity index improver in lubricating industry.

Tubthim (1996) produced synthetic diester lubricating base oil via esterification of palm oil and its free fatty acids such as oleic acid, stearic acid and palmitic acid with 1,3-propanediol, 1,5-pentanediol, 2,2-dimethyl-1,3-propanediol, 1,4-butanediol and 2-ethyl-1,3-hexanediol using sulfuric as a catalyst. The products from these reactions had 2 phases, i.e., a liquid phase and solid phase. Due to inability to determine the physical properties (e.g., kinematic viscosity and pour point) of the solid phase, they were deemed to be unsuitable as automotive lubricants. Each product from transesterification and esterification with 2-ethyl-1,3-hexanediol was liquid phase and had good physical and chemical properties. These results indicate that the synthetic diester obtained from transesterification of palm oil and esterification of its free fatty acid with 2-ethyl-1,3-hexanediol are suitable to used as automotive lubricating base oil.

Vatanaputi (1996) produced synthetic ester lubricating base oil from coconut oil via transesterification with an alcohol such as 1-hexanol, cyclohexanol, 1-octanol, and 2-ethyl-1-hexanol using sulfuric acid as a catalyst and then hydrogenation. The transesterification of coconut oil with cyclohexanol was carried out at 90 °C for 3 hours. The cyclohexyl ester was potential used as a lubricating oil with posses its high viscosity index, low pour point and good thermal and oxidation stability.

Eiamsupasawat (1999) produced synthetic ester lubricating base oil from rice bran oil via transesterification with an alcohol such as 1-butanol, 1-hexanol, 1-octanol, 2-ethyl-1-hexanol and 4-methyl-2-pentanol using sulfuric acid as a catalyst. This reaction was carried out at the boiling point of the mixture, for 2, 3 and 4 hours. The 2-ethyl-1-hexyl ester was the monoester, which had good physical and chemical properties due to high viscosity index, good oxidation stability and a low pour point. With these properties, it could be used as lubricating base oil. Furthermore, when the small amount of 2-ethyl-1-hexyl ester was blended with petroleum lubricating base oil 150 SN, the viscosity index of the blended oil was increases when compared with

petroleum lubricating base oil. This result showed that it could also be used as viscosity index improver in lubricating industry.

Panchoowong (1999) produced synthetic diester lubricating agents were prepared by esterification reaction of fatty acid such as lauric acid, myristic acid, palmitic acid and stearic acid with 1,2-ethanediol and 1,2-propanediol using sulfuric acid as a catalyst. Most of diester products were solid except diester from lauric acid with 1,2-propanediol. The solid products were wax-like nature and had good chemical and physical properties, i.e., high flash point and good thermal and oxidation stabilities. These types of diester could be possible to use in various industrial applications, For example, as emulsifier, antifoaming agent and friction modifier in processing polymer, cosmetic, pharmacy and lubricating technology.

Lee et al. (2000) studied about reason of the reducing of the tendency of alkyl esters to crystallize at low temperatures, branched-chain alcohols were used to esterifies various fats and oil, and the crystallization properties of the branched esters were compared with those of methyl esters using differential scanning calorimeter (DSC), cloud point and pour point. Compared with the methyl esters, branched-chain esters greatly reduced the crystallization of neat esters and their corresponding methyl ester blends.

Lang et al. (2001) produced methyl ester, ethyl ester, isopropyl ester and 1-butyl ester from canola and linseed oil through transesterification using potassium hydroxide and/or sodium alkoxides as catalysts. In addition, methyl ester and ethyl ester were prepared from rapeseed and sunflower oil using the same catalysts. The bio-alkyl esters were characterized for their physical and chemical properties including density, kinematic viscosity, iodine value, acid value, cloud point, pour point, gross heat of combustion and volatility. Methyl ester and ethyl ester prepared from a particular vegetable oil had similar viscosities, cloud point and pour point, whereas methyl ester, ethyl ester, isopropyl ester and 1-butyl ester derived from a particular vegetable oil had similar gross heating values.

Glyglewicz et al. (2003) studied of the possibility of using some nature fats such as rapeseed oil, olive oil and lard as starting material for the preparation of

neopentyl glycol (NPG) and trimethylpropane (TMP) esters is reported. The syntheses of final products were performed by transesterification fatty acid methyl esters, obtained from natural fat with the appropriate polyhydric alcohol using calcium methoxide as a catalyst. The basic physicochemical properties of the NPG and TMP esters synthesized were the following: kinematic viscosity, pour point and viscosity index. In generally, the esters of NPG and TMP were characterized by higher stability in thermo-oxidative conditions in comparison to native triglycerides.

Dörmő et al. (2004) produced bio-lubricant via esterification reaction of fusel oil with oleic acid, where immobilized Novozym 435 lipase enzyme was used as a biocatalyst. The oleochemical ester produced does not have aquatic toxicity and the determined tribological, physical and chemical properties of the lubricant proved that it is an environmental friendly product.

Choo et al. (2005) produced methyl ester and ethyl ester of crude palm oil and crude palm stearin via transesterification reaction and subsequently evaluated for their properties such as kinematic viscosity, density, sulfur content, pour point, cloud point, distillation, gross heat of combustion and flash point.

Hong et al. (2005) produced synthetic lubricating base oil from epoxidized soybean oil via transesterification with Guerbet alcohol such as C12-, C14-, C16- and C18-Guerbet alcohol using sulfuric acid as a catalyst. The alkyl esters were characterized for their physical and chemical properties including pour point, oxidative stability and kinematic viscosity compared with mineral base oil and synthetic oil such as polyalphaolefins, diisododecyl adipate.

Glyglewicz et al. (2005) produced dibasic acid ester with varied chemical structure. These include didecyl carbonate, didecyl adipate and didecyl sebacate as well as modern oligomeric esters of adipic acid and sebacic acid. These esters were tested in term of their suitability as additives of fully synthetic engine oil. It was noted that an addition 10% of the respective esters to oil based on polyalphaolefins led to an improvement of their properties. The pour point of the oil as well as their low temperature viscosity was reduced. The viscosity index and oil lubricity improved. Esters of oligomeric structures synthesized by the transesterification of

dimethyl adipate or dimethyl sebacate with a mixture of neopentyl glycol and decanol showed particularly suitable properties. The tested esters were compatible with the other oil components, forming a stable solution in a wide temperature range.



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CHAPTER III

EXPERIMENTS

This chapter consists of experimental systems and procedures are used in this study. The chapter is divided into four sections. The materials and apparatus for alkyl esters production are explained in section 3.1. The procedures and methodology of transesterification of palm stearin with various alcohols are described in section 3.2. The analytical tests for analyzing alkyl esters quality are shown in section 3.3. Finally, the details of experimental error are illustrated in section 3.4.

3.1 Materials and Apparatus

3.1.1 Raw material

The raw material in this studied was palm stearin. The palm stearin was a part of product from a process of palm oil refinery.

3.1.2 Chemicals

The details of all chemicals in the production procedure and characterization of alkyl esters are shown in Table 3.1. The material safety data sheets of all chemical are shown in Appendix G.

3.1.3 Apparatus for testing properties

The details of all apparatus that used for testing physical and chemical properties of alkyl esters and reactant are shown in Table 3.2.

3.1.4. Experimental apparatus

A reflux set: The reflux set consists of a two-necked 1000 ml round-bottomed flask, an oil bath, hot plate and magnetic stirrer, magnetic bar, temperature controller, thermocouple and condenser.

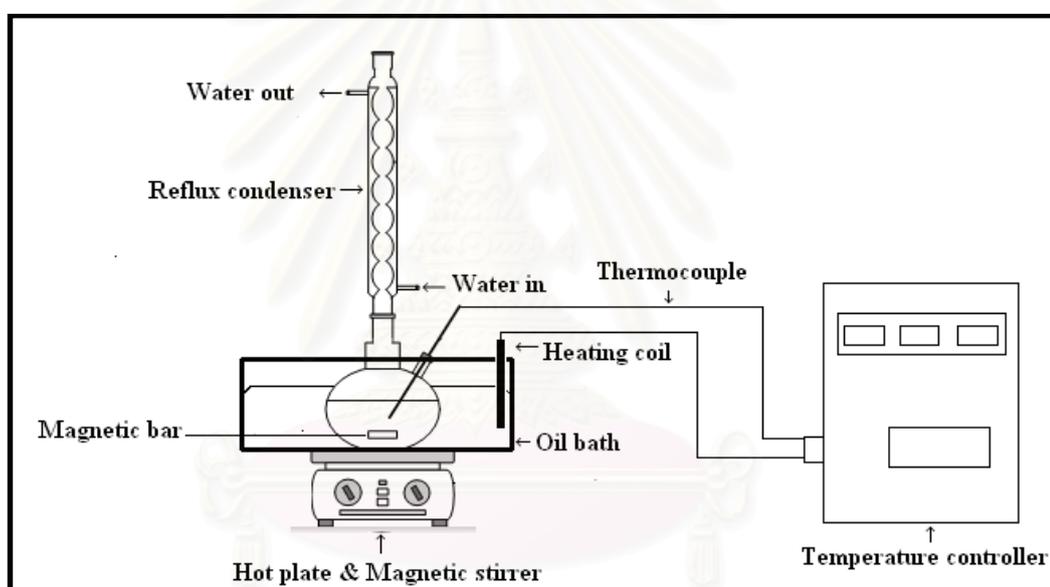
A two-necked 1000 ml round-bottomed flask with magnetic bar equipped with a reflux condenser and thermocouple, respectively. It was immersed into a constant temperature oil bath which put on a hot plate and magnetic stirrer in order to provide agitation. The experimental setup is shown in Figure 3.1.

Table 3.1 The chemicals used in the experiments

Chemical	Purity	Supplier
1-Propanol	99.8%, AR Grade	Ajax Chemicals
Isopropanol	99.8%, AR Grade	Burdick & Jackson
1-Butanol	99.9%, AR Grade	Fisher Scientific
1-Hexanol	98.0%, AR Grade	Ajax Chemicals
1-Octanol	99.0%, AR Grade	Panreac Sintesis
2-Ethyl-1-hexanol	99.0%, AR Grade	Fluka
Sulfuric acid	98.0%, AR Grade	J.T. Baker
Lubricating base oil (150 SN)	Commercial grade	Thai Lube Base Ltd.
Sodium sulfate anhydrous	AR Grade	Ajax Chemicals
Phenolphthalein	AR Grade	Ajax Chemicals
Sodium Hydroxide	99.8%, AR Grade	UNILAB
Ethanol, 95%	95.0%, AR Grade	Fisher Scientific
Potassium hydroxide	98.0%, AR Grade	Ajax Chemicals
Toluene	AR Grade	Ajax Chemicals

Table 3.2 The apparatus used in the properties test of alkyl esters

Apparatus	Model	Manufacturer
Automatic density meter	DMA-5000	Anton Parr
Automatic viscometer	CAV-2000	Cannon
Automatic pour point tester	CPP-5Gs	ISL
Automatic flash point tester	FP93-G2	ISL
Automatic color tester (Tintometer)	PFX 990	Lovibond
Copper corrosion bath	-	Stanhope seta

**Figure 3.1** The experimental apparatus setup for transesterification reaction.

3.2 Experimental procedure

This section of the thesis consists of alkyl esters production step and properties verification. Owing to the main objective of this thesis is alkyl esters production and modification of lubricant base oil quality by using alkyl esters. Therefore, the production step of alkyl esters is much concerned. The diagram of the production procedure of alkyl esters via transesterification reaction is shown in Figure 3.2.

3.2.1 Analysis of palm stearin

The palm stearin was analyzed the kinematic viscosity (ASTM D445), Acid value (AOCS Cd-3D-63) and Saponification value (AOCS Cd-3B-76) in order to check its properties based on density at 60 °C.

3.2.2 Transesterification of palm stearin with various alcohols using sulfuric acid as catalyst.

3.2.2.1 Prepared palm stearin

The refined palm stearin was firstly prepared because it was used as a reactant in this study. The preparing step began with heating it up to 70 °C until it became a clear yellow liquid and separated the unpurified substance by using filter paper (Whatman No.4). Then, it was heated up to 100 °C and was boiled at that temperature with vigorous stirring using a magnetic stirrer for 30 minutes to get rid of the water.

3.2.2.2 Alkyl esters preparation

The alkyl esters that used in each sample were prepared from palm stearin via transesterification with the different types of alcohols. Those are 1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol. In the preparation recipe, the amount between palm stearin and each alcohol were set as 100% excess from its stoichiometric ratio. Moreover, the amount of sulfuric acid that used as a catalyst in this reaction was also set as 3% wt. of palm stearin. So, the amount of various alcohols and sulfuric acid that involved with the production of alkyl esters in each sample were calculated is shown in Appendix B. Also, the reaction time for transesterification reaction is 3 hours. Therefore, the factor affecting the preparation of alkyl esters that are determined in this study are the types of alcohol (1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol).

The details of procedures are as follows: Firstly, 200 grams of the prepared palm stearin was filled into a two-necked glass round bottomed flask (1000 ml). Then, mixing of sulfuric acid (3% by weight of palm stearin) and various alcohols (1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol) in homogeneously phase are put into the two-necked glass round bottomed flask which existed with palm stearin and heated up to desired temperature with a reflux condenser to avoid each alcohols losing while reaction occurred. The reaction temperature depends on the types of alcohol which are carried out at 97, 82, 117, 130, 130 and 130 °C, respectively. The mixture was mixed vigorously using magnetic stirrer with a reflux condenser set. Leave reaction continue until reaching the desired time (3 hours) for transesterification. Then, the samples should be separated by separating funnel at room temperature for 24 hours in order to divide the solutions into two layers (the upper layer is the solution of alkyl esters and the lower is the solutions of glycerol) and separate from each other.

After that, the upper solution (alkyl ester) and lower solution (glycerol) was weighed and boiling to remove the residual alcohols by vacuum distillation. However, after the time passed for 3 hours, the reaction has not completed yet because the conversion of product that occurred at that time still be low. Therefore, the incompletely alkyl ester product was re-transesterification again follow by experiment procedure until reaction was complete.

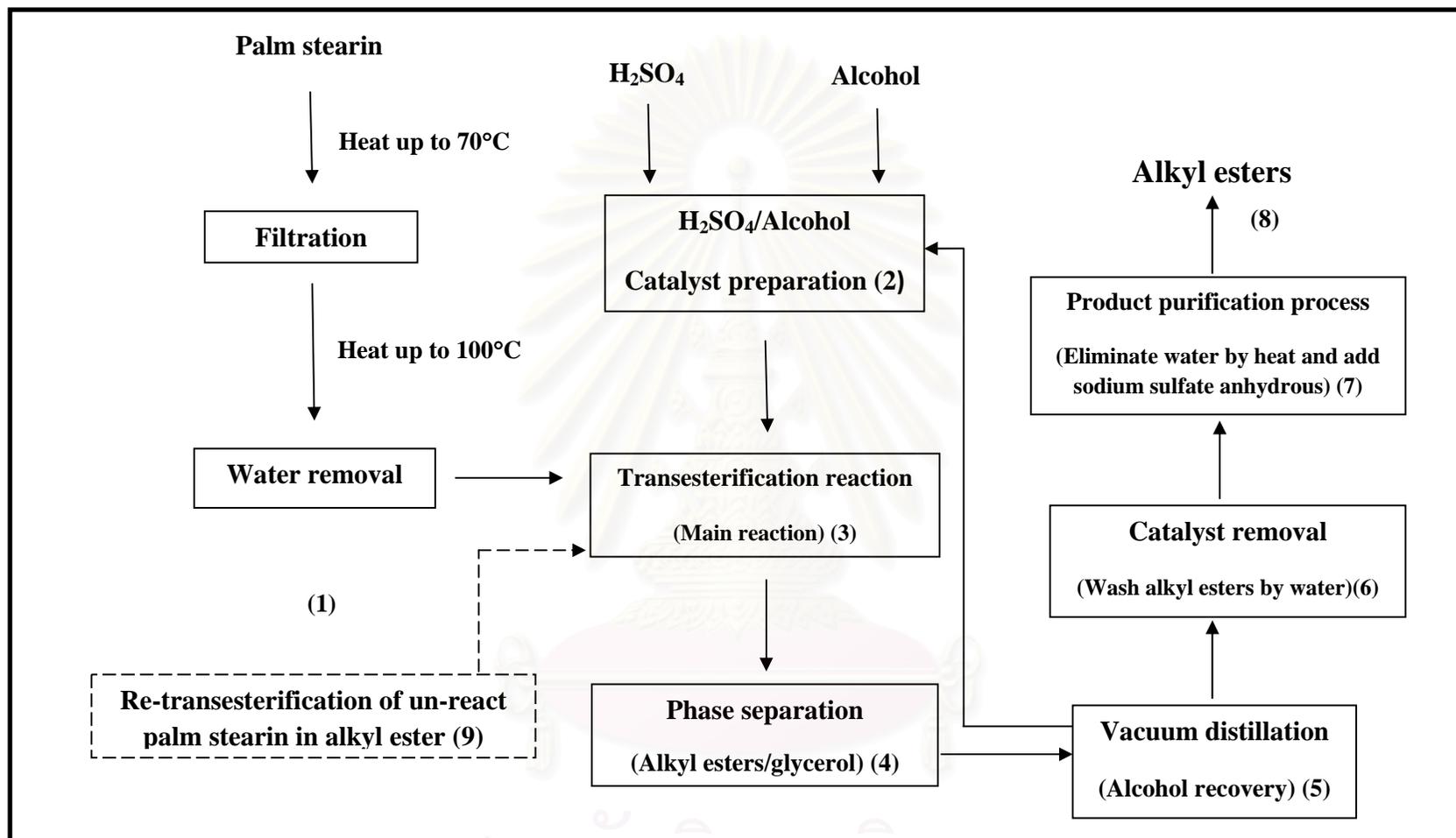


Figure 3.2 The BFD of transesterification of palm stearin via acid catalyst. The diagram concludes the following: feedstock pretreatment (1), catalyst preparation (2), transesterification reaction (3), phase separation (4), alcohol recovery (5), acid catalyst removal (6), alkyl esters purification process (7), alkyl esters product (8) and re-transesterification of un-react palm stearin in alkyl esters (9).

In the next step, the alkyl ester product was washed with warm water until the pH of the washing water increased to 7 to avoid the product from acidity and remove the catalyst. Finally, the water presence in the product is eliminated by sodium sulfate (Na_2SO_4) before analyzing the physical and chemical properties (kinematic viscosity, viscosity index, API Gravity, pour point, flash point, ASTM color, weld load and copper strip corrosion). The reaction time of each alkyl esters is different because the composition of each sample is different. The condition for each experiment was shown in Table 3.3.

Table 3.3 Conditions used for transesterification of palm stearin with various alcohol.

Type of Alcohol	Palm stearin (g)	Amount of alcohol (g)	Amount of sulfuric acid (g)	Temp. (°C)
1-Propanol	200 (0.227 mol)	82.0 (1.367 mol)	6	97
Isopropanol	200 (0.227 mol)	82.0 (1.367 mol)	6	82
1-Butanol	200 (0.227 mol)	101.1 (1.366 mol)	6	117
1-Hexanol	200 (0.227 mol)	139.2 (1.365 mol)	6	130
1-Octanol	200 (0.227 mol)	177.6 (1.366 mol)	6	130
2-Ethyl-1-hexanol	200 (0.227 mol)	177.6 (1.366 mol)	6	130

3.3 The physical and chemical properties characterization

3.3.1 Alkyl esters characterization

After transesterification, the product (alkyl esters) was analyzed the physical and chemical properties as followed:

- | | | |
|---|----|-------------|
| 1. Kinematic viscosity at 40 and 100 °C | by | ASTM D 445 |
| 2. Viscosity Index | by | ASTM D 2270 |
| 3. API Gravity | by | ASTM D1298 |
| 4. Pour Point | by | ASTM D 97 |
| 5. Flash Point | by | ASTM D 92 |
| 6. Color | by | ASTM D 1500 |
| 7. Copper strip corrosion | by | ASTM D 130 |
| 8. Four-ball test | by | ASTM D 2266 |

All physical and chemical properties can indicate the alkyl esters qualification that produces from palm stearin with various alcohols.

3.3.2 Verify of the alkyl esters by blending with mineral base oil 150 SN

The alkyl esters which produced from palm stearin with various alcohols were checked by blending with mineral base oil 150 SN in order to verify its can be use as viscosity index improver to improve properties and quality of mineral base oil 150 SN. The certificate of mineral base oil 150 SN is shown in Appendix F.

The alkyl esters which received from procedure 3.2 were weighed in different amount of alkyl esters (3, 5, 8, 13, 18 and 20% of the total mixture). For example, if the sample is 3% alkyl ester of the total mixture, the alkyl esters and the lubricating base oil 150 SN should be weighed 3 g and 97 g, respectively. The alkyl esters were

mixed together with base oil 150 SN by magnetic stirrer until homogeneously. After that, each sample was analyzed in the following properties.

- | | | |
|---|----|-------------|
| 1. Kinematic viscosity at 40 and 100 °C | by | ASTM D 445 |
| 2. Viscosity Index | by | ASTM D 2270 |
| 3. Pour Point | by | ASTM D 97 |
| 4. Color | by | ASTM D 1500 |

3.4 Experimental and Analytical error

3.4.1 Experimental error

In this section, the experiments are conducted to verify repeatability, an average, and a standard deviation value of the experiment. Equation (3.5) and Equation (3.6) defined an average value and percent deviation respectively.

$$\text{Average value, } \bar{X} = \frac{\sum x}{n} \quad (3.5)$$

$$\text{Percent deviation from average value} = \sqrt{\frac{\sum (X - \bar{X})^2}{n-1}} \times 100 \quad (3.6)$$

Some experiments are repeated to study the error that might occur during each experiment of alkyl esters production. The experiment is repeated for 3 times at the same condition to verify the result of experiment. The experiment error of production of alkyl esters was shown in Appendix C.

CHAPTER IV

RESULTS AND DISCUSSIONS

This chapter presents experimental results and discussions of production of alkyl esters from palm stearin via transesterification reaction with various alcohols using sulfuric acid as a catalyst. The alcohols used in this study included 1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol. The studied conditions are summarized in Table 3.3. The experimental procedure is described in section 3.2. The physical and chemical properties of alkyl esters, i.e., kinematic viscosity, viscosity index, flash point, pour point, ASTM color, API Gravity, copper strip corrosion and weld load were analyzed. The results and discussion are divided into four parts as followed:

- 4.1 Characterization of palm stearin
- 4.2 Preliminary experimental results
- 4.3 Effects of molecular size and molecular structure of alcohol on the physical and chemical properties of alkyl esters
- 4.4 Effect of alkyl ester quantity on the physical properties of the blended mineral base oil 150 SN

4.1 Characterization of palm stearin

Palm stearin used in this study was obtained from the palm oil refinery industries. It is a co-product of palm olein which is produced by fractionation process of palm oil. Because of its low price and abundance, palm stearin is competitive to be used as raw material in this study. The appearance of palm stearin is white solid at room temperature and becomes liquid phase at higher temperature. The physical and chemical properties of palm stearin are shown in Table 4.1.

Table 4.1 The properties of palm stearin

Identity characteristic	Method	Palm stearin
ASTM Color	ASTM D1500	L0.5
Density at 60 °C, g/cm ³	ASTM D1298	0.889
Kinematic viscosity at 100 °C, cSt	ASTM D445	9.01
Melting point, °C	ASTM D127	51.5
Pour point, °C	ASTM D97	35.5
Flash point, °C	ASTM D92	338
Acid value, mg KOH/g	AOCS Cd-3D-63	0.13
Saponification Value, mg KOH/g	AOCS Cd-3B-76	191.31
% Free fatty acid (as palmitic acid)	AOCS Cd-3D-63	0.06
Mean molecular weight, g/mole	-	879.89

*Kinematic viscosity at 40°C cannot determine because palm stearin is a solid state at 40 °C

The measured melting point of palm stearin is about 51.5 °C according to ASTM D127 method. Palm stearin contains relatively low acid value which implies low percentage of free fatty acid. The calculated mean molecular weight of palm stearin is approximately 879.89 g/mole. The molecular weight is calculated from its acid value and saponification value is shown in Appendix A.

The chemical structure of palm stearin is shown in Equation 2.2 where R₁, R₂ and R₃ represented fatty acids. The fatty acid compositions are determined via transesterification of palm stearin with methanol. The product of this reaction is fatty acid methyl esters (FAME) which is analyzed by Gas Chromatography (GC) and converts to equivalent amount of fatty acid. The result in Table 4.2 shows the fatty acid composition of palm stearin. It shows that palm stearin consists of 65% saturated fat and 35% unsaturated fat. The major fatty acid component is saturated fatty acids which are palmitic acid and stearic acid. The minor fatty acid component is unsaturated fatty acids which are oleic acid (mono-unsaturated) and linoleic acid

(poly-unsaturated). These results indicated that palm stearin is suitable to use as reactant because it contains low unsaturated fatty acids which are easier to oxidize than saturated fatty acids.

Table 4.2 Fatty acid composition of palm stearin

Fatty Acids	Formula	% Composition
Palmitic acid	$C_{16}H_{32}O_2$	59.38
Stearic acid	$C_{18}H_{34}O_2$	7.5
Oleic acid included Linoleic acid	$C_{18}H_{32}O_2$	28.13
Others	-	5.34

4.2 Preliminary experimental results

In recent year, there are many researchers have studied about the production of alkyl esters from the transesterification of palm stearin with methanol and ethanol to produce methyl ester and ethyl ester, respectively, but the research about the transesterification of palm stearin with other alcohols have rarely to be studied. In order to verify our experimental results, a set of preliminary experiment is conducted to compare the results of this study with the previous work.

In this preliminary experiment, the transesterification of palm stearin with methanol and ethanol are carried out with 100% excess alcohol from its stoichiometric ratio. The catalyst used in both reactions is 3 % wt. of sulfuric acid based on the weight of palm stearin. The reaction temperature of transesterification of palm stearin with methanol and ethanol is 65 and 78 °C, which is the boiling point of methanol and ethanol, respectively. The conditions of each experiment are summarized in Table 4.3.

Table 4.3 Condition of preliminary experimental

Condition	Methyl ester	Ethyl ester
Palm stearin (g)	200 (0.227 mol)	200 (0.227 mol)
Types of alcohol	Methanol	Ethanol
Amount of alcohol (g)	44 (1.375 mol)	63 (1.370 mol)
Amount of sulfuric acid (g)	6	6
Reaction temperature (°C)	65	78

The transesterification of palm stearin with methanol and ethanol are conducted in a 1000 ml two-necked round-bottomed flask which equipped with a thermometer and a reflux condenser. The round-bottomed flask was immersed into an oil-bath which is controlled the temperature. The mixture of reactant was mixed vigorously using magnetic stirrer as shown in Figure 3.1. The reaction was carried out until reaching the desired time (3 hours). After the time passed for 3 hours, the reaction has not completed because the conversion of product that occurred at that time still be low which can be observed by quantity of glycerol forming. Therefore, the incomplete alkyl ester product should be re-transesterification again by following the experiment procedure until reaction completed which can observe by no longer glycerol forming. In each step, the samples should be separated by separating funnel at room temperature for 24 hours in order to divide the solutions into two layers, i.e., mixture of alkyl ester with un-reacted palm stearin (upper layer) and glycerol (lower layer). After that, both of the upper solution and lower solution were weighed and boiled to remove the residual alcohols by vacuum distillation. After the reaction completed, the total product of alkyl ester and glycerol of methyl ester and ethyl ester are weighed. After that, the methyl ester and ethyl ester were analyzed physical and chemical properties, i.e., kinematic viscosity, specific gravity, density, pour point, flash point, ASTM color and copper strip corrosion are shown in Table 4.5.

Table 4.4 Product of transesterification of palm stearin with methanol and ethanol

Sample	Step	Reactant				Product	
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)	Alkyl ester (g)	Glycerol (g)
Methyl ester	1	200	-	43.9	6	201	12.8
	2	-	197	44.1	6	195	5.6
	3	-	193	44.0	6	191	1.2
	Total	-	-	-	-	191	18.6
Ethyl ester	1	200	-	62.9	6	209	10.9
	2	-	207	63.0	6	204	4.3
	3	-	203	63.0	6	200	2.9
	4	-	200	62.8	6	197	1.1
	Total	-	-	-	-	197	19.2

Note: The molecular weight of methyl and ethyl esters is 294.63 and 308.63 g/mole, respectively.

The results of transesterification of palm stearin with methanol and ethanol are shown in Table 4.4. It shows that the step of transesterification of palm stearin with methanol and ethanol are 3 and 4 times (9 and 12 hours), respectively because the methanol can react easier than ethanol which has a larger molecular size than methanol. The yield of alkyl esters are defined as a ratio of mole of alkyl esters to mole of palm stearin as shown in Equation 4.1. The yield of methyl ester and ethyl ester are 95.17% and 94.01%, respectively.

$$\% \text{ Yield of alkyl ester} = \frac{\text{Weight of alkylester (g)}}{3 \times \text{Molecular weight of alkylester (g/mole)}} \times 100 \quad (4.1)$$

$$\% \text{ Yield of alkyl ester} = \frac{\text{Weight of palm stearin (g)}}{\text{Molecular weight of palm stearin (g/mole)}} \times 100$$

Table 4.5 The physical and chemical properties of methyl ester and ethyl ester

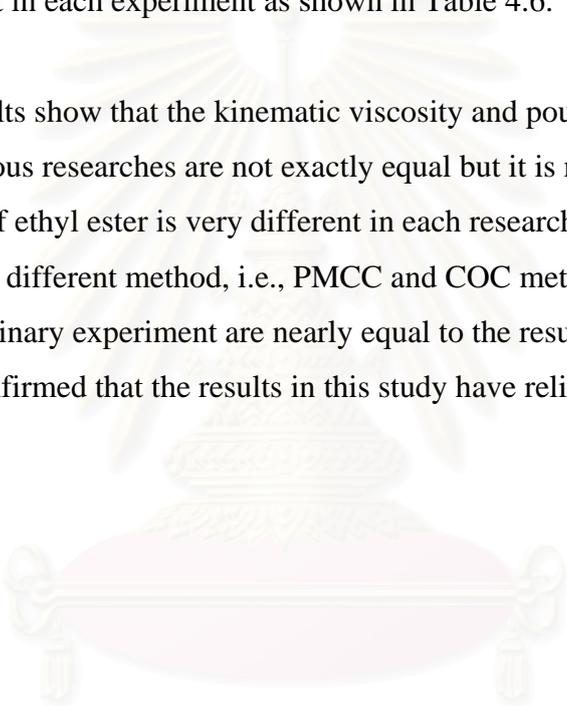
Properties	Methyl ester	Ethyl ester
Specific Gravity at 60/60 °F	0.872	0.873
Density at 20 °C, g/cm ³	0.868	0.869
Kinematic Viscosity at 40 °C, cSt	4.55	4.81
Kinematic Viscosity at 100 °C, cSt	1.796	1.949
Viscosity index	Not possible	Not possible
Pour point, °C	16	13
Flash point (PMCC), °C	165.2	173.2
ASTM Color	L1.0	L1.0
Appearance	Bright & Clear	Bright & Clear
Copper Strip Corrosion at 100 °C for 3 hrs.	1a	1a

The results in Table 4.5 show that the specific gravity at 60 °F and density at 20 °C of methyl ester are slightly less than ethyl ester. The kinematic viscosity of methyl ester and ethyl ester is 4.55 and 4.81 cSt at 40 °C and 1.790 and 1.949 cSt at 100 °C, respectively. The kinematic viscosity of ethyl ester is higher than methyl ester because the methyl ester has smaller molecular size than ethyl ester. However, the viscosity index of both alkyl esters is not able to calculate because the kinematic viscosity at 100 °C of both alkyl esters is still lower than 2.0 cSt. The pour point of ethyl ester is lower than methyl ester which it can be indicated that the ethyl ester has better cold-temperature properties, i.e., pour point and cloud point than methyl ester. The flash point of methyl ester, 165.2 °C, is lower than ethyl ester, 173.2 °C, according to ASTM D-93 (Pensky-Martens Closed Cup Method, PMCC). Other properties of both alkyl esters, i.e., ASTM color and copper strip corrosion are not

different. All results indicated that the types of alcohol has affected on the physical and chemical properties of alkyl esters.

The purpose of preliminary experiment is to ensure the reliability of this experimental result by comparing with previous research. Udomsap (2005) and Chonsaranon et al. (2006) studied the production of alkyl esters from palm stearin with methanol and ethanol, respectively, but Choo et al. (2005) produced methyl ester and ethyl ester from transesterification of palm stearin. They found that the physical properties of alkyl ester, i.e., kinematic viscosity, pour point and flash point are slightly different in each experiment as shown in Table 4.6.

The results show that the kinematic viscosity and pour point of both alkyl esters from various researches are not exactly equal but it is not significant. However, the flash point of ethyl ester is very different in each research because it was measured by the different method, i.e., PMCC and COC method. In conclusion, all results of preliminary experiment are nearly equal to the results of other researches. So, it can be confirmed that the results in this study have reliability.



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Table 4.6 The comparison between this preliminary experimental results and previous work.

Researchers	Year	Raw material	Catalyst	Methyl ester			Ethyl ester		
				Kinematic viscosity (cSt)	Pour point (°C)	Flash point (°C)	Kinematic viscosity (cSt)	Pour point (°C)	Flash point (°C)
Choo et al.	2005	Palm stearin	NaOH	4.5	18	165 (PMCC)	4.8	15	-
Udomsap	2006	Palm stearin	KOH	-	-	-	4.82	-	152 (COC)
Chonsaranon	2005	Palm stearin	NaOH	5.07	12	170 (PMCC)	-	-	-
Preliminary experiment	2007	Palm stearin	H ₂ SO ₄	4.55	16	165.2 (PMCC)	4.81	13	173.2 (PMCC)

4.3 Effect of types of alcohol on the physical and chemical properties of alkyl esters

In this section discussed about the production of alkyl esters which produced from transesterification of palm stearin with various alcohols (1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol) for using as lubricating base oil. The products of transesterification reaction are 1-propyl ester, isopropyl ester, 1-butyl ester, 1-hexyl ester, 1-octyl ester and 2-ethyl-1-hexyl ester, respectively. The reactions are carried out with 100% excess alcohol from its stoichiometric ratio. The catalyst used in the reactions is 3% wt. sulfuric acid based on the weight of palm stearin. The reaction temperature for each sample depends on the types and the boiling point of alcohol but should not over than 130 °C. If the temperature is higher than 130 °C, the color of the samples will become darker than the reaction at lower temperature. So, the reaction temperature in the production of 1-propyl ester, isopropyl ester, 1-butyl ester, 1-hexyl ester, 1-octyl ester and 2-ethyl-1-hexyl ester are 97, 82, 117, 130, 130 and 130 °C, respectively. The condition of each sample is shown in Table 3.3. The retention time is 3 hours per step until the reaction completes which can observe by no longer glycerol forming. The details of this procedure were explained in section 4.2. After the reaction completed, the total product of alkyl esters and glycerol of alkyl esters are weighed.

The amount of product in each step of transesterification from palm stearin with various alcohols is shown in Appendix C. The results indicated that the retention time of the production of 1-propyl ester, isopropyl ester, 1-butyl ester, 1-hexyl ester, 1-octyl ester and 2-ethyl-1-hexyl ester are 4, 4, 4, 5, 6 and 6 times (12, 12, 12, 15, 18 and 18 hours), respectively. The time period that required in each sample is different because the molecular size of each alcohol is different. The alcohol which has smaller molecular size can react easier than the larger molecular size. Moreover, the reaction temperature of 1-hexanol, 1-octanol and 2-ethyl-1-hexanol is lower than their boiling point while 1-propanol, isopropanol, 1-butanol can operate at their boiling point. Therefore, the difference time period that is required in each sample may cause from the different types of alcohol and reaction temperature. The yield of 1-propyl ester, isopropyl ester, 1-butyl ester, 1-hexyl ester, 1-octyl ester and 2-ethyl-1-hexyl

ester which calculated by Equation 4.1 are 93.18, 93.79, 92.94, 93.58, 92.38 and 92.88 %, respectively. After that, the alkyl esters were analyzed physical and chemical properties, i.e., kinematic viscosity, API Gravity, pour point, flash point, ASTM color, weld load and copper strip corrosion are shown in Table 4.7.

The results show that the physical and chemical properties of each alkyl ester which were produced from the different types of alcohol. Owing to each alcohol have its own unique properties such as molecular structure and molecular size, the properties of each alkyl ester that shown in Table 4.7 are not the same value. So, in this section, the discussions about the effect of alcohol types on the alkyl ester properties such as ASTM color, kinematic viscosity, viscosity index, pour point, flash point and API Gravity are referenced from molecular structure and molecular size of alcohols. The molecular structure of alcohol in this experiment is divided into two groups, i.e., straight-chain alcohol and branched-chain alcohol. The methanol, ethanol, 1-propanol, 1-butanol, 1-hexanol and 1-octanol are the straight-chain alcohol while as the isopropanol and 2-ethyl-1-hexanol is the branched-chain alcohol. The molecular size of straight-chain alcohol which is arranged in order from small to large size is methanol, ethanol, 1-propanol, 1-butanol, 1-hexanol and 1-octanol, respectively. The comparison of the molecular size of each alcohol is shown in Figure 4.1. Moreover, there are two couples of alcohols which have the same molecular size but different molecular structure. The first couple is 1-propanol and isopropanol and another couple is 1-octanol and 2-ethyl-1-hexanol. The isopropanol and 2-ethyl-1-hexanol alcohols have higher degree of branched-chain than 1-propanol and 1-octanol, respectively. The discussion of the effect of molecular size and molecular structure of alcohol on physical and chemical properties of the alkyl ester as followed:

Table 4.7 The physical and chemical properties of alkyl esters

Properties	Mineral Base Oil 150 SN	The various types of alkyl esters					
		1-Propyl ester	Isopropyl ester	1-Butyl ester	1-Hexyl ester	1-Octyl ester	2-Ethyl-1-hexyl ester
Color, ASTM	L0.5	L1.5	L1.5	L3.0	L3.0	L4.0	L4.0
Kinematic Viscosity at 40 °C, cSt	30.30	5.513	6.414	6.509	7.842	8.148	8.667
Kinematic Viscosity at 100 °C, cSt	5.25	2.038	2.188	2.324	2.616	2.713	2.782
Viscosity index	104	208	169	212	195	202	188
Pour point, °C	-12.0	+11.0	+6.0	+9.0	+8.0	+5.0	-5.0
Flash point (COC), °C	234	196.0	193.0	202	210	240	236
API Gravity at 60/60°F	-	31.39	31.34	31.23	31.19	31.14	31.11
Appearance	Bright&Clear	Bright&Clear	Bright&Clear	Bright&Clear	Bright&Clear	Bright&Clear	Bright&Clear
Copper Strip Corrosion at 100 °C for 3 hrs.	1a	1a	1a	1a	1a	1a	1a

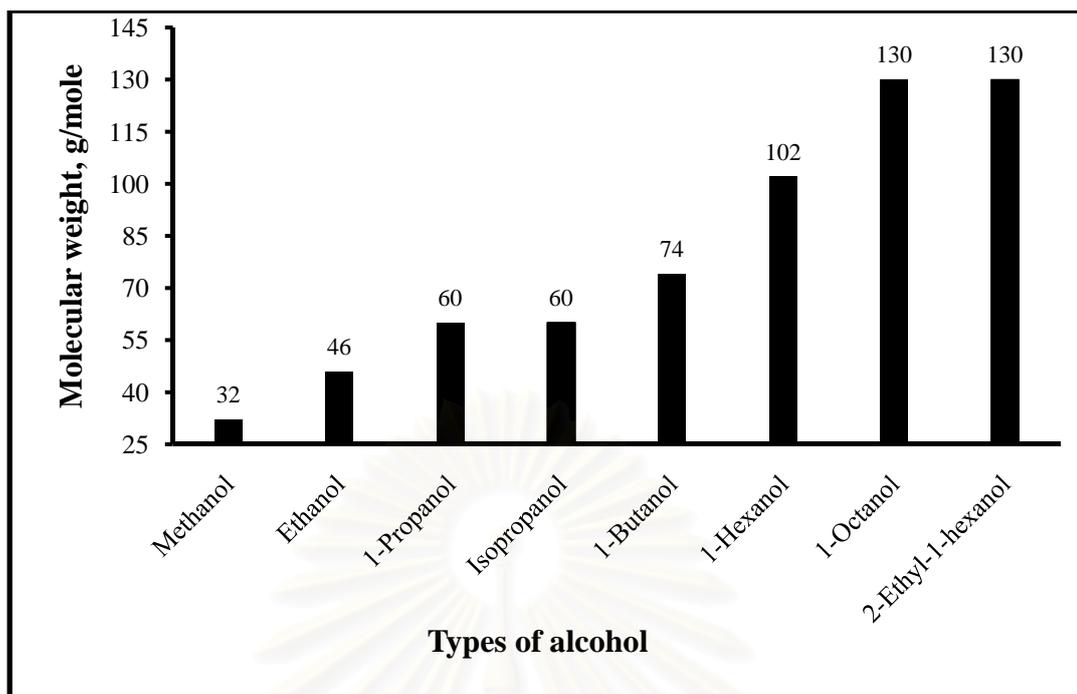


Figure 4.1 The molecular weight of alcohol

4.3.1 ASTM Color

ASTM Color is a color of the products that closely matches with the color of a specific glass standard. The effect of molecular size and molecular structure of alcohols on ASTM color of the alkyl esters is shown in Table 4.8.

The results show that the ASTM color of the alkyl ester increases with increasing of operating temperature from 65 °C to 130 °C which is depended on the types of alcohol, but the molecular size and molecular structure of alcohol did not have any significant affect on the ASTM color of alkyl esters. These results agreed with the study of Phattanaphakdee (1995) which produced alkyl esters via transesterification from rice bran oil with various alcohols such as 1-butanol, 1-hexanol, cyclohexanol and 2-ethyl-1-hexanol. They found that ASTM color of their alkyl ester products increased from L0.5 to L2.0 for the reaction temperature was increased from 80 °C to 120 °C. The results can be concluded that the ASTM color of alkyl ester increases with increasing of the reaction temperature of transesterification reaction.

Table 4.8 The ASTM color of alkyl esters

Alkyl ester	Reaction Temperature (°C)	ASTM Color
Methyl ester	65	L1.0
Ethyl ester	78	L1.0
1-Propyl ester	82	L1.5
Isopropyl ester	97	L1.5
1-Butyl ester	117	L3.0
1-Hexyl ester	130	L3.0
1-Octyl ester	130	L4.0
2-Ethyl-1-hexyl ester	130	L4.0

The results in Table 4.8 show that the ASTM color of the alkyl esters which produced from palm stearin are in the range of L1.0 to L4.0. It is accepted in the specification of lubricating base oil which is the maximum acceptable ASTM color of lubricating base oil is 6.0.

4.3.2 Kinematic viscosity

The kinematic viscosity is the most important physical property of lubricating oil. It is an index for measuring of internal resistance in the motion of the lubricating oil by reason of the cohesion forces between molecules. The kinematic viscosity of lubricating oil change with temperatures; it increases while the temperature decreases, in the opposite, it decreases while the temperature increases.

The effect of molecular size and molecular structure of alcohols on the kinematic viscosity at 40 °C and 100 °C of alkyl esters is illustrated in Figure 4.2 and 4.3, respectively. The results show that the kinematic viscosity at 40 °C and 100 °C

of alkyl ester increases with increasing of molecular size and/or degrees of branched-chain of alcohols. These results agreed with the study of Eiamsupasawat (1999) which produced alkyl esters via transesterification of rice bran oil with various alcohols such as 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol. They found that the kinematic viscosity of their alkyl esters increased from 10.46 to 13.60 cSt at 40 °C and 3.15 to 3.72 cSt at 100 °C when the molecular size of alcohol was increased from 1-butanol to 1-octanol, and the kinematic viscosity of their alkyl esters increased from 13.60 to 14.16 at 40 °C and 3.72 to 3.74 cSt at 100 °C when the degrees of branched-chain of alcohols was increased from 1-octanol to 2-ethyl-1-hexanol, which have different molecular structure of eight carbon atoms of alcohols. All results can be concluded that the molecular structure and molecular size of alcohols affected on the kinematic viscosity of alkyl esters which it increases with increasing degrees of branched-chain and/or molecular size of alcohols.

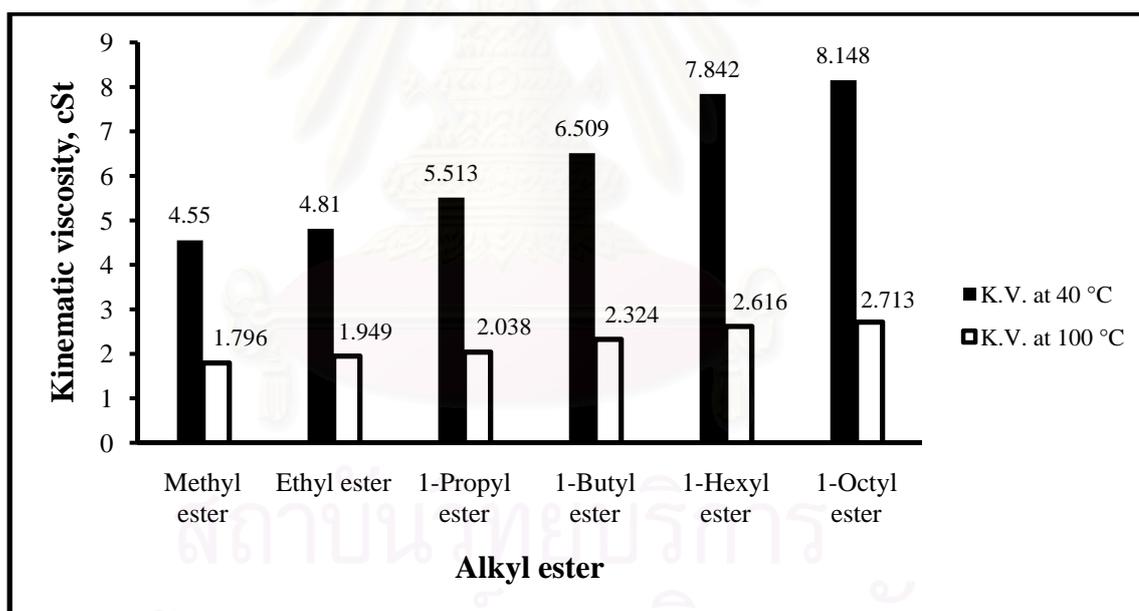


Figure 4.2 The effect of molecular size of alcohol on kinematic viscosity at 40 °C and 100 °C of the alkyl esters.

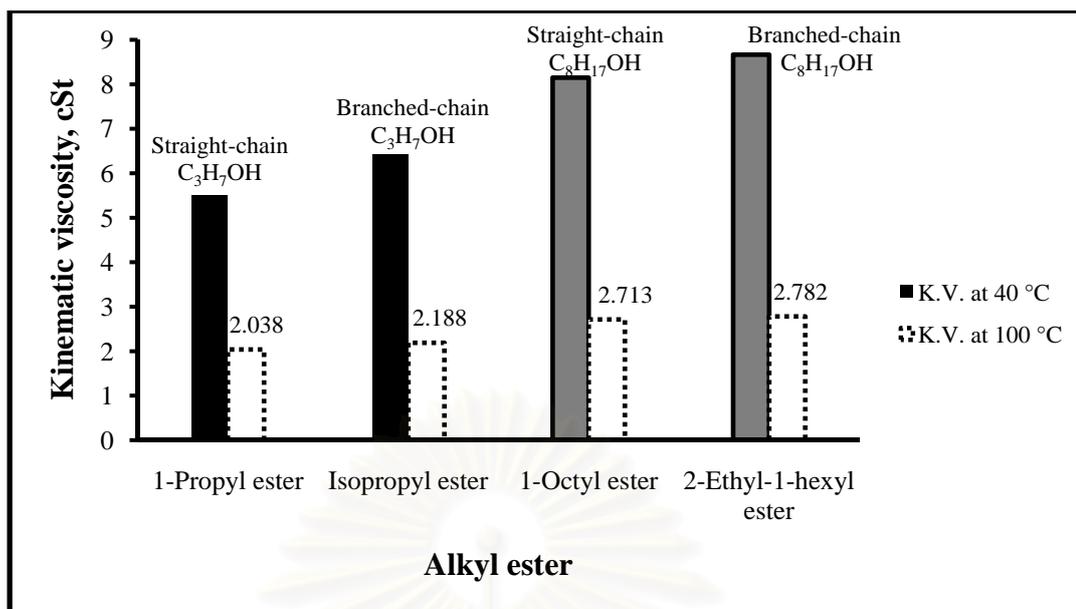


Figure 4.3 The effect of molecular structure of alcohol on kinematic viscosity at 40 °C and 100 °C of the alkyl esters.

The kinematic viscosity of hydrocarbon depends on the chemical structure of the hydrocarbons (Ellis L., 1945). Hegel et al. (1931) observed that for the same molecular weight hydrocarbons, the kinematic viscosity of cyclic hydrocarbon is greater than the kinematic viscosity of paraffin. Introduction of side chain, as well as unsaturated, had little effect on the kinematic viscosity of acrylic hydrocarbons. With cyclic hydrocarbons, the introduction of side chains, particularly methyl group, had a specific effect though out always in the same direction. Thus, the changing of kinematic viscosity of the alkyl esters indicated that the molecular structure of the alcohol was changed.

The results in Figure 4.2 and 4.3 show that the kinematic viscosity of the alkyl esters are in range of 4.55 to 8.667 cSt at 40 °C and 1.796 to 2.782 cSt at 100 °C, while the 2-ethyl-1-hexyl ester has higher kinematic viscosity than other alkyl esters. Although, these results did not match with any types of lubricating base oil in Table 4.9, the alkyl esters may be used as substitute of ready mixed lubricating base oil. In lubricating oil production, the kinematic viscosity of the lubricating base oil is adjusted to meet the specifications of lubricating base oil by blending with several lubricating base oil grades having different kinematic viscosity. However, other

properties of lubricating base oil such as viscosity index, pour point and flash point are also considered.

Table 4.9 The specification of standard lubricating base oil.

Types of lubricating base oil	Physical properties				
	Kinematic viscosity (cSt)		Viscosity index	Pour point (°C)	Flash point (°C)
	40 °C	100 °C			
Engine oil	-	4.1 – 6.6	>90	<-5	>190
2 stroke oil	-	5.6 – 7.8	>95	<-5	>70
Automotive gear oil	-	13.5 – 15.5	>85	<-5	>200
Industrial gear oil	28.8 – 35.2	-	>90	<-10	>200
Hydraulic oil	9.0 – 11.0	2.5 Typical	>75	<12	>125
Turbine oil	28.8 – 35.2	5.0 Typical	>90	<-6	>160
Refrigerator compressor oil	28.8 – 35.2	5.7 Typical	>90	<-20	>200
Air compressor oil	28.8 – 35.2	5.6 Typical	>90	<-10	>200
Heat transfer oil	28.8 – 35.2	5.7 Typical	>90	-	>200
Mineral base oil 150 SN	29.0-31.0	-	>100	<-12	>234

4.3.3 Viscosity Index

The viscosity index is an arbitrary number indicating the effect of changing temperature on the kinematic viscosity of alkyl esters. A high viscosity index signifies a relatively small change of kinematic viscosity with temperature.

The effect of molecular size and molecular structure of alcohols on viscosity index of the alkyl esters is shown in Figure 4.4 and 4.5, respectively. The results show that the viscosity index of alkyl ester decreases with increasing degree of branched-chain of alcohol, but the molecular size of alcohol did not have any significant affect on the viscosity index of alkyl esters. The viscosity index of 1-propyl ester (straight-chain structure) higher than isopropyl ester (branched-chain structure), which it has three carbon atoms of alcohol chain length with different molecular structure because the isopropyl ester has Van Der Waals Interaction lower than 1-propyl ester. With the same reason, 2-ethyl-1-hexyl ester has a lower viscosity index than 1-octyl ester which has different structure of eight carbon atoms of alcohol chain length. These results agreed with the studies of Phattanaphakdee (1995), Suwanprasert (1996), Vatanaputi (1996) and Eiamsupasawat (1999) that observed the viscosity index of alkyl esters. They found that the viscosity index of their alkyl esters decreases with increasing degrees of branched-chain of alcohol. All results can be concluded that the molecular structure of alcohol affected on the viscosity index of alkyl ester, which it decreases with increasing degrees of branched-chain of alkyl ester.

The viscosity index of alkyl esters shows a greater value than the limit of lubricating base oil specifications as shown in Table 4.9. The specification of the lubricating base oil limits the value of viscosity index at more than 75, while the viscosity index of alkyl esters are in range of 169 to 212 which are accepted in specification of lubricating base oil. The alkyl esters were used as viscosity index improving in lubricant industries because it has high viscosity index which discussed in section 4.4.

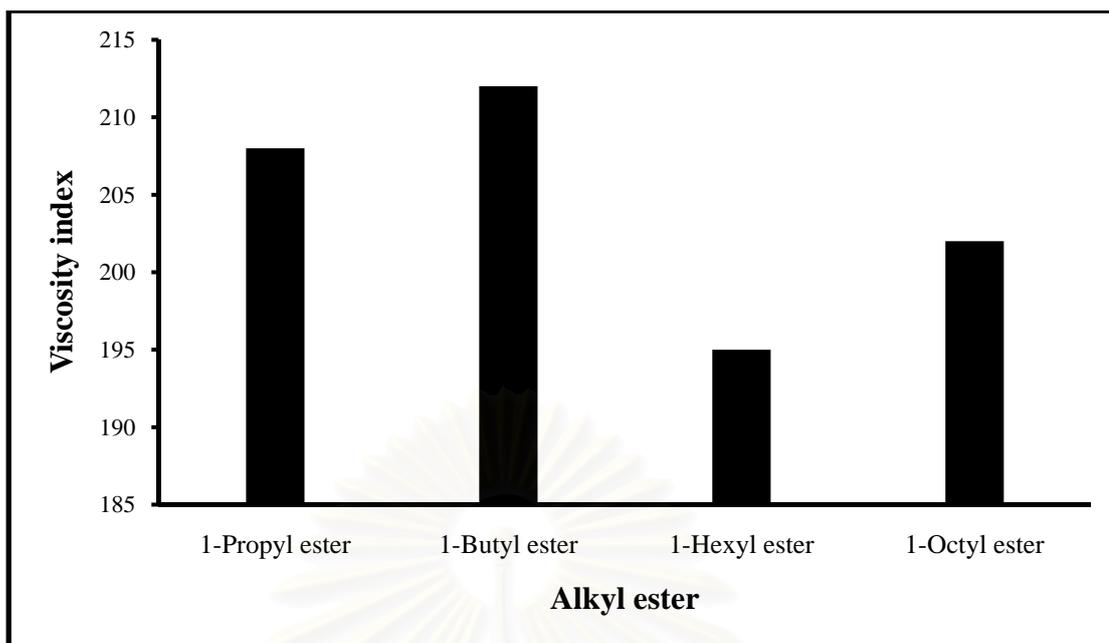


Figure 4.4 The effect of molecular size of alcohol on viscosity index of the alkyl esters.

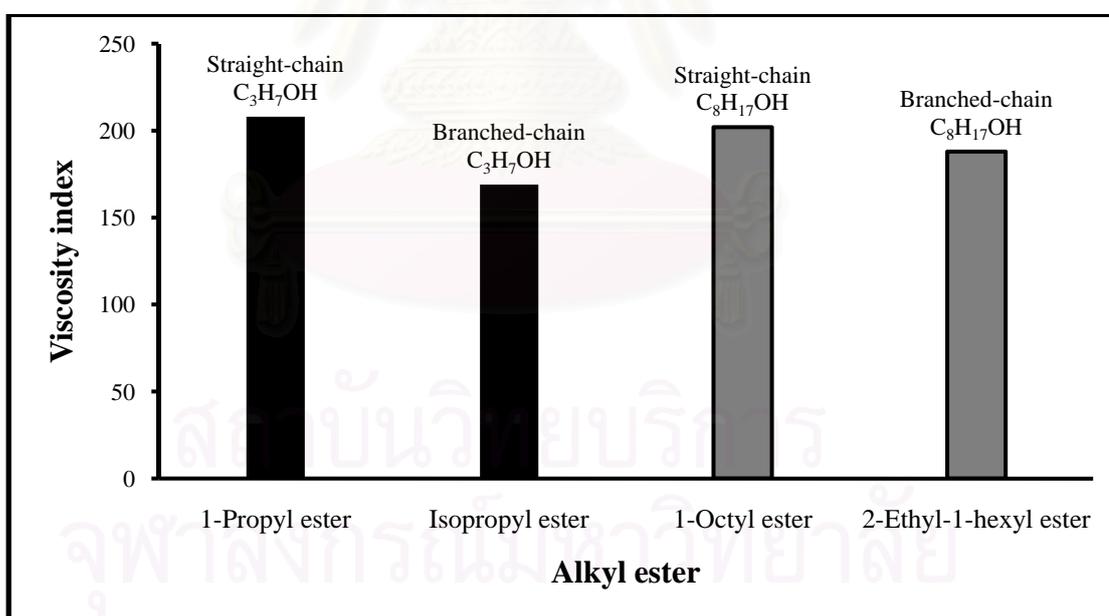


Figure 4.5 The effect of molecular structure of alcohol on viscosity index of the alkyl esters.

4.3.4 Pour point

The pour point of substance is the lowest temperature which it will pour or flow when it is chilled without disturbance under prescribed conditions. The results in Figure 4.6 and 4.7 show the effect of molecular size and molecular structure of alcohols on pour point of alkyl esters, respectively.

The results show that the pour point of the alkyl ester decreases with increasing molecular size and/or degree of branched-chain of alcohols. The results agreed with the study of Vatanaputi (1996) and Hong et al. (2005) which produced alkyl esters via transesterification of coconut and soybean oil with various alcohols, respectively. They found that the pour point of their alkyl esters decreased when the molecular size and degree of branched-chain of alcohol was increased. All results can be concluded that the pour point of alkyl ester decreases with increasing the molecular size of alcohol and/or degree of branched-chain of alcohol increases.

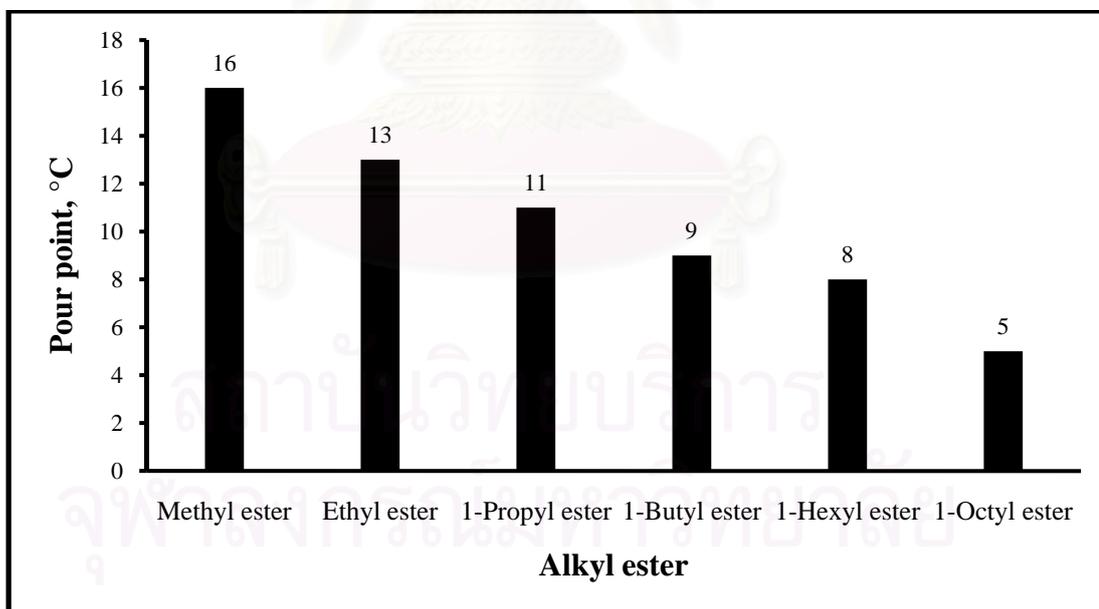


Figure 4.6 The effect of molecular size of alcohol on pour point of the alkyl esters.

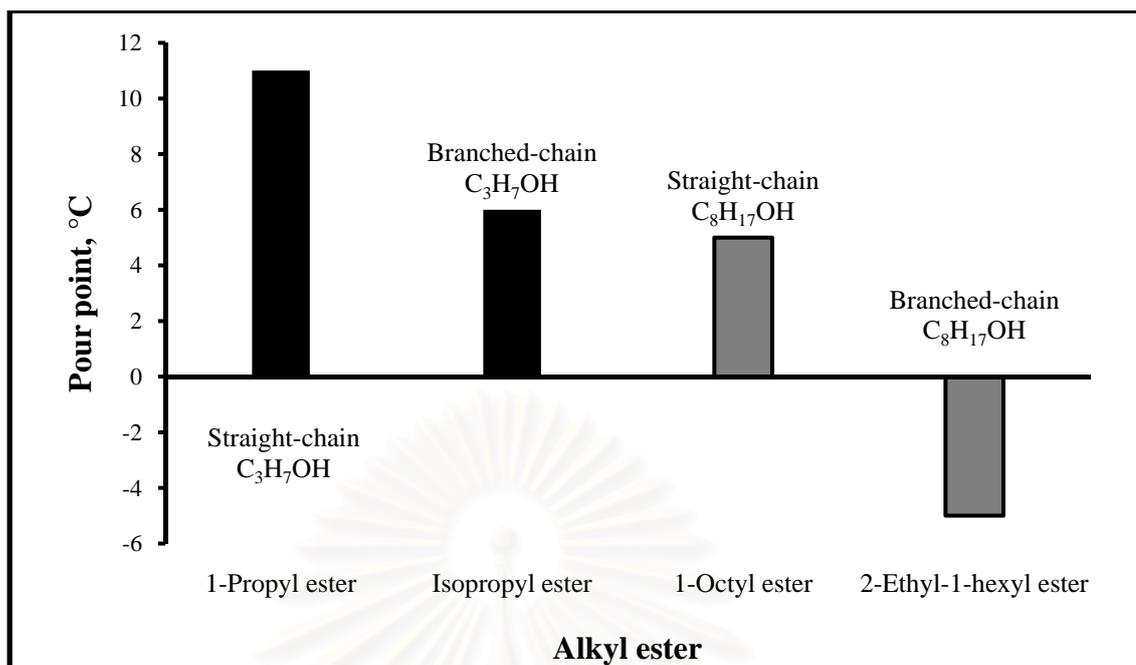


Figure 4.7 The effect of molecular structure of alcohol on pour point of the alkyl esters.

The pour point of alkyl esters meets all of the specification of lubricating base oil is shown in Table 4.9. The pour point values of alkyl esters are in range of 16 °C to -5 °C which is acceptable in specification of hydraulic oil, engine oil and automotive gear oil. In lubricating base oil production, the pour point of the lubricating base oil is adjusted to meet the specifications of lubricating base oil by blending with pour point depressant in order to improve cold properties of lubricating base oil.

4.3.5 Flash point

The flash point of substance is the temperature at which the substance releases enough vapor at its surface to ignite when an open flame is applied. The flash point of substance varies according to the degree of kinematic viscosity; higher kinematic viscosity substances tend to have higher flash point. The effect of molecular size and molecular structure of alcohols on flash point of alkyl esters are illustrated in Figure 4.8 and 4.9.

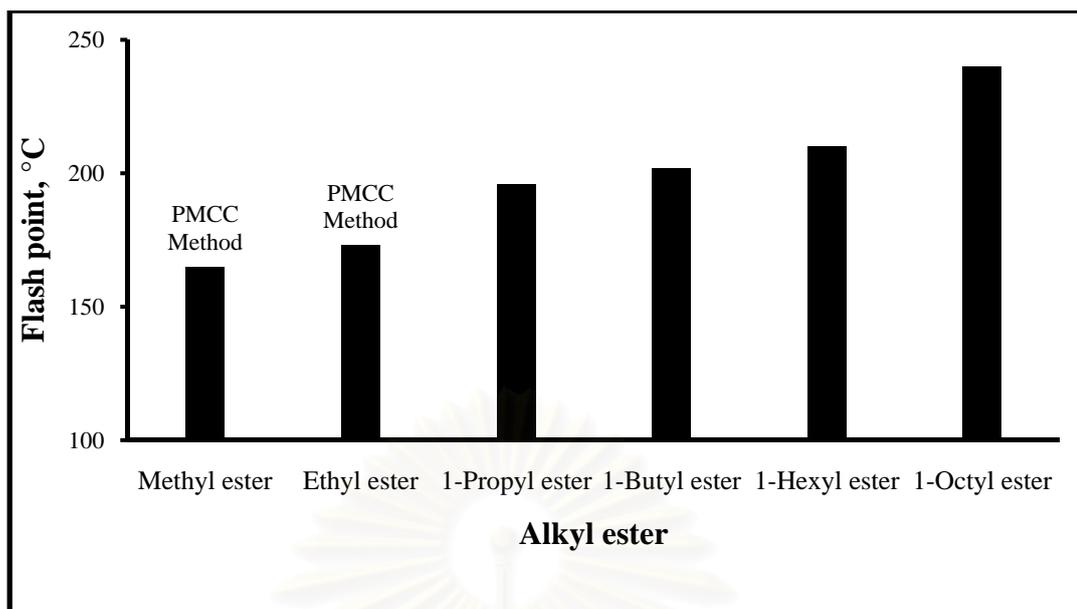


Figure 4.8 The effect of molecular size of alcohol on flash point of the alkyl esters.

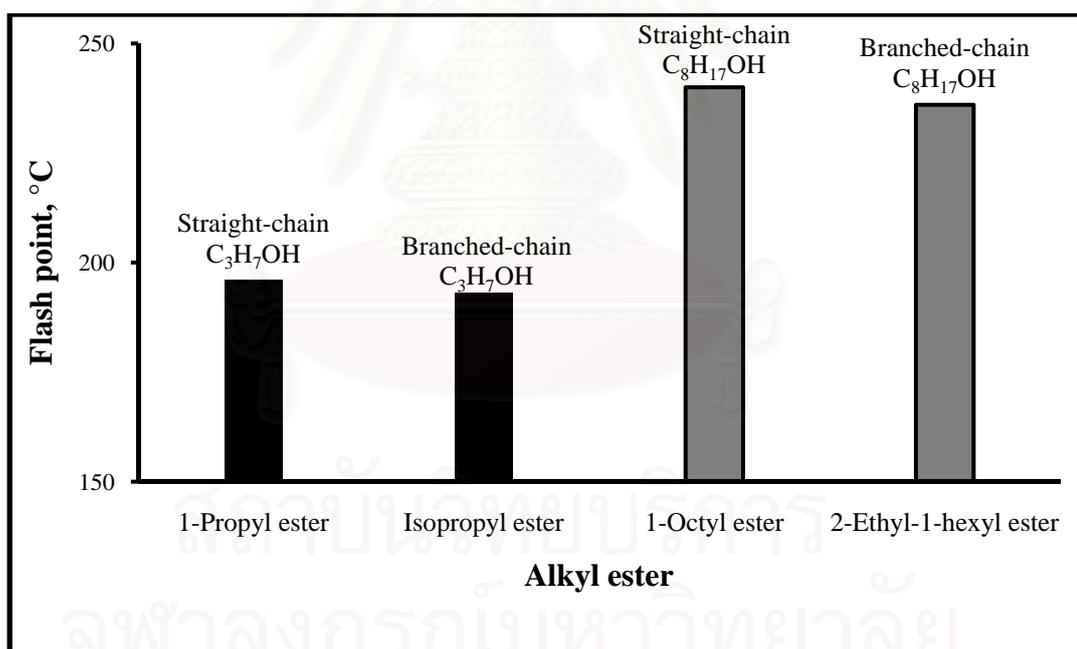


Figure 4.9 The effect of molecular structure of alcohol on flash point of the alkyl esters.

The results show that flash point of the alkyl ester increases with increasing of molecular size and decreases with increasing degree of branched-chain structure of alcohols. These results agreed with the study of Vatanaputi (1996) who showed that the flash point of alkyl esters increased from 195 °C to 198 °C for the molecular size of alcohol was increased from 1-hexanol to 1-octanol, and decreased from 198 °C to 197 °C for degree of branch-chain of alcohol was increased from 1-octanol to 2-ethyl-1-hexanol, which have different molecular structure of eight carbon atoms of alcohols. All results can be concluded that the flash point of alkyl ester increases as the molecular size of alcohol increases and decreases as the degree of branched-chain of alcohol increases.

The flash point of a petroleum product is also used to detect contamination. A substantially lower flash point than expectation for a product is a reliable indicator that a product has become contaminated with more volatile product such as gasoline. The flash point is also an aid in establishing the identity of a particular petroleum product. A further aspect of volatility that receives considerable attention is the vapor pressure of petroleum and its constituent fractions. The vapor pressure is the force exerted on the walls of a closed container by the vaporized portion of a liquid. Conversely, it is the force that must be exerted on the liquid to prevent it from vaporizing further. The vapor pressure increases with temperature for any given gasoline, liquefied petroleum gas, or other product. The temperature at which the vapor pressure of a liquid equals 1 atm is designated as the boiling point of the liquid.

In each homologous series of hydrocarbons, the boiling points increase with molecular size and molecular structure also has a marked influence. It is a general rule that branched-paraffin isomers have lower boiling points than the corresponding n-alkane. However, the most dramatic illustration of the variation in boiling point with carbon number is an actual plot for different hydrocarbon as shown in Figure 4.10. In any given series, steric effects notwithstanding, there is an increase in boiling point with an increasing in carbon number of the alkyl side chain as shown in Figure 4.11. This particularly applies to alkyl aromatic compounds where alkyl substituted aromatic compounds can have higher boiling points than

poly-condensed aromatic systems. These theoretical can be summarized that the flash point of substance changes with the boiling point of substance; it increases as the boiling point of substance increases and decreases as the boiling point of substance decreases. The boiling point of substance increases with increasing molecular size and/or decrease the degree of branched-chain of substance.

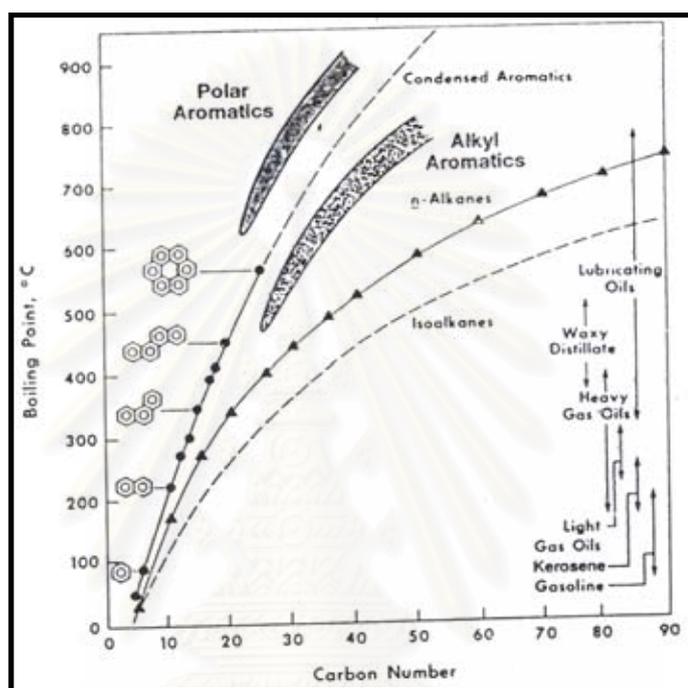


Figure 4.10 The relationship of boiling point to carbon number for hydrocarbon compounds (James, G.S., 2007)

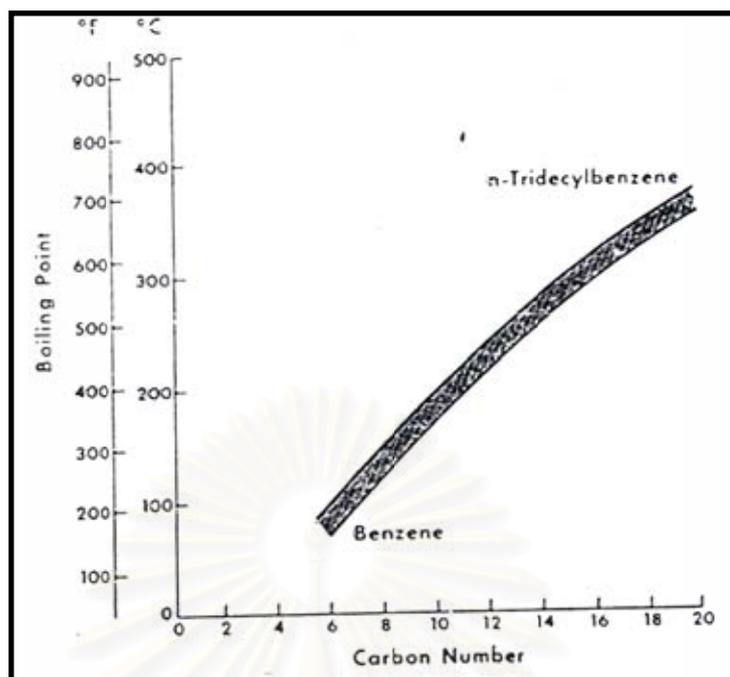


Figure 4.11 The relationship of boiling point to carbon number for the alkylbenzenes (James, G.S., 2007)

The flash point of alkyl esters meets all of the specification of lubricating base oil as shown in Table 4.9. The specification values of lubricating base oil are in range 125 °C to 234 °C while the flash point values of alkyl esters are in range of 165 °C to 240 °C which is accepted in specification of lubricating base oil such as engine oil, 2-stroke oil, turbine oil and air compressor oil.

4.3.6 API Gravity

API gravity is an arbitrary scale, calibrated in degrees and related to the specific gravity and density. The API gravity value increases as the specific gravity decrease. The results in Table 4.7 show that the API gravity of alkyl ester decreases with increasing molecular size and/or degree of branched-chain of alcohols.

The specific gravity, density and API gravity are influenced by the chemical composition of petroleum, but quantitative correlation is difficult to establish. Nevertheless, it is generally recognized that increased amounts of aromatic compounds result in an increase in density, whereas an increase in saturated

compounds results in a decrease in density. Indeed, it is also possible to recognize certain preferred trends between the density and API gravity of petroleum and other physical properties such as viscosity as shown in Figure 4.12 and 4.13 (James, G.S., 2007).

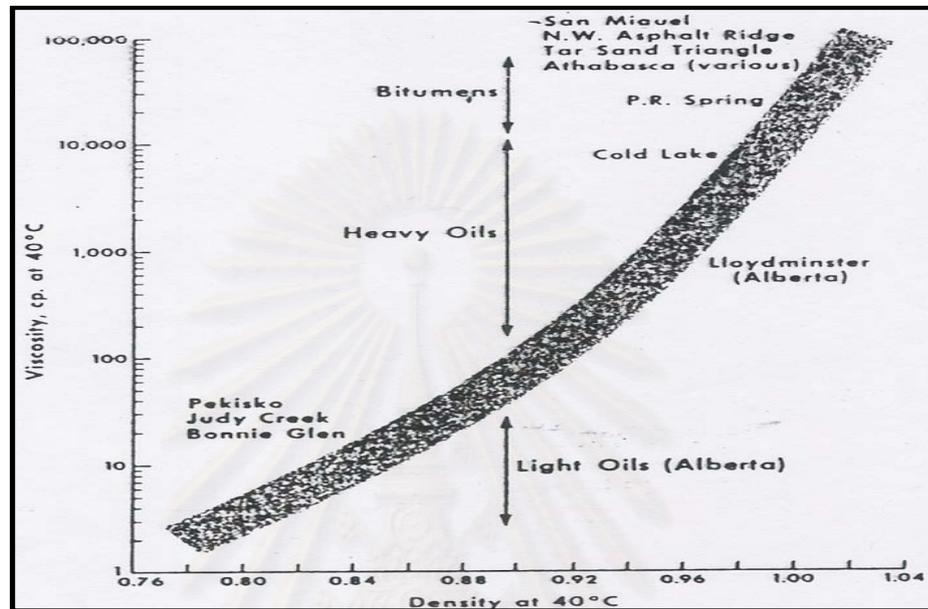


Figure 4.12 Relationship of density and viscosity (James, G.S., 2007)

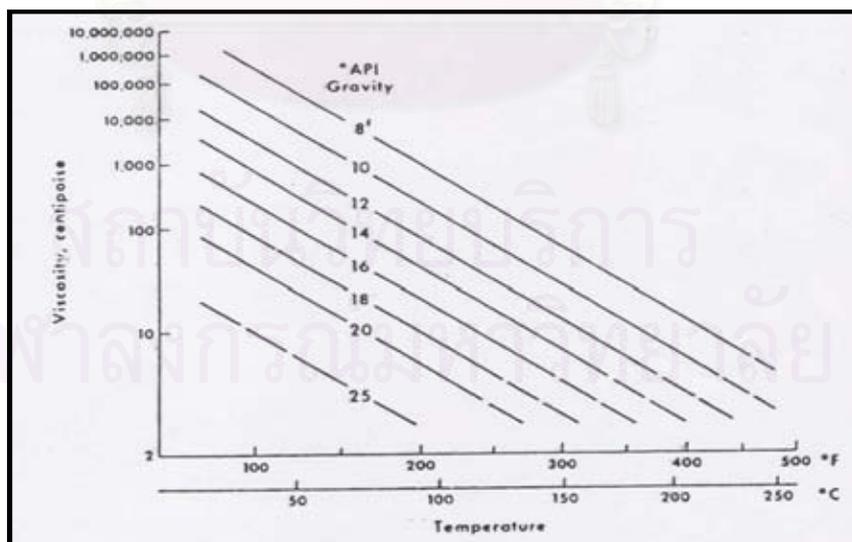


Figure 4.13 The viscosity as a function of temperature and API Gravity (James, G.S., 2007)

Figure 4.12 and 4.13 show that the density increases with increasing the viscosity of petroleum, but the API gravity increases with decreasing the viscosity of petroleum. It can be described by the relationship between kinematic viscosity (ν), dynamic viscosity (η) and density (or specific gravity) of substance as shown in Equation 4.2 to Equation 4.4

$$\nu \rho = \eta \quad (4.2)$$

$$\text{From} \quad \text{S.G. gravity} = \left(\frac{141.5}{\text{API Gravity} + 131.5} \right) \quad (4.3)$$

\therefore Equation (4.3) substituted in Equation (4.2);

$$\nu \left(\frac{141.5}{\text{API Gravity} + 131.5} \right) = \eta \quad (4.4)$$

Equation 4.2 and 4.4 can be confirmed that the density increases with increasing the viscosity of substance, but the API gravity decreases with increasing the viscosity of substance.

4.3.7 Copper strip corrosion

The copper strip corrosion was measured the relative degree of corrosives of a sample. A polish copper strip is immersed in a given quantity of sample and heated at a temperature and for a time characteristic of the material being tested.

The results in Table 4.7 show that the molecular size and molecular structure of alcohols did not have any significant affect on the copper strip corrosion of alkyl esters. The copper strip corrosion of alkyl esters is 1a which meets all of the specification of mineral base oil 150 SN. The specification of mineral base oil 150 SN limits the maximum copper strip corrosion at 100 °C for 3 hours is 1b, which it has lower quality than alkyl esters.

4.3.8 Four-ball test (Weld load)

The four-ball wear test method can be used to determine the relative wear-preventing properties of lubricants in sliding steel-on-steel applications under the test condition and if the test conditions are changed the relative rating may be different. For evaluating the Extreme-Pressure (load-carrying) capacity of lubricants, the normal load at which welding occurs at the contact interface can be recorded.

The effect of molecular size and molecular structure of alcohols on weld load of alkyl esters is shown in Table 4.10. The results show that the molecular size and molecular structure of alcohols did not have significant affect on the weld load of alkyl esters, but the types of alcohol which used to produce alkyl esters have affected on the weld load of the alkyl esters. However, the weld load of alkyl esters meets all of the specification of hydraulic oil; minimum 160 kg, while the weld load of alkyl esters are in range of 160 - 250 kg.

Table 4.10 The effect of molecular size and molecular structure of alcohols on the weld load of alkyl esters.

Alkyl esters	Weld Load, kg
1-Propyl ester	160
Isopropyl ester	160
1-Butyl ester	200
1-Hexyl ester	200
1-Octyl ester	200
2-Ethyl-1-hexyl-ester	250

All results indicated that the molecular size and molecular structure of alcohols affected on the physical and chemical properties of alkyl esters; such as ASTM color, kinematic viscosity, viscosity index, pour point, flash point, API

gravity, weld load and copper strip corrosion as summarized in Table 4.11. Our results also indicated that 2-ethyl-1-hexyl ester has suitable physical and chemical properties to use as synthetic lubricating base oil such as hydraulic oil because it has a high viscosity index, low pour point, high flash point and highest kinematic viscosity which meet in specification of hydraulic oil.

Table 4.11 The effect of molecular size and molecular structure of alcohols on the properties of alkyl esters.

Properties	Effect of molecular size of alcohol	Effect of degree of branched-chain of alcohol
ASTM Color	Not significant	Not significant
Kinematic viscosity	Yes	Yes
Viscosity index	Not significant	Yes
Pour point	Yes	Yes
Flash point	Yes	Yes
API Gravity	Yes	Yes
Copper strip corrosion	Not significant	Not significant
Weld load	Not significant	Not significant

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4.4 The effect of alkyl esters quantity on mineral base oil 150 SN properties

The results from previous section show that the properties of each alkyl esters which are produced from transesterification of palm stearin and alcohols are quite suitable to use as additive in lubricants. Owing to every alkyl esters have their own distinctive point; there are developed to be an additive in lubricating base oil. Especially, the viscosity index of each alkyl esters is rather higher than viscosity index of mineral base oil 150 SN. These special properties of alkyl esters lead to an idea of blending mineral base oil with alkyl esters in order to improve lubricating base oil property. So, in this section studies about the opportunity to use these alkyl esters as the viscosity index improver in mineral base oil 150 SN. The various amounts of the alkyl esters, i.e., 3, 5, 8, 13, 18 and 20 % wt. are blended with mineral base oil 150 SN in order to observe the trend and investigate the possibility to be the viscosity index improver. The experimental procedure is described in section 3.3.2. After that, the physical and chemical properties of blended oil which are kinematic viscosity, viscosity index, pour point and ASTM color are analyzed and shown in Table 4.11. The results also show the effect of alkyl ester quantities on mineral base oil 150 SN properties. The relations of the alkyl ester quantity and blended oil properties are shown in Figure 4.14 to 4.17.

The results in Table 4.12 and Figure 4.14 to 4.17 indicated that the blended oil has different properties from the mineral base oil 150 SN without blending. The blended oil which mixed with the different types of alkyl esters shows the same trend but the unequal value as shown in Figure 4.14 to 4.17. The higher quantity of alkyl ester in blended oil makes the wider gap comparing with mineral base oil 150 SN without blending. So, the types and quantity of alkyl ester has an influence on the mineral base oil 150 SN properties which are divided into four parts as followed:

Table 4.12 Properties of mineral base oil 150 SN blended with various alkyl esters

Properties Lube base oil		ASTM Color	Kinematic Viscosity (cSt)		Viscosity index	Pour point (°C)
			40 °C	100 °C		
Mineral base oil 150 SN		L0.5	30.30	5.25	104	-12.0
1-Propyl ester (wt. %) in MBO 150 SN	3 %	L0.5	27.91	5.01	105	-11.0
	5 %	L0.5	26.44	4.87	106	-9.0
	8 %	L0.5	24.50	4.70	109	-7.0
	13 %	L0.5	21.73	4.41	113	-4.0
	18 %	L0.5	19.38	4.15	117	-4.0
	20 %	L1.0	18.66	4.07	118	-2.0
Isopropyl ester (wt. %) in MBO 150 SN	3 %	L 0.5	27.92	5.03	106	-11.0
	5 %	L0.5	26.72	4.90	106	-9.0
	8 %	L0.5	24.89	4.73	109	-8.0
	13 %	L0.5	22.24	4.46	112	-6.0
	18 %	L1.0	20.00	4.21	115	-6.0
	20 %	L1.0	18.97	4.11	118	-5.0
1-Butyl ester (wt. %) in MBO 150 SN	3 %	L0.5	28.35	5.07	106	-11.0
	5 %	L0.5	26.50	4.97	113	-9.0
	8 %	L0.5	25.08	4.83	115	-6.0
	13 %	L1.0	22.14	4.54	120	-3.0
	18 %	L1.0	20.03	4.29	122	-3.0
	20 %	L1.0	19.03	4.25	132	-3.0
1-Hexyl ester (wt. %) in MBO 150 SN	3 %	L0.5	28.98	5.11	104	-11.0
	5 %	L0.5	27.71	5.10	113	-9.0
	8 %	0.5	26.05	4.95	115	-6.0
	13 %	L1.0	24.69	4.82	117	-4.0
	18 %	L1.0	22.90	4.67	123	-4.0
	20 %	L1.0	22.05	4.60	126	-4.0
1-Octyl ester (wt. %) in MBO 150 SN	3 %	L0.5	29.25	5.17	106	-11.0
	5 %	0.5	27.99	5.12	112	-11.0
	8 %	L1.0	27.21	5.06	114	-9.0
	13 %	1.0	25.99	4.97	117	-6.0
	18 %	L1.5	23.56	4.76	124	-6.0
	20 %	L1.5	22.67	4.69	127	-4.0
2-Ethyl-1-hexyl ester (wt. %) in MBO 150 SN	3 %	L0.5	29.56	5.20	106	-12.0
	5 %	0.5	28.25	5.15	111	-11.0
	8 %	L1.0	27.45	5.10	115	-9.0
	13 %	L1.0	26.28	5.04	118	-9.0
	18 %	1.0	24.87	4.92	121	-9.0
	20 %	L1.5	23.55	4.78	125	-6.0

- **Kinematic viscosity**

The results in Table 4.11 indicated that the alkyl esters affected on the kinematic viscosity of mineral base oil 150 SN because the kinematic viscosity at 40 °C and 100 °C of blended oils are lower than the mineral base oil 150 SN without blending. Moreover, the increasing of alkyl esters in blended oil affected on the lower kinematic viscosity value. The results also show that the blended oil with 2-ethyl-1-hexyl ester has the least affect on mineral base oil 150 SN because of the less changing in kinematic viscosity from 3 to 20 % wt. of alkyl ester. While as the blended oil with 1-propyl ester has the most affect on the kinematic viscosity of mineral base oil 150 SN.

- **Viscosity Index**

The result in Figure 4.16 shows that the viscosity index of mineral base oil 150 SN increased when the amount of alkyl ester increasing from 3 to 20 % wt. So, the amounts of alkyl esters have significantly influenced on of mineral base oil 150 SN. As this result, it shows that the possibility to use these alkyl esters to be a viscosity index improver in lubricant industries. Especially, the 1-butyl ester demonstrates the highest efficiency of viscosity index improver which can increase the viscosity index of mineral base oil 150 SN from 104 to 130.

- **Pour point**

The alkyl esters affected on the pour point of the mineral base oil 150 SN because the increasing of pour point follows by the increasing of alkyl ester quantity. However, the effect of alkyl ester quantity is insignificant when the quantity of alkyl esters is more than 13 % wt. because are slightly change of the pour point in each sample as shown in Figure 4.17. The results also show that 1-propyl ester and 2-ethyl-1-hexyl ester have the most and least affect on the pour point property, respectively. In the production of lubricant, the lubricating base oil is put the pour point depressant in order to keep and adjust the pour point of lubricating base oil.

- ASTM color

The changing of ASTM color depended on the types and amount of alkyl esters. The darker color of blended oil increases with the increasing amount of alkyl esters from 3 to 20 % wt.. The results also show that 1-octyl ester is the most affect on the ASTM color of mineral base oil 150 SN.

All the results can be concluded that the alkyl esters which produced from palm stearin with various alcohols, i.e., 1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol can be used as the additive to improve and develop the mineral base oil 150 SN properties. These alkyl esters are able to decrease kinematic viscosity and increase viscosity index, pour point and ASTM color of mineral base oil 150 SN. Moreover, there is the possibility to apply these alkyl esters to be the additive in other types of lubricating base oil. It depends on the standard range of properties in each type of lubricating base oil that shows in Table 4.9. The type and quantity of alkyl esters that are selected to use as the additive should be suitable for the required properties because the blending with these alkyl esters not only change one property but also change many properties of lubricating base oil. Sometimes, must be adding other kind of additive when using these alkyl esters to adjust the properties. For example, if the 1-butyl ester is blended with engine oil, the pour point of engine oil will be higher than the standard value. So, the pour point depressant (other kind of additive) should be required to drop the pour point and improve the quality of product. According to the above discussion, this thesis also proves that the alkyl esters which are produced from palm stearin can use as the viscosity index improver to increase the viscosity index of lubricating base oil.

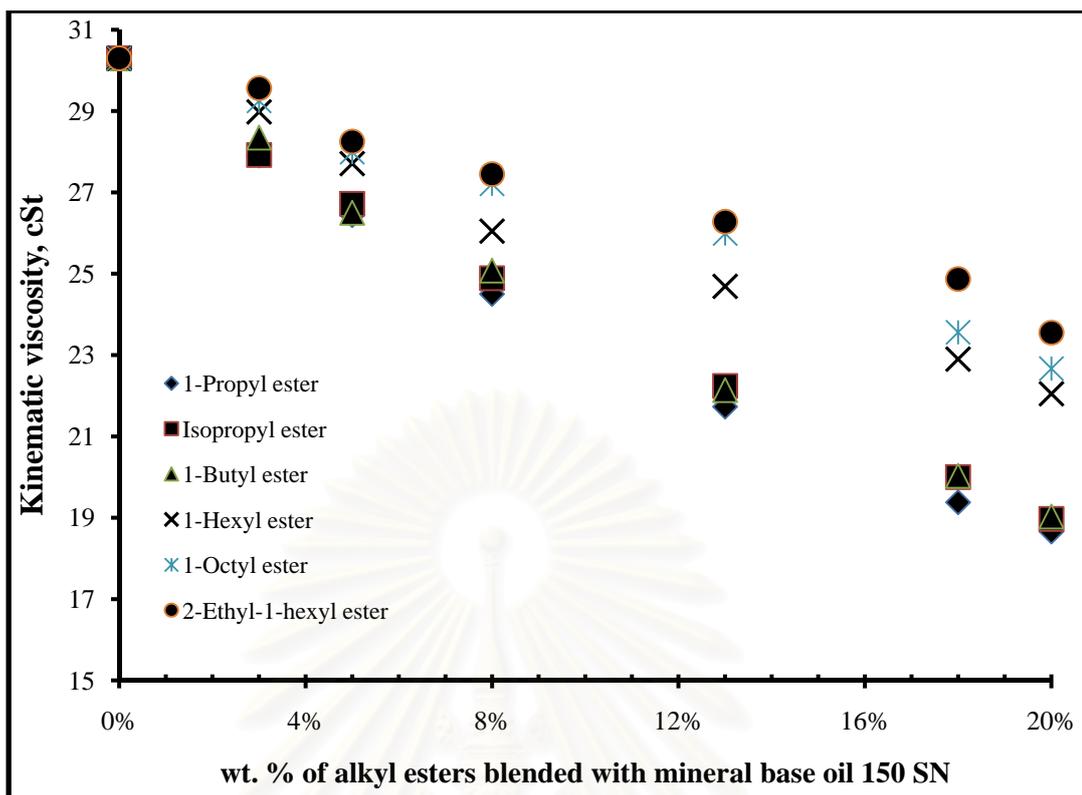


Figure 4.14 The kinematic viscosity at 40 °C of the blended oil

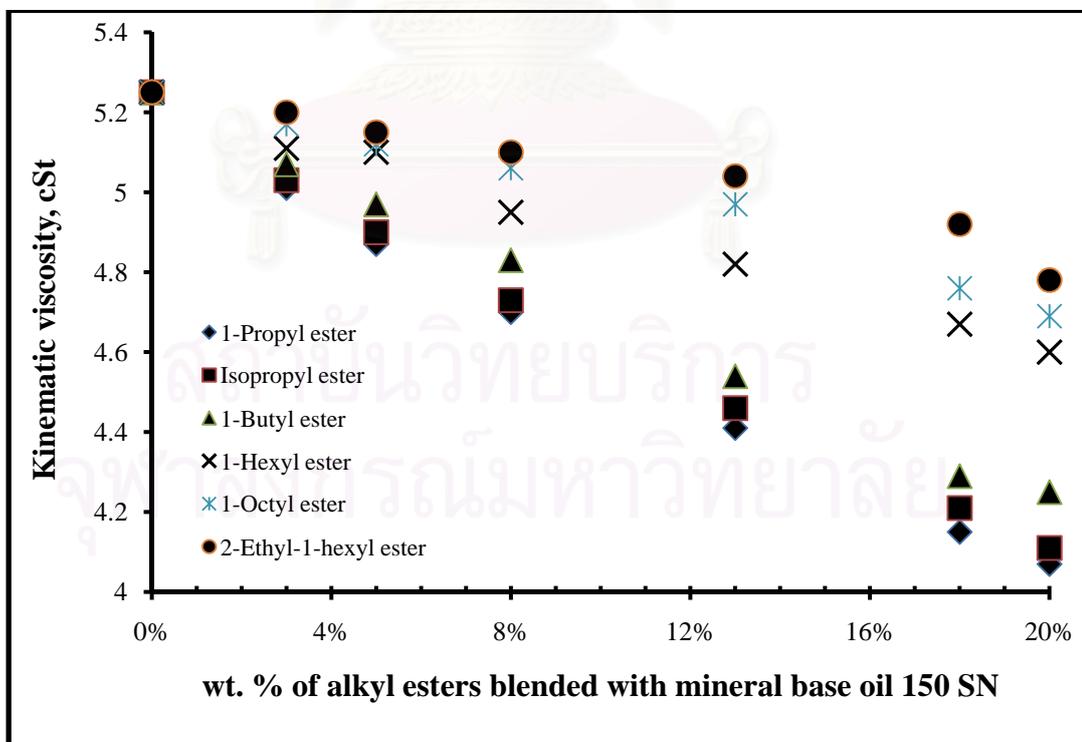


Figure 4.15 The kinematic viscosity at 100 °C of the blended oil

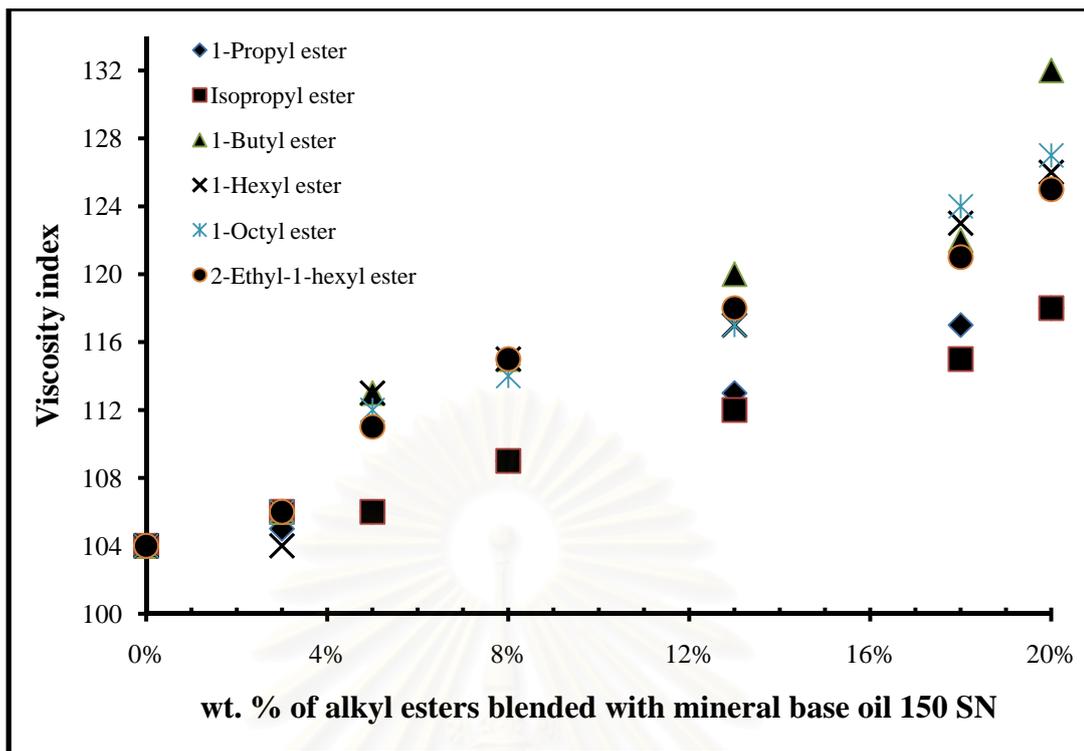


Figure 4.16 The viscosity index of the blended oil

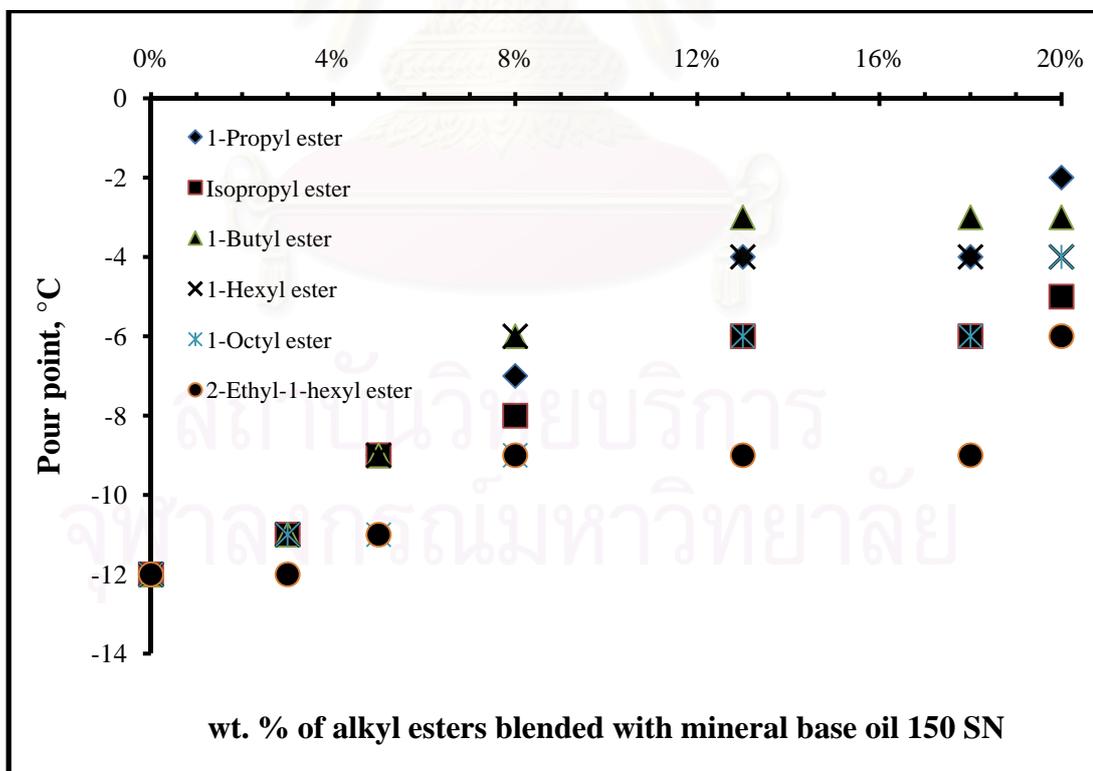


Figure 4.17 The pour point of the blended oil

CHAPTER V

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The conclusions of the present research are the following:

1. The alkyl esters can be produced by transesterification from palm stearin with various alcohols (1-propanol, isopropanol, 1-butanol, 1-hexanol, 1-octanol and 2-ethyl-1-hexanol) using sulfuric acid as a catalyst.
2. The various types of alcohol have an influence on the physical and chemical properties of alkyl esters because each alcohol has different molecular size and molecular structure.
3. The higher molecular size and higher degree of branched-chain structure of alcohol provide the advantage to alkyl esters properties to have higher kinematic viscosity and flash point, lower pour point which are suitable to use as lubricating base oil.
4. The 2-ethyl-1-hexyl ester is only one alkyl ester that can perform as lubricating base oil, hydraulic oil, because its properties keep in the specification of hydraulic oil.
5. The 1-butyl ester play role as the most effective viscosity index improver in this study.
6. The alkyl esters which are produced from palm stearin have possibility to use as the additive in lubricating base oil 150 SN in order to improve the quality of lubricating base oil.

5.2 Recommendations

From the previous conclusions, the following recommendations for future studies can be proposed.

1. Owing to the limit of the reaction temperature which are lower than boiling point of 1-hexanol, 1-octanol and 2-ethyl-1-hexanol, it affect on the rate of reaction. So, it should be added with some azeotrope compound such as toluene to reduce the boiling point of mixture.
2. The other alcohols which have different molecular structure, i.e., cyclic alcohol should be studied to confirm the effect of molecular structure on alkyl esters properties.
3. The alkyl esters should be blended with other types of lubricating base oil in order to investigate the effect of alkyl ester on lubricating base oil properties such as kinematic viscosity, viscosity index, flash point and pour point.

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APPENDICES

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APPENDIX A

CHARACTERIZATION OF PALM STEARIN

A1. Acid Value (Free Fatty Acid Content), AOCS Official Method Cd 3d-63

The acid value is the number of milligrams of potassium hydroxide necessary to neutralize the free acids in 1 gram of sample. With samples that contain virtually no free acids other than fatty acids, the acid value may be directly converted by means of suitable factor to percent free fatty acids.

Apparatus

1. Erlenmeyer flasks 250 ml and Magnetic stirring device
2. Burette—10 ml, graduated in 0.05 ml divisions with a tip drawn to a fine opening and extending at least 10 cm below the stopcock.

Reagents and chemicals

1. Potassium hydroxide (KOH), 0.1 N—baker “Reagent Grade” KOH having a carbonate specification of 0.5% Max, or 0.1 N KOH is available from Baker with NIST traceable standardization to ± 1 part in 1000 in solvents of water, methanol or ethanol
2. Solvent mixture consisting of equal parts by volume of isopropyl alcohol (AOCS Specification H 18-58) and toluene (AOCS Specification H 19-58)
3. Phenolphthalein indicator solution—1.0% in isopropyl alcohol

Procedure

1. Add indicator solution to the required amount of solvent in ratio of 2 ml and neutralize with alkali to a faint but permanent pink color.
2. Determine the sample size from the following table:

Table A1 Table of predict sample size for acid value determination

Acid value	Mass of sample (± 10%), g	Weighing accuracy, ± g
0 – 1	20	0.05
1 – 4	10	0.02
4 – 15	2.5	0.01
15 – 75	0.5	0.001
75 and over	0.1	0.0002

3. Weight the specified amount of well-mixed liquid sample into an Erlenmeyer flask.
4. Add 125 ml of the neutralized solvent mixture. Be sure that the sample is completely dissolved before titrating. Warming may be necessary in some cases.
5. Shake the sample vigorously while titrating with standard alkali to the first permanent pink color of the same intensity as that of the neutralized solvent before the latter was added to the sample. The color must persist for 30 sec.

Calculations

$$\text{Acid value (A.V.), mg KOH/g of sample} = \frac{(A - B) \times N \times 56.1}{W}$$

Where A = volume, ml of standard alkali used in the titration

B = volume, ml of standard alkali used in the titrating blank

N = normality of standard alkali

W = mass, grams of sample

To express in terms of free fatty acids as percent oleic, lauric or palmitic acid divide the acid value by 1.99, 2.81 or 2.19, respectively

Table A2 Acid Value using AOCS Official Method Cd 3d-63

	unit	1	2	3
Weight of Palm stearin	g	20.0035	20.0189	20.0247
Volume of solvent mixture	ml	125	125	125
Conc. of KOH	N	0.1	0.1	0.1
Volume of KOH, used in sample	ml	0.60	0.60	0.65
Volume of KOH,used in Blank	ml	0.15	0.15	0.15
Acid Value (A.V.)	Mg KOH/g	0.1262	0.1261	0.1401
Average Acid Value	Mg KOH/g	0.1308 \approx 0.13		
% Free Fatty acid (as Palmitic Acid)	-	0.06		

A2. Saponification Value, AOCS Official Method Cd 3b-76

The saponification value is the amount of alkali necessary to saponify a definite quantity of the sample. It is expressed as the number of milligrams of potassium hydroxide required to saponify 1 gram of the sample.

Apparatus

1. Erlenmeyer flasks—alkali resistant, 250 or 300 ml with ground-glass joint
2. Air condensers—minimum 65 cm long, with ground-glass joint to fit Erlenmeyer flasks
3. Distillation flask—2 L with ground-glass joint fitted with water-cooled condenser, for refluxing and distilling 95% ethanol as noted in Reagents 2

Reagents and chemicals

1. Hydrochloric acid, 0.5 N—accurately standardized
2. Phenolphthalein indicator solution—1.0% in 95% ethyl alcohol
3. Toluene—reagent grade
4. Alcoholic potassium hydroxide (KOH)—prepared by placing 5-10 g of reagent-grade KOH pellets in a 2-L flask, adding 1-1.5 L of 95% ethyl alcohol and boiling in a water bath under reflux for 30-60 min. Distill and collect the alcohol in a clean, glass-stopper receiver. Dissolve 40 g of potassium hydroxide in 1 L of the distilled alcohol with cooling to keep the temperature at or below 15 °C

Procedure

1. Heat the sample at 100 °C until liquid and then mix thoroughly.
2. Accurate weight about $2 \text{ g} \pm 0.1 \text{ mg}$ of the melted sample into the Erlenmeyer flask.
3. Add 25 ml of toluene and heat gently to dissolve the sample.
4. Add 50 ml of the alcoholic KOH with a pipet and allow the pipet to drain for a definite period of time.
5. Prepare and conduct blank determinations simultaneously with the sample and similar in all respects, except omitting the fat or oil.
6. Add several boiling chips to the flask and connect the air condenser. Boil gently, but steadily, until the sample is completely saponified. This usually requires about 1 hr for normal sample.
7. After the flask and condenser have cooled somewhat, but not sufficiently to form a gel, wash down the inside of the condenser with a small quantity of distilled water. Disconnect the condenser, add about 1 ml of phenolphthalein indicator and titrate with 0.5 N HCl until the pink color just disappears. Record the volume of 0.5 N HCl required for the titration.

Calculations

$$\text{Saponification value (S.V.), mg KOH/g of sample} = \frac{(B - S) \times N \times 56.1}{W}$$

Where B = ml 0.5 HCl required to titrate blank

S = ml 0.5 HCl required to titrate sample

N = normality of HCl solution

W = weight of sample in grams

Table A3 Saponification Value using AOCS Official Method Cd 3b-76

	1	2	3
Weight of Palm stearin, g	2.0411	2.0089	2.0346
Conc. of HCl, N	0.5	0.5	0.5
Volume of HCl, used in sample, ml	56.20	56.50	56.40
Volume of HCl, used in Blank, ml	70.2	70.2	70.2
Saponification Value (S.V.), Mg KOH/g	192.40	191.29	190.25
Average Saponification Value	191.31		

A3. Calculation of molecular weight of palm stearin

$$\text{M.MW.} = 3(\text{MW.}) + 38$$

$$\text{MW.} = \frac{56108 - (12.67)(\text{S.V.}_{neutral})}{\text{S.V.}_{neutral}}$$

$$\text{S.V.}_{neutral} = \frac{56108 \times \text{S.V.}}{56108 + (12.67)(\text{A.V.})}$$

Where M.MW. = mean molecular weight of triglyceride

MW. = molecular weight of fatty acid in triglyceride

The mean molecular weight of palm stearin maybe calculated:

$$\begin{aligned}
 S.V. \text{ neutral} &= \frac{56108 \times 191.31}{56108 + (12.67)(0.13)} \\
 &= 191.30 \\
 MW. &= \frac{56108 - (12.67)(191.30)}{191.30} \\
 &= 280.63 \\
 M.MW. &= 3(280.63) + 38 \\
 &= 879.89 \text{ g/mole}
 \end{aligned}$$

∴ Molecular weight of palm stearin is 879.89 g/mole

A4. Density of palm stearin

For this study, the density of palm stearin was determined at 60 °C by using pyknometer 10 ml.

Table A4 Density of palm stearin at 60 °C

	1	2	3
Weight of pyknometer, g	17.4725	17.4721	17.4719
Weight of pyknometer + palm stearin, g	26.3394	26.3251	26.3097
Weight of palm stearin, g	8.8669	8.8530	8.8378
Volume of pyknometer, g	10.0000	10.0000	10.0000
Density. g/ml	0.8867	0.8853	0.8838
Average Density	0.8853		

APPENDIX B

CALCULATION

B1. Calculation of the amount of alcohols

The amount of alcohols used in transesterification of palm stearin with various alcohols was 100% excess from its stoichiometric ratio (1:6 of molar ratio). The calculation is based on mole of palm stearin.

$$\text{Amount of alcohol (g)} = \frac{\text{Wt. of palm stearin(g)}}{\text{MW. of palm stearin}} \times 6 \times \text{MW. of alcohol}$$

Where; Wt. of palm stearin = Weight of palm stearin (g)

MW. of palm stearin = Molecular weight of palm stearin (g/mole)

MW. of alcohol = Molecular weight of alcohol (g/mole)

For example: 1-Propanol

The amount of 1-propanol for transesterification with 300 g of palm stearin was calculated follows:

$$\begin{aligned} \text{Wt. of palm stearin} &= 200 \text{ g} \\ \text{MW. of palm stearin} &= 879.89 \text{ g/mole} \\ \text{MW of 1-propanol} &= 60.1 \text{ g/mole} \end{aligned}$$

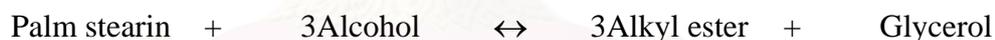
$$\begin{aligned} \therefore \text{Amount of 1-propanol (g)} &= \frac{200(\text{g})}{879.89(\text{g/mole})} \times 6 \times 60.1(\text{g/mole}) \\ &= 82.0 \text{ g} \end{aligned}$$

Table B1 Calculation of the amount of alcohols

Alcohol	Palm stearin (g)	Molecular weight of alcohol (g/mole)	Amount of alcohol (g)
1-Propanol	200 (0.227 mol)	60.1	82.0 (1.367 mol)
Isopropanol	200 (0.227 mol)	60.1	82.0 (1.367 mol)
1-Butanol	200 (0.227 mol)	74.1	101.1 (1.366 mol)
1-Hexanol	200 (0.227 mol)	102.1	139.2 (1.365 mol)
1-Octanol	200 (0.227 mol)	130.2	177.6 (1.366 mol)
2-Ethyl-1-hexanol	200 (0.227 mol)	130.2	177.6 (1.366 mol)

B2. Calculation of the theoretical amount of alkyl esters

The theoretical amount of alkyl ester can determine from mass balance was following as



$$\text{Theoretical amount of alkyl ester} = \frac{\text{Wt. of palm stearin (g)}}{\text{MW. of palm stearin}} \times 3 \times \text{MW. of alkyl ester}$$

Where; wt. of palm stearin = Weight of palm stearin (g)

MW. of palm stearin = Molecular weight of palm stearin (g/mole)

MW. of alkyl ester = Molecular weight of alkyl ester (g/mole)

For example: If 200 g of palm stearin converts to 1-propyl ester completely, the theoretical amount of 1-propylester would be:

$$\begin{aligned} \text{Theoretical amount of alkyl ester} &= \frac{200(\text{g})}{879.89(\text{g/mole})} \times 3 \times 322.63 (\text{g/mole}) \\ &= 220.0 \text{ g} \quad \text{###} \end{aligned}$$

Table B2 Calculation of the theoretical amount of alkyl ester

Alkyl ester	Palm stearin (g)	Molecular weight of alkyl ester (g/mole)	Theoretical amount of alkyl ester (g)
1-Propyl ester	200	322.63	220.0
Isopropyl ester	200	322.63	220.0
1-Butyl ester	200	336.63	229.5
1-Hexyl ester	200	364.63	248.6
1-Octyl ester	200	392.63	267.7
2-Ethyl-1-hexyl ester	200	392.63	267.7

B3. Calculation of the percent of yield of alkyl esters

The percent of yield (% yield) of alkyl ester was defined as:

$$\% \text{ Yield} = \frac{\frac{\text{Mass of alkylester (g)}}{3 \times \text{Molecular weight of alkylester (g/mole)}}}{\frac{\text{Mass of palm stearin (g)}}{\text{Molecular weight of palm stearin (g/mole)}}} \times 100$$

For example: Calculation % yield of 1-propylester Ex.1 which produced from 200 g palm stearin with 1-propanol

Total Weight of 1-propyl ester Ex.1 = 206 g

Molecular weight of 1-propyl ester = 322.63 g/mole

$$\begin{aligned} \text{\% Yield of 1-propylester Ex.1} &= \frac{\frac{206 \text{ (g)}}{3 \times 322.63 \text{ (g/mole)}}}{\frac{200 \text{ (g)}}{879.89 \text{ (g/mole)}}} \times 100 \\ &= 93.64 \text{ \%} \end{aligned}$$

\therefore %Yield of 1-propyl ester Ex.1 was 93.64%

B4. Calculation of viscosity index from kinematic viscosity at 40 and 100 °C, ASTM 2270

Calculate the viscosity index, VI, of the alkyl esters as follows:

$$\text{VI} = \left[\frac{(\text{antilog } N) - 1}{0.00715} \right] + 100$$

$$N = (\log H - \log U) / \log Y$$

Where: H = Kinematic viscosity at 40 °C of alkyl ester of 100 viscosity index having the same kinematic viscosity at 100 °C as the oil whose viscosity index is to be calculated mm²/s (cSt), from Table ASTM D2270 as shown in APPENDIX E

U = Kinematic viscosity at 40 °C of the oil whose viscosity index is to be calculated mm²/s (cSt)

Y = Kinematic viscosity at 100 °C of the oil whose viscosity index is to be calculated mm²/s (cSt)

For example: Calculation viscosity index of 1-propyl ester

Kinematic viscosity at 40 °C of 1-propyl ester = 5.513 cSt

Kinematic viscosity at 100 °C of 1-propyl ester = 2.038 cSt

∴ From Table ASTM 2270 (by interpolation); H = 6.584

$$\begin{aligned} \text{H substituting in: } N &= (\log 6.584 - \log 5.513) / \log 2.038 \\ &= 0.2493 \end{aligned}$$

$$\begin{aligned} \text{N substituting in } VI &= [(\text{antilog } 0.2493) - 1] / 0.00715 + 100 \\ &= 208.44 \approx 208 \end{aligned}$$

∴ Viscosity index of 1-propyl ester is 208 ###

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APPENDIX C

THE RAW DATA OF TRANSESTERIFICATION OF PALM STEARIN

Table C1 The transesterification of palm stearin with various alcohols

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
1-Propyl ester Ex.1	1	200	-	81.9	6	97	3	286	251	34.1
	2	-	220	82.0	6	97	3	301	268	32.7
	3	-	218	81.8	6	97	3	298	267	31.2
	4	-	217	81.9	6	97	3	296	265	30.6
1-Propyl ester Ex.2	1	200	-	82.0	6	97	3	287	252	34.9
	2	-	219	81.9	6	97	3	300	266	33.4
	3	-	218	81.9	6	97	3	298	266	31.7
	4	-	216	81.9	6	97	3	297	266	31.4

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
1-Propyl ester Ex.3	1	200	-	81.8	6	97	3	287	253	33.9
	2	-	221	81.9	6	97	3	299	265	33.5
	3	-	217	81.9	6	97	3	299	267	31.9
	4	-	218	82.0	6	97	3	296	265	30.8
Isopropyl ester Ex.1	1	200	-	81.9	6	82	3	286	265	21.1
	2	-	227	82.0	6	82	3	306	283	22.8
	3	-	216	81.8	6	82	3	301	279	21.9
	4	-	213	81.9	6	82	3	300	280	19.8
Isopropyl ester Ex.2	1	200	-	81.9	6	82	3	287	264	22.9
	2	-	228	81.9	6	82	3	304	284	20.1
	3	-	217	81.9	6	82	3	300	278	20.9
	4	-	214	81.9	6	82	3	299	282	19.9

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
Isopropyl ester Ex.3	1	200	-	82.0	6	82	3	287	264	21.3
	2	-	225	81.9	6	82	3	306	283	22.5
	3	-	217	81.9	6	82	3	302	280	21.9
	4	-	214	81.8	6	82	3	298	278	19.8
1-Butyl ester Ex.1	1	200	-	101.1	6	117	3	308	284	23.7
	2	-	236	101.0	6	117	3	327	303	23.9
	3	-	234	101.2	6	117	3	328	305	22.9
	4	-	229	100.9	6	117	3	324	300	23.1
1-Butyl ester Ex.2	1	200	-	101.1	6	117	3	304	279	24.5
	2	-	234	101.2	6	117	3	328	304	23.8
	3	-	233	101.2	6	117	3	328	305	22.3
	4	-	229	101.2	6	117	3	325	301	23.9

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
1-Butyl ester Ex.3	1	200	-	101.2	6	117	3	306	281	24.6
	2	-	235	101.2	6	117	3	326	303	22.9
	3	-	235	101.1	6	117	3	327	307	19.1
	4	-	228	101.1	6	117	3	323	302	20.9
1-Hexyl ester Ex.1	1	200	-	139.2	6	130	3	342	317	24.5
	2	-	256	139.1	6	130	3	399	372	26.7
	3	-	255	139.1	6	130	3	397	372	24.9
	4	-	258	139.2	6	130	3	401	375	28.2
	5	-	260	139.1	6	130	3	403	378	24.9
1-Hexyl ester Ex.2	1	200	-	139.2	6	130	3	340	315	24.8
	2	-	255	139.2	6	130	3	400	372	27.1
	3	-	257	139.2	6	130	3	399	371	27.9
	4	-	259	139.2	6	130	3	401	376	24.8

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
1-Hexyl ester Ex.2 (cont.)	5	-	259	139.2	6	130	3	400	376	23.4
1-Hexyl ester Ex.3	1	200	-	139.2	6	130	3	341	318	22.9
	2	-	257	139.1	6	130	3	396	374	21.7
	3	-	257	139.0	6	130	3	398	374	27.5
	4	-	260	139.9	6	130	3	402	372	29.1
	5	-	261	139.1	6	130	3	402	378	23.5
1-Octyl ester Ex.1	1	200	-	177.6	6	130	3	380	344	34.5
	2	-	277	177.6	6	130	3	460	426	33.9
	3	-	278	177.4	6	130	3	460	427	32.4
	4	-	280	177.6	6	130	3	461	428	32.9
	5	-	279	177.5	6	130	3	459	428	30.9
	6	-	281	177.5	6	130	3	463	432	32.4

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
1-Octyl ester Ex.2	1	200	-	177.6	6	130	3	379	345	33.4
	2	-	278	177.6	6	130	3	459	425	33.9
	3	-	278	177.6	6	130	3	460	428	31.3
	4	-	279	177.4	6	130	3	459	428	30.6
	5	-	281	177.5	6	130	3	461	427	33.8
	6	-	280	177.6	6	130	3	462	429	32.4
1-Octyl ester Ex.3	1	200	-	177.6	6	130	3	380	344	34.5
	2	-	280	177.6	6	130	3	460	426	33.9
	3	-	281	177.6	6	130	3	460	427	32.4
	4	-	279	177.6	6	130	3	459	426	32.6
	5	-	278	177.4	6	130	3	462	426	33.4
	6	-	278	177.4	6	130	3	462	427	32.1

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
2-Ethyl-1-hexyl ester Ex.1	1	200	-	177.6	6	130	3	379	346	32.5
	2	-	278	177.6	6	130	3	459	427	31.9
	3	-	277	177.6	6	130	3	458	430	27.8
	4	-	279	177.4	6	130	3	458	429	28.3
	5	-	278	177.5	6	130	3	461	431	29.9
	6	-	281	177.6	6	130	3	462	432	29.1
2-Ethyl-1-hexyl ester Ex.2	1	200	-	177.6	6	130	3	380	347	33.1
	2	-	279	177.4	6	130	3	460	428	32.5
	3	-	278	177.6	6	130	3	459	431	28.4
	4	-	280	177.6	6	130	3	459	430	29.0
	5	-	279	177.6	6	130	3	462	432	30.5
	6	-	282	177.5	6	130	3	463	432	29.8

Table C1 The transesterification of palm stearin with various alcohols (continued)

Sample	Step	Reactant				Temp. (°C)	Time (Hrs.)	Product		
		Palm stearin (g)	Alkyl ester (g)	Alcohol (g)	H ₂ SO ₄ (g)			Total (g)	Top phase (g)	Bottom phase (g)
2-Ethyl-1-hexyl ester Ex.3	1	200	-	177.6	6	130	3	381	346	32.9
	2	-	277	177.4	6	130	3	459	429	29.5
	3	-	276	177.5	6	130	3	459	430	28.7
	4	-	280	177.6	6	130	3	463	431	31.1
	5	-	282	177.5	6	130	3	460	428	31.8
	6	-	281	177.6	6	130	3	461	429	31.9

Table C2 Total product of alkyl ester

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
1-Propyl ester Ex.1	1	251	209	34.1	8.9	-	8.9	-
	2	268	208	32.7	4.6	-	13.5	-
	3	267	208	31.2	3.9	-	17.4	-
	4	265	206	30.6	1.2	206	18.6	93.64
1-Propyl ester Ex.2	1	252	210	34.9	9.6	-	9.6	-
	2	266	207	33.4	5.2	-	14.8	-
	3	266	206	31.7	3.6	-	18.4	-
	4	266	205	31.4	-	205	18.4	93.18
1-Propyl ester Ex.3	1	253	212	33.9	8.3	-	8.3	-
	2	265	205	33.5	5.0	-	13.3	-
	3	267	204	31.9	4.1	-	17.4	-
	4	265	204	30.8	0.9	204	18.3	92.73

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
Isopropyl ester Ex.1	1	265	216	21.1	9.9	-	9.9	-
	2	283	212	22.8	6.0	-	15.9	-
	3	279	210	21.9	2.1	-	18.0	-
	4	280	207	19.8	0.9	207	18.9	94.09
Isopropyl ester Ex.2	1	264	215	22.9	9.5	-	9.5	-
	2	264	214	20.1	6.6	-	16.1	-
	3	278	214	20.9	2.3	-	18.4	-
	4	282	205	19.9	-	205	18.4	93.18
Isopropyl ester Ex.3	1	264	214	21.3	9.4	-	9.4	-
	2	283	210	22.5	5.9	-	15.3	-
	3	280	208	21.9	2.6	-	17.9	-
	4	278	207	19.8	1.0	207	18.9	94.09

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
1-Butyl ester Ex.1	1	284	224	23.7	10.2	-	10.2	-
	2	303	220	23.9	5.1	-	15.3	-
	3	305	216	22.9	2.9	-	18.2	-
	4	300	214	23.1	-	214	18.2	93.23
1-Butyl ester Ex.2	1	275	222	24.5	9.5	-	9.5	-
	2	304	218	23.8	4.6	-	14.1	-
	3	305	213	22.3	3.2	-	17.3	-
	4	301	212	23.9	1.1	212	18.4	92.35
1-Butyl ester Ex.3	1	281	223	24.6	10.1	-	10.1	-
	2	303	219	22.9	4.8	-	14.9	-
	3	307	215	19.1	2.0	-	16.9	-
	4	302	214	20.9	1.4	214	18.3	93.23

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
1-Hexyl ester Ex.1	1	317	244	24.5	7.6	-	7.6	-
	2	372	239	26.7	5.0	-	12.6	-
	3	372	236	24.9	3.3	-	15.9	-
	4	375	233	28.2	2.1	-	18.0	-
	5	378	233	24.9	0.6	233	18.6	93.71
1-Hexyl ester Ex.2	1	315	243	24.8	8.2	-	8.2	-
	2	372	238	27.1	4.8	-	13.0	-
	3	371	237	27.9	3.4	-	16.4	-
	4	376	234	24.8	1.9	-	18.3	-
	5	376	233	23.4	-	233	18.3	93.71
1-Hexyl ester Ex.3	1	318	242	22.9	8.4	-	8.4	-
	2	374	240	21.7	5.2	-	13.6	-

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
1-Hexyl ester Ex.3 (cont.)	3	374	236	27.5	3.3	-	16.9	-
	4	372	233	29.1	1.4	-	18.3	-
	5	378	232	23.5	-	232	18.3	93.31
1-Octyl ester Ex.1	1	344	265	34.5	6.9	-	6.9	-
	2	426	259	33.9	4.1	-	11.0	-
	3	427	254	32.4	3.3	-	14.3	-
	4	428	250	32.9	2.2	-	16.5.	-
	5	428	249	30.9	1.7	-	18.2	-
	6	432	248	32.4	-	248	18.2	92.63
1-Octyl ester Ex.2	1	345	266	33.4	6.1	-	6.1	-
	2	425	260	33.9	4.8	-	10.9	-

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
1-Octyl ester Ex.2 (cont.)	3	428	255	31.3	3.5	-	14.4	-
	4	428	253	30.6	2.4	-	16.8	-
	5	427	250	33.8	1.1	-	17.9	-
	6	429	247	32.4	-	247	17.9	92.26
1-Octyl ester Ex.3	1	344	263	34.5	6.7	-	6.7	-
	2	426	257	33.9	5.2	-	11.9	-
	3	427	255	32.4	3.9	-	15.8	-
	4	426	252	32.6	1.9	-	17.7	-
	5	426	248	33.4	0.5	-	18.2	-
	6	427	247	32.1	-	247	18.2	92.26
2-Ethyl-1-hexyl ester Ex.1	1	346	264	32.5	7.1	-	7.1	-
	2	427	261	31.9	5.4	-	12.5	-

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol and catalyst (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
2-Ethyl-1-hexyl ester Ex.1 (cont.)	3	430	258	27.8	3.9	-	16.4	-
	4	429	255	28.3	1.4	-	17.8	-
	5	431	251	29.9	0.5	-	18.3	-
	6	432	249	29.1	-	249	18.3	93.00
2-Ethyl-1-hexyl ester Ex.2	1	347	265	33.1	6.9	-	6.9	-
	2	428	260	32.5	5.3	-	12.2	-
	3	431	257	28.4	3.7	-	15.9	-
	4	430	254	29.0	1.5	-	17.4	-
	5	432	248	30.5	0.6	-	18.0	-
	6	432	248	29.8	-	248	18.0	92.63
2-Ethyl-1-hexyl ester Ex.3	1	346	261	32.9	6.5	-	6.5	-
	2	429	259	29.5	5.1	-	11.6	-

Table C2 Total product of alkyl ester (continued)

Sample	Step	Top phase		Bottom phase		Total product		
		Alkyl esters (g)	Alkyl ester after remove alcohol (g)	Glycerol	Glycerol after remove alcohol and catalyst (g)	Final alkyl ester (g)	Glycerol all steps (g)	% Yield of alkyl esters
2-Ethyl-1-hexyl ester Ex.3 (cont.)	3	430	258	28.9	3.9	-	15.5	-
	4	431	254	31.1	1.7	-	17.2	-
	5	428	249	31.8	0.8	-	18.0	-
	6	425	249	31.9	-	249	18.0	93.00

APPENDIX D

PHYSICAL AND CHEMICAL PROPERTIES OF ALKYL ESTERS

Table D1 The physical and chemical properties of 1-propyl ester

Properties	1-Propyl ester Ex.1	1-Propyl ester Ex.2	1-Propyl ester Ex.3	Average	S.D.
Color, ASTM	L1.5	L1.5	L1.5	L1.5	-
Kinematic viscosity at 40° C, cSt	5.512	5.514	5.513	5.513	0.001
Kinematic viscosity at 100° C, cSt	2.034	2.043	2.037	2.038	0.005
Viscosity index	206	211	208	208	2.52
Pour point, °C	+11	+11	+11	+11	0
Flash point, °C	196	196	196	196	0
API Gravity at (60°F/60°F)	31.37	31.41	31.38	31.39	0.021
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

Table D2 The physical and chemical properties of isopropyl ester

Properties	Isopropyl ester Ex.1	Isopropyl ester Ex.2	Isopropyl ester Ex.3	Average	S.D.
Color, ASTM	L1.5	L1.5	L1.5	L1.5	-
Kinematic viscosity at 40° C, cSt	6.385	6.475	6.382	6.414	0.052
Kinematic viscosity at 100° C, cSt	2.192	2.198	2.175	2.188	0.012
Viscosity index	172	170	171	170	1.000
Pour point, °C	+8	+8	+8	+8	0
Flash point, °C	192	194	194	193	1.155
API Gravity at (60°F/60°F)	31.35	31.24	31.43	31.34	0.095
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

Table D3 The physical and chemical properties of 1-butyl ester

Properties	1-Butyl ester Ex.1	1-Butyl ester Ex.2	1-Butyl ester Ex.3	Average	S.D.
Color, ASTM	L3.0	L3.0	L3.0	L3.0	-
Kinematic viscosity at 40° C, cSt	6.595	6.403	6.528	6.509	0.097
Kinematic viscosity at 100° C, cSt	2.352	2.287	2.333	2.324	0.033
Viscosity index	214	209	214	212	2.887
Pour point, °C	+10	+9	+9	+9	0.577
Flash point, °C	202	202	202	202	0
API Gravity at (60°F/60°F)	31.21	31.31	31.18	31.23	0.608
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

Table D4 The physical and chemical properties of 1-hexyl ester

Properties	1-Hexyl ester Ex.1	1-Hexyl ester Ex.2	1-Hexyl ester Ex.3	Average	S.D.
Color, ASTM	L3.0	L3.0	L3.0	L3.0	-
Kinematic viscosity at 40° C, cSt	7.840	7.851	7.835	7.842	0.008
Kinematic viscosity at 100° C, cSt	2.616	2.618	2.613	2.616	0.003
Viscosity index	195	196	196	196	0.577
Pour point, °C	+8	+8	+8	+8	0
Flash point, °C	211	210	209	210	1
API Gravity at (60°F/60°F)	31.19	31.18	31.19	31.19	0.006
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

Table D5 The physical and chemical properties of 1-octyl ester

Properties	1-Octyl ester Ex.1	1-Octyl ester Ex.2	1-Octyl ester Ex.3	Average	S.D.
Color, ASTM	L4.0	L4.0	L4.0	L4.0	-
Kinematic viscosity at 40° C, cSt	8.152	8.142	8.149	8.148	0.005
Kinematic viscosity at 100° C, cSt	2.721	2.705	2.713	2.713	0.008
Viscosity index	204	202	202	202	1.155
Pour point, °C	+5	+5	+5	+5	0
Flash point, °C	240	240	240	240	0
API Gravity at (60°F/60°F)	31.14	31.15	31.13	31.14	0.01
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

Table D6 The physical and chemical properties of 2-ethyl-1-hexyl ester

Properties	2-Ethyl-1-hexyl ester Ex.1	2-Ethyl-1-hexyl ester Ex.2	2-Ethyl-1-hexyl ester Ex.3	Average	S.D.
Color, ASTM	L4.0	L4.0	L4.0	L4.0	-
Kinematic viscosity at 40° C, cSt	8.652	8.553	8.797	8.667	0.123
Kinematic viscosity at 100° C, cSt	2.748	2.763	2.834	2.782	0.046
Viscosity index	180	190	193	188	6.806
Pour point, °C	-5	-5	-5	-5	0
Flash point, °C	236	236	236	236	0
API Gravity at (60°F/60°F)	31.11	31.09	31.13	31.11	0.02
Appearance	Bright & clear	Bright & clear	Bright & clear	Bright & clear	-
Copper Strip Corrosion	1a	1a	1a	1a	-

APPENDIX E

TABLE FOR VISCOSITY INDEX CALCULATION

Table E1. Basic values for L and H for kinematic viscosity in 40 °C and 100 °C systems

K.V. @100°C (cSt)	L	H	K.V. @100°C (cSt)	L	H	K.V. @100°C (cSt)	L	H
2.00	7.994	6.394	7.00	78.00	48.57	12.0	201.9	108.0
2.10	8.640	6.894	7.10	80.25	49.61	12.1	204.8	109.4
2.20	9.309	7.410	7.20	82.39	50.69	12.2	207.8	110.7
2.30	10.00	7.944	7.30	84.53	51.78	12.3	210.7	112.0
2.40	10.71	8.496	7.40	86.66	52.88	12.4	213.6	113.3
2.50	11.45	9.063	7.50	88.85	53.98	12.5	216.6	114.7
2.60	12.21	9.647	7.60	91.04	55.09	12.6	219.6	116.0
2.70	13.00	10.25	7.70	93.20	56.20	12.7	222.6	117.4
2.80	13.80	10.87	7.80	95.43	57.31	12.8	225.7	118.7
2.90	14.63	11.50	7.90	97.72	58.45	12.9	228.8	120.1
3.00	15.49	12.15	8.00	100.0	59.60	13.0	231.9	121.5
3.10	16.36	12.82	8.10	102.3	60.74	13.1	235.0	122.9
3.20	17.26	13.51	8.20	104.6	61.89	13.2	238.1	124.2
3.30	18.18	14.21	8.30	106.9	63.05	13.3	241.2	125.6
3.40	19.12	14.93	8.40	109.2	64.18	13.4	244.3	127.0
3.50	20.09	15.66	8.50	111.5	65.32	13.5	247.4	128.4
3.60	21.08	16.42	8.60	113.9	66.48	13.6	250.6	129.8
3.70	22.09	17.19	8.70	116.2	67.64	13.7	253.8	131.2
3.80	23.13	17.97	8.80	118.5	68.79	13.8	257.0	132.6
3.90	24.19	18.77	8.90	120.9	69.94	13.9	260.1	134.0
4.00	25.32	19.56	9.00	123.3	71.10	14.0	263.3	135.4
4.10	26.50	20.37	9.10	125.7	72.27	14.1	266.6	136.8
4.20	27.75	21.21	9.20	128.0	73.42	14.2	269.8	138.2
4.30	29.07	22.05	9.30	130.4	74.57	14.3	273.0	139.6
4.40	30.48	22.92	9.40	132.8	75.73	14.4	276.3	141.0
4.50	31.96	23.81	9.50	135.3	76.91	14.5	279.6	142.4
4.60	33.52	24.71	9.60	137.7	78.08	14.6	283.0	143.9
4.70	35.13	25.63	9.70	140.1	79.27	14.7	286.4	145.3
4.80	36.79	26.57	9.80	142.7	80.46	14.8	289.7	146.8
4.90	38.50	27.53	9.90	145.2	81.67	14.9	293.0	148.2
5.00	40.23	28.49	10.0	147.7	82.87	15.0	296.5	149.7
5.10	41.99	29.46	10.1	150.3	84.08	15.1	300.0	151.2
5.20	43.76	30.43	10.2	152.9	85.30	15.2	303.4	152.6
5.30	45.53	31.40	10.3	155.4	86.51	15.3	306.9	154.1
5.40	47.31	32.37	10.4	158.0	87.72	15.4	310.3	155.6
5.50	49.90	33.34	10.5	160.6	88.95	15.5	313.9	157.0
5.60	50.87	34.32	10.6	163.2	90.19	15.6	317.5	158.6
5.70	52.64	35.29	10.7	165.8	91.40	15.7	321.1	160.1
5.80	54.42	36.26	10.8	168.5	92.65	15.8	324.6	161.6
5.90	56.20	37.23	10.9	171.2	93.92	15.9	328.3	163.1
6.00	57.97	38.19	11.0	173.9	95.19	16.0	331.9	164.6
6.10	59.74	39.17	11.1	176.6	96.45	16.1	335.5	166.1
6.20	61.52	40.15	11.2	179.4	97.71	16.2	339.2	167.7
6.30	63.32	41.13	11.3	182.1	98.97	16.3	342.9	169.2
6.40	65.18	42.14	11.4	184.9	100.2	16.4	346.6	170.7
6.50	67.12	43.18	11.5	187.6	101.5	16.5	350.3	172.3
6.60	69.16	44.24	11.6	190.4	102.8	16.6	354.1	173.8
6.70	71.29	45.33	11.7	193.3	104.1	16.7	358.0	175.4
6.80	73.48	46.44	11.8	196.2	105.4	16.8	361.7	177.0
6.90	75.72	47.51	11.9	199.0	106.7	16.9	365.6	178.6

Table E1. Basic values for L and H for kinematic viscosity in 40 °C and 100 °C systems (continued)

K.V. @100°C (cSt)	L	H	K.V. @100°C (cSt)	L	H	K.V. @100°C (cSt)	L	H
17.0	369.4	180.2	24.0	683.9	301.8	42.5	1935	714.9
17.1	373.3	181.7	24.2	694.5	305.6	43.0	1978	728.2
17.2	377.1	183.3	24.4	704.2	309.4	43.5	2021	741.3
17.3	381.0	184.9	24.6	714.9	313.0	44.0	2064	754.4
17.4	384.9	186.5	24.8	725.7	317.0	44.5	2108	767.6
17.5	388.9	188.1	25.0	736.5	320.9	45.0	2152	780.9
17.6	392.7	189.7	25.2	747.2	324.9	45.5	2197	794.5
17.7	396.7	191.3	25.4	758.2	328.8	46.0	2243	808.2
17.8	400.7	192.9	25.6	769.3	332.7	46.5	2288	821.9
17.9	404.6	194.6	25.8	779.7	336.7	47.0	2333	835.5
18.0	408.6	196.2	26.0	790.4	340.5	47.5	2380	849.2
18.1	412.6	197.8	26.2	801.6	344.4	48.0	2426	863.0
18.2	416.7	199.4	26.4	812.8	348.4	48.5	2473	876.9
18.3	420.7	201.0	26.6	824.1	352.3	49.0	2521	890.9
18.4	424.9	202.6	26.8	835.5	356.4	49.5	2570	905.3
18.5	429.0	204.3	27.0	847.0	360.5	50.0	2618	919.6
18.6	433.2	205.9	27.2	857.5	364.6	50.5	2667	933.6
18.7	437.3	207.6	27.4	869.0	368.3	51.0	2717	948.2
18.8	441.5	209.3	27.6	880.6	372.3	51.5	2767	962.9
18.9	445.7	211.0	27.8	892.3	376.4	52.0	2817	977.5
19.0	449.9	212.7	28.0	904.1	380.6	52.5	2867	992.1
19.1	454.2	214.4	28.2	915.8	384.6	53.0	2918	1007
19.2	458.4	216.1	28.4	927.6	388.8	53.5	2969	1021
19.3	462.7	217.7	28.6	938.6	393.0	54.0	3020	1036
19.4	467.0	219.4	28.8	951.2	396.6	54.5	3073	1051
19.5	471.3	221.1	29.0	963.4	401.1	55.0	3126	1066
19.6	475.7	222.8	29.2	975.4	405.3	55.5	3180	1082
19.7	479.7	224.5	29.4	987.1	409.5	56.0	3233	1097
19.8	483.9	226.2	29.6	998.9	413.5	56.5	3286	1112
19.9	488.6	227.7	29.8	1011	417.6	57.0	3340	1127
20.0	493.2	229.5	30.0	1023	421.7	57.5	3396	1143
20.2	501.5	233.0	30.5	1055	432.4	58.0	3452	1159
20.4	510.8	236.4	31.0	1086	443.2	58.5	3507	1175
20.6	519.9	240.1	31.5	1119	454.0	59.0	3563	1190
20.8	528.8	243.5	32.0	1151	464.9	59.5	3619	1206
21.0	538.4	247.1	32.5	1184	475.9	60.0	3676	1222
21.2	547.5	250.7	33.0	1217	487.0	60.5	3734	1238
21.4	556.7	254.2	33.5	1251	498.1	61.0	3792	1254
21.6	566.4	257.8	34.0	1286	509.6	61.5	3850	1270
21.8	575.6	261.5	34.5	1321	521.1	62.0	3908	1286
22.0	585.2	264.9	35.0	1356	532.5	62.5	3966	1303
22.2	595.0	268.6	35.5	1391	544.0	63.0	4026	1319
22.4	604.3	272.3	36.0	1427	555.6	63.5	4087	1336
22.6	614.2	275.8	36.5	1464	567.1	64.0	4147	1352
22.8	624.1	279.6	37.0	1501	579.3	64.5	4207	1369
23.0	633.6	283.3	37.5	1538	591.3	65.0	4268	1386
23.2	643.4	286.8	38.0	1575	603.1	65.5	4329	1402
23.4	653.8	290.5	38.5	1613	615.0	66.0	4392	1419
23.6	663.3	294.4	39.0	1651	627.1	66.5	4455	1436
23.8	673.7	297.9	39.5	1691	639.2	67.0	4517	1454
			40.0	1730	651.8	67.5	4580	1471
			40.5	1770	664.2	68.0	4645	1488
			41.0	1810	676.6	68.5	4709	1506
			41.5	1851	689.1	69.0	4773	1523
			42.0	1892	701.9	69.5	4839	1541
						70.0	4905	1558

APPENDIX F

CERTIFICATE OF MINERAL BASE OIL 150 SN

Analysis	Method	Units	Specification	Result
Specific Gravity @ 60/60 °F	ASTM D-1298 Equivalent to D-4052	-	Report	0.8710
Kinematic Viscosity @ 40 °C	ASTM D-445	cSt	29.0-31.0	30.30
Kinematic Viscosity @ 100 °C	ASTM D-445	cSt	-	5.245
Viscosity Index	ASTM D-2270	-	Min. 100	104
Pour Point	ASTM D-97	° C	Max. -9	-9
Total Sulfur Content	ASTM D-4294	Mass%	-	0.381
ASTM Colour	ASTM D-1500	-	Max. 1.0	1.0.5
Acid Number	ASTM D-664	Mg KOH/g	Max. 0.05	0.02
Flash Point (COC)	ASTM D-92	° C	Report	234
Flash Point (PMCC)	ASTM D-93	° C	Min. 204	211.0
Carbon Residue, Micro Method	ASTM D-4530	% (m/m)	Max. 0.05	0.01
Water Content	ASTM D-95	% vol.	Nil	0.00
Appearance	Visual	-	Bright & Clear	B & C
Copper Strip Corrosion @ 100 °C for 3 Hrs.	ASTM D-130	-	Max. 1b	1a
Evaporation Loss (NOACK)	DIN 51581	% wt.	Max. 16	9.8
Water Separability (Demulsibility @ 54 °C)	ASTM D-1401	min	Max. 20	40-40-0(5)
Ash Content	ASTM D-482	Mass%	Max. 0.01	0.000

1. This report is certified only the tested sample.
Copy of some part of this report without permission from Laboratory Manager is not allowed.

2. ◆: Supplement Data

Remark: **PASSED**

Density @ 15 °C = 0.8704 kg/l

FR-INS-13-00-01

Rev. 8 Effective Date 1 มิถุนายน 2550

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APPENDIX G

MATERIAL SAFETY DATA SHEETS

G1. 1-Propanol

General

Synonyms	:	n-Propanol, Propan-1-ol, n-Propyl alcohol
Molecular formula	:	C ₃ H ₈ O
Molecular weight	:	60.10

Physical data

Form	:	liquid
Color	:	colorless
Odor	:	alcohol-like
Boiling point	:	97 °C
Melting point	:	-127 °C
Ignition temperature	:	405 °C
Flash point	:	22 °C [method: close cup]
Explosion limits	:	2.1 – 13.7 vol.%
Vapor pressure	:	14.9 mmHg @20 °C
Density	:	0.80 g/cm ³
Solubility in water	:	soluble @20 °C

Stability and reactivity

Conditions to be avoided: strong heating

Substances to be avoided: alkali metals, alkaline earth metals, strong oxidizing agents

Hazardous decomposition products: carbon monoxide, carbon dioxide

Further information: Explosive with air in a vapor/gas state

Hazards identification

Highly flammable, Risk of serious damage to eyes, Vapors may cause drowsiness and dizziness

Toxicology

Inhalation: Irritation symptoms in the respiratory tract

Skin contact: Irritant effect

Eye contact: Irritant effect

Swallowing: Rapid absorption; headache, dizziness, inebriation

Uptake of large quantities: respiratory paralysis, coma

First aid measure

After inhalation: If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen

After skin contact: In case of contact, immediately wash skin with soap and copious amount of water.

After eye contact: In case of contact, immediately flush eyes with copious amount of water for at least 15 minutes.

After ingestion: If swallowed, wash out mouth with water provided person is conscious. Call a physician

Personal Protective equipment

Respiratory protection: government approved respirator

Eye protection: chemical safety goggles

Hand protection: compatible chemical-resistant gloves

Industrial hygiene: Immediately change contaminated clothing. Application of skin-protective barrier cream recommended. Should be wash hands and face after working with substance.

G2. Isopropanol

General

Synonyms : 2-Propyl alcohol, Isopropanol (IPA), i-Propanol,
Isopropyl alcohol, n-Propan-2-ol

Molecular formula : C_3H_8O

Molecular weight : 60.10

Physical data

Form : liquid

Color : colorless

Odor : characteristic

Boiling point : $82^{\circ}C$

Melting point : $-89.5^{\circ}C$

Auto-ignition temp	:	399° C
Flash point	:	12° C
Explosion limits	:	2.5 - 12 vol. %
Vapor pressure	:	33 mmHg @20° C
Density	:	0.785 g/cm ³
Solubility in water	:	soluble @20° C

Stability and reactivity

Conditions to be avoided: strong heating,

Substances to be avoided: alkali metals, alkaline earth metals, aluminum, oxidizing agents, organic nitro compounds

Hazardous decomposition products: carbon monoxide, carbon dioxide

Further information: highly flammable; hygroscopic, explosive with air in a vapor/gas state

Hazards identification

Highly flammable, Irritating to eyes, Vapors may cause drowsiness and dizziness

Toxicology

Inhalation: Irritation symptoms in the respiratory tract

Skin contact: Degreasing effect on the skin, possibly followed by secondary inflammation

Eye contact: Irritation

Swallowing: After accident swallowing the substance may pose a risk of aspiration. Passage into the lung can result in a condition resembling pneumonia

Absorption: Headache, dizziness, inebriation, unconsciousness, narcosis

Uptake of large quantities: Respiratory paralysis, coma

First aid measure

After inhalation: If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen.

After skin contact: In case of contact, immediately wash skin with soap and copious amount of water.

After eye contact: In case of contact, immediately flush eyes with copious amount of water for at least 15 minutes.

After ingestion: If swallowed, wash out mouth with water provided person is conscious. Call a physician.

Personal Protective

Personal protective equipment: Protective clothing should be selected specifically for the working place, depending on concentration and quantity of the hazardous substance handled. The resistance of the protective clothing to chemicals should be ascertained with the respective supplier.

Respiratory protection: government approved respirator

Eye protection: chemical safety goggles

Hand protection: compatible chemical-resistant gloves

Industrial hygiene: Immediately change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working substance.

G3. 1-Butanol

General

Synonyms : 1-Butyl alcohol, n-Butyl alcohol, Butan-1-ol, n-Butan-1-ol, n-Butanol

Molecular formula : $C_4H_{10}O$

Molecular weight : 74.12

Physical data

Form : liquid

Color : colorless

Odor : ethanolic

Boiling point : 117.7 °C

Melting point : -90.0 °C

Auto-ignition temp : 343 °C

Flash point : 35 °C

Explosion limits : 1.4 – 11.2 vol.%

Vapor pressure : 4 mmHg @20° C

Density : 0.811 g/cm³

Solubility in water : soluble @20° C

Stability and reactivity

Conditions to be avoided: heating, moisture

Substances to be avoided: alkali metals, alkaline earth metals, aluminum, strong oxidizing agents, halogens

Hazardous decomposition products: carbon monoxide, carbon dioxide

Further information: inflammable, unsuitable working material; various plastic & rubber

Hazards identification

Flammable, Harmful if swallowed, Irritation to respiratory system and skin, Risk of serious damage to eyes, Vapors may cause drowsiness and dizziness

Toxicology

Inhalation: May be harmful if inhaled.

Skin contact: Degreasing effect on the skin, possibly followed by secondary inflammation

Eye contact: Irritation

Swallowing: After accident swallowing the substance may pose a risk of aspiration. Passage into the lung can result in a condition resembling pneumonia

Absorption: Headache, dizziness, inebriation, unconsciousness, narcosis

Uptake of large quantities: Respiratory paralysis, coma

First aid measure

After inhalation: If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen

After skin contact: In case of contact, immediately wash skin with soap and copious amount of water.

After eye contact: In case of contact, immediately flush eyes with copious amount of water for at least 15 minutes.

After ingestion: If swallowed, wash out mouth with water provided person is conscious. Call a physician.

Personal Protective

Personal protective equipment: Protective clothing should be selected specifically for the working place, depending on concentration and quantity of the hazardous substance handled. The resistance of the protective clothing to chemicals should be ascertained with the respective supplier.

Respiratory protection: government approved respirator

Eye protection: chemical safety goggles

Hand protection: compatible chemical-resistant gloves

Industrial hygiene: Immediately change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working

G4. 1-Hexanol

General

Synonyms : n-Hexyl alcohol

Molecular formula : $C_6H_{14}O$

Molecular weight : 102.18

Physical data

Form	:	liquid
Color	:	colorless
Odor	:	characteristic
Boiling point	:	157 °C
Melting point	:	-45 °C
Auto-ignition temp	:	285 °C
Flash point	:	58 °C
Explosion limits	:	1.2-7.7 vol. %
Vapor pressure	:	1 hPa @20° C
Density	:	0.82 g/cm ³
Solubility in water	:	5.8 g/l @20° C

Stability and reactivity

Conditions to be avoided: no information available

Substances to be avoided: oxidizing agent

Hazardous decomposition products: no information available

Toxicology

Skin contact: Irritations

Eye contact: Irritations

Swallowing: irritations of mucous membranes in the mouth, pharynx, esophagus, and gastrointestinal tract: After inhalation: Irritations of the mucous membranes and coughing. After absorption of large quantities: narcosis

First aid measure

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water for at least 10 minutes with the eyelid held wide open. Summon eye specialist.

After swallowing: make the victim drink plenty of water, induce vomiting. Summon doctor.

Personal Protective

Respiratory protection: required when vapors/aerosols are generated. Filter A (acc. to DIN 3181) for vapor of organic compounds

Eye protection: required

Hand protection: required

Industrial hygiene: Change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working with substance.

G5. 1-Octanol

General

Synonyms : n-Octyl alcohol

Molecular formula : $C_8H_{18}O$

Molecular weight : 130.23

Physical data

Form	:	liquid
Color	:	colorless
Odor	:	characteristic
Boiling point	:	~ 195 °C
Melting point	:	-16 °C
Auto-ignition temp	:	270 °C
Flash point	:	81 °C
Explosion limits	:	0.8 vol.%
Vapor pressure	:	0.3 mbar @20° C
Density	:	0.83 g/cm ³
Solubility in water	:	almost insoluble

Stability and reactivity

Conditions to be avoided: no information available

Substances to be avoided: no information available

Hazardous decomposition products: no information available

Further information: unsuitable working materials: various plastics, rubber

Toxicology

Skin contact: Irritations.

Eye contact: Irritations.

Inhalation: Irritations of the mucous membranes, coughing, and sdyspnoea.

Swallowing: irritations of mucous membranes in the mouth, pharynx, esophagus and gastrointestinal tract, nausea and vomiting

Absorption: CNS disorders, Drowsiness, dizziness, euphoria, excitation, spasms, in certain circumstances narcosis

First aid measure

After inhalation: fresh air.

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water with the eyelid held wide open. Summon eye specialist.

After swallowing: make victim drink plenty of water, induce vomiting, summon doctor. Keep airways free. Risk of aspiration! Laxative: Sodium sulfate (1 tablespoon/1/4 l water). No milk. No castor oil.

Personal Protective

Respiratory protection: required when vapors/aerosols are generated. Filter A (acc. to DIN 3181) for vapors of organic compounds

Eye protection: required

Hand protection: required

Industrial hygiene: Immediately change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working with substance.

G6. 2-Ethyl-1-hexanol

General

Synonyms : iso-Octanol, iso-Octylalcohol

Molecular formula : $C_8H_{18}O$

Molecular weight : 130.23

Physical data

Form : liquid

Color : colorless

Odor : aromatic

Boiling point : 185 °C

Melting point : -76 °C

Auto-ignition temp : 250 °C

Flash point : 81 °C

Explosion limits : 1.8 – 12.4 vol. %

Vapor pressure : 0.5 hPa @ 20° C

Density : 0.83 g/cm³

Solubility in water : 1.4 g/l @20° C

Stability and reactivity

Conditions to be avoided: Strong heating.

Substances to be avoided: strong oxidizing agents, strong acids

Hazardous decomposition products: no information available

Further information: combustible, incompatible with various plastics, Exposable with air in a vaporous/gaseous state.

Toxicology

Inhalation: Irritation symptoms in the respiratory tract. Risk of absorption

Skin contact: Irritations. Danger with skin absorption, Degreasing effect on the skin, possibly followed by secondary inflammation

Long-term exposure: dermatitis

Eye contact: Irritation

Swallowing: irritations of mucous membranes in the mouth, pharynx, esophagus and gastrointestinal tract, Nausea and vomiting

Systemic effects: After absorption of large quantities: Drowsiness, dizziness, euphoria, excitation, spasms, in certain circumstances narcosis.

First aid measure

After inhalation: fresh air.

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water with the eyelid held wide open. Summon eye specialist.

After swallowing: make victim drink plenty of water, induce vomiting, summon doctor.

Personal Protective

Respiratory protection: required when vapors are generated.

Eye protection: required

Hand protection: required

Industrial hygiene: Change contaminated clothing. Application of skin-protective barrier cream recommended. Wash hands after working with substance.

G7. Sulfuric acid

General

Synonyms : Battery acid, Electrolyte acid

Molecular formula : H_2SO_4

Molecular weight : 98.08

Physical data

Form : liquid

Color : colorless

Odor : odorless

Boiling point : 330 °C

Melting point : -15 °C

Auto-ignition temp : not available

Flash point : not available

Explosion limits : not available

Vapor pressure : ~ 0.0001 mbar @20° C

Density : 1.84 g/cm³

Solubility in water : soluble @20° C

Stability and reactivity

Conditions to be avoided: Strong heating.

Substances to be avoided: water , alkali metals , alkali compounds , ammonia , alkaline earth metals , alkalis , acids , alkaline earth compounds , metals , metal alloys , phosphorus oxides , phosphorus , hydrides , halogen-halogen compounds , oxyhalogenic compounds , permanganates , nitrates , carbides , combustible substances , organic solvents , acetylidene , nitrides , organic nitro compounds , anilines , peroxides , picrates , nitrides , lithium silicate

Hazardous decomposition products: in the event of fire: sulfur oxides

Further information: hygroscopic, has a corrosive effect, incompatible with metals, animal/vegetable tissues

Toxicology

Inhalation: damage to the affected mucous membranes.

Skin contact: severe burns with formation of scabs.

Eye contact: burns, corneal lesions.

Swallowing: severe pain (risk of perforation), nausea, vomiting and diarrhea, after a latency period of several weeks possibly pyloric stenosis

First aid measure

After inhalation: fresh air. Summon doctor.

After skin contact: wash off with plenty of water. Dab with polyethylene glycol 400. Immediately remove contaminated clothing.

After eye contact: rinse out with plenty of water for at least 10 minutes with the eyelid held wide open. Immediately summon eye specialist.

After swallowing: make victim drink plenty of water (if necessary several liters), avoid vomiting (risk of perforation!). Immediately summon doctor. Do not attempt to neutralize.

Personal Protective

Protective clothing should be selected specifically for the working place, depending on concentration and quantity of the hazardous substances handled. The resistance of the protective clothing to chemicals should be ascertained with the respective supplier.

Respiratory protection: required when vapors/aerosols are generated.

Eye protection: required

Hand protection: required

Other protective equipment: Acid-resistant protective clothing.

Industrial hygiene: Change contaminated clothing and immerse in water. Apply skin-protective barrier cream. Wash hands and face after working with substance.

G8. Toluene

General

Synonyms : Toluene, Methylbenzene

Molecular formula : C₇H₈

Molecular weight : 92.14

Physical data

Form : liquid

Color : colorless

Odor : characteristic

Boiling point : 111 °C

Melting point : -95 °C

Auto-ignition temp : 535 °C

Flash point : 6 °C

Explosion limits : 1.2-7.0 vol. %

Vapor pressure : 29 mbar @ 20° C

Density : 0.87 g/cm³

Solubility in water : 0.5 g/l @20° C

Stability and reactivity

Conditions to be avoided: Strong heating.

Substances to be avoided: halogen-halogen compounds, nitric acid, nitrogen oxides, organic nitro compounds, oxidizing agent, uranium hexafluoride, sulfur / heat

Hazardous decomposition products: no information available

Further information: highly inflammable; unsuitable working materials: various plastics, rubber, Exposable with air in a vaporous/gaseous state.

Toxicology

Inhalation: Irritation symptoms in the respiratory tract.

Skin contact: After long-term exposure to the chemical: dermatitis Degreasing effect on the skin, possibly followed by secondary inflammation.

Eye contact: Irritations. Mucosal irritations

Swallowing causes nausea and vomiting. Risk of aspiration upon vomiting

Systemic effects: After absorption of large quantities: CNS disorders, inebriation, spasms, unconsciousness, respiratory arrest, cardiovascular failure

First aid measure

After inhalation: fresh air. Keep airways free.

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water for at least 10 minutes with the eyelid held wide open. Summon eye specialist.

After swallowing: Paraffin oil (3 ml/kg). Sodium sulfate (1tablespoon/1/4 l water), Summon doctor, No castor oil. No milk. No alcohol.

Personal Protective

Respiratory protection: required when vapors/aerosols are generated.

Eye protection: required

Hand protection: required

Industrial hygiene: Change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working with substance.

G9. Ethanol

General

Synonyms : Ethyl alcohol

Molecular formula : C_2H_5OH

Molecular weight : 46.07

Physical data

Form : liquid

Color : colorless

Odor : characteristic

Boiling point : 78 °C

Melting point : -117 °C

Auto-ignition temp : 425 °C

Flash point : 12 °C

Explosion limits : 3.5-15 vol. %

Vapor pressure : ~ 59 mbar @20° C

Density : 0.81 g/cm³

Solubility in water : soluble @20° C

Stability and reactivity

Conditions to be avoided: Strong heating, Exposable with air in a vaporous/gaseous state.

Substances to be avoided: alkali metals, alkaline earth metals, alkali oxide, strong oxidizing agents

Hazardous decomposition products: no information available

Further information inflammable

Toxicology

Inhalation: slight mucosal irritations Risk of absorption.

Eye contact: Slight irritations.

Swallowing: nausea and vomiting

Systemic effects: euphoria

Absorption: dizziness, inebriation, narcosis, respiratory paralysis

First aid measure

Suitable extinguishing media: Water, CO₂, foam, powder.

Special risks: combustible, vapors heavier than air, formation of exposable mixtures possible with air, take measures to prevent electrostatic charging

Special protective equipment for firefighting: When large amounts of substance are released, work only with self-enclosed respirators and suitable chemical-resistant protective clothing.

Other information: Cool container with spray water from a safe distance.

Personal Protective

Respiratory protection: required when vapors/aerosols are generated. Filter A (acc. to DIN 3181) for vapors of organic compounds

Eye protection: required

Hand protection: required

Industrial hygiene: Change contaminated clothing. Wash hands after working with substance.

G10. Potassium hydroxide

General

Synonyms : caustic potash, potassium hydrate, potassium oxide hydrate

Molecular formula : KOH

Molecular weight : 56.11

Physical data

Form : solid

Color : white

Odor : odorless

Boiling point : 1320 °C

Melting point : 360 °C

Auto-ignition temp : not available

Flash point : not available

Explosion limits : not available

Density : 2.04 g/cm³

Solubility in water : ~1100 g/l @20° C

Stability and reactivity

Conditions to be avoided: no information available

Substances to be avoided: metals (in the presence of atmospheric oxygen and/or moisture), light metals: Formation of: hydrogen; ammonium compounds: Formation of: ammonia; alkaline earth metals, halogens, halogen-halogen compounds, halogenated hydrocarbons, nonmetallic oxyhalides, halogen oxides, organic nitro compounds, phosphorus, nonmetallic oxides, hydrocarbons, anhydrides, strong acids

Hazardous decomposition products: no information available

Further information: extremely hygroscopic; exothermic dissolution process with water incompatible with metals, various plastics, glass, animal/vegetable tissues

Toxicology

Contact with substance: Burns of skin, mucous membranes and eyes (Risk of corneal clouding)

Swallowing: Burns of: esophagus, stomach (strong pain (risk of perforation)), dizziness

First aid measure

After inhalation: fresh air. Summon doctor.

After skin contact: wash off with plenty of water. Dab with polyethylene glycol 400. Immediately remove contaminated clothing.

After eye contact: rinse out with plenty of water for at least 10 minutes with the eyelid held wide open. Immediately summon eye specialist.

After swallowing: make victim drink plenty of water (if necessary several liters), avoid vomiting (risk of perforation!). Immediately summon doctor. Do not attempt to neutralize.

Personal Protective

Respiratory protection: required when dusts are generated.

Eye protection: required

Hand protection: required

Industrial hygiene: Immediately change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working with substance.



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VITA

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