

# CHAPTER 1

## INTRODUCTION

### 1.1 Background

The majority of the world energy needs have been supplied from petroleum resources. While petroleum resources have been an efficient energy provider, their usage has caused pollution problems that warrant the need to seek for an efficient and cleaner energy sources.

This and the concern on depleting petroleum resources have driven the scientific community worldwide and industrial researches to explore other energy potentials that satisfy both the energy and environmental demands. One of the feasible energy sources is the use of plant oils, which is readily available and environmentally acceptable [1-3].

Biodiesel is an alternative diesel fuel consisting of alkyl monoesters of fatty acids from vegetable oils (VOs) or animal fats. However, there have been many problems associated with the direct use of VOs in diesel engines. Problems, such as oil ring sticking and gelling of the engine lubricant oil, appeared in testing the VOs for longer periods of time due to the high viscosities and lower volatilities of VOs [2, 4, 5].

The utilization of bio-renewable resources has played a significant part in the advances of human societies. Before that, cultivated grains, grasses, and fermented sugars were used to provide power and as transportation fuels [6]. The history of using bio-renewable resources such as VOs as alternative diesel fuel has begun since 1900s with the invention of diesel engine by Rudolph Diesel, when the original diesel engine has been designed to run on peanut oil [7-9].

Table 1.1 Biodiesel and diesel ASTM standards [10]

Property	Diesel	Methyl ester (biodiesel)
Standard	ASTM D975	ASTM D6751
Composition	HC <sup>a</sup> (C10-C21)	FAME <sup>b</sup> (C12-C22)
Kinematic viscosity (mm <sup>2</sup> /s) at 40 °C	1.9-4.1	1.9-6.0
Specific gravity (g/mL)	0.85	0.88
Flash point (°C)	60-80	100-170
Cloud point (°C)	-15 to 5	-3 to 12
Pour point (°C)	-35 to -15	-15 to 16
Water, % vol.	0.05	0.05
Carbon, wt. %	87	77
Hydrogen, wt. %	13	12
Oxygen, wt. %	0	11
Sulfur, wt. %	0.05	0.05
Cetane number	40-55	48-60
HFRR <sup>c</sup> , microns	685	314
BOCLE <sup>d</sup> scuff, (g)	3,600	>7,000

<sup>a</sup>Hydrocarbons, <sup>b</sup>Fatty Acid Methyl Esters, <sup>c</sup>High Frequency Reciprocating Rig, <sup>d</sup>Ball-on-Cylinder Lubricity Evaluator

Although the use of VOs as engine fuels seems insignificant at that time, Rudolph put a future idea that in the course of time, biodiesel will be as important as petroleum as it holds promising alternative as diesel engine fuel [2]. Since 1900s a great deal of research had taken place and biodiesel was going to become of great importance for its advantages, which are over the conventional diesel fuel, while their properties being quite similar.

Table 1.1 showed that the properties of biodiesel match well with that of standard diesel specifications. However, biodiesel is superior to diesel for its renewability, high oxygen content, biodegradability and null greenhouse effect given the photosynthetic origin of the lipid feedstocks. Furthermore, biodiesel shows a higher

lubricity which extends an engine's lifetime and makes less frequent engine part replacement [10].

Other benefits of the biodiesel includes environmental benign, non toxic for humans, better biodegradability, reduced CO and CO<sub>2</sub> emissions, lower or almost zero sulfur emissions and no particulate matter released to the atmosphere [11, 12]. Hence, the research to improve biodiesel production to make it more economical viable becomes a priority today [3].

Oils and fats belong to a chemical family of lipids, which are produced from the metabolism of living organism. Most lipids are hydrophobic where they are soluble in common non-polar solvents. Lipid is called oil if it is liquid at 25°C, and other than that is fat. Fats are more saturated than oils.

Still, both contain complex fatty acids beside the phospholipids, glycelipids, sterols, carbohydrates, vitamins, water, odorants, other impurities in low concentrations, and free fatty acid (FFA) which depends on the lipids sources, treatments and storage conditions. Both oils and fats are usually triglycerides (TGs), with a different number of branched carbon chain of different length (4 – 24 carbons) and degree of saturation (1–3 carbon carbon double bonds) called fatty acids [2, 13, 14].

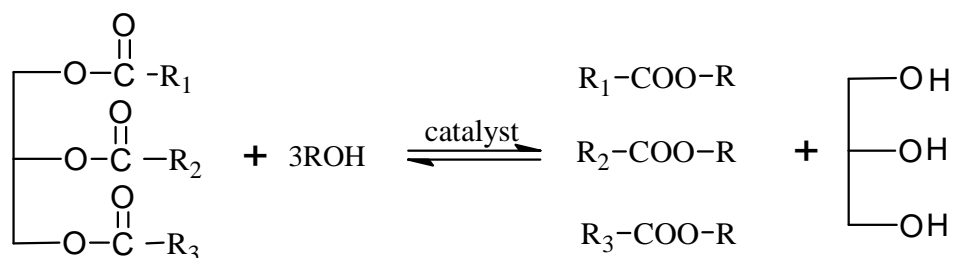
VOs are mainly composed of fatty acids. These include, palmitic (Hexadecanoic, C16:0) acid, oleic (Octadec-9-enoic, C18:1) acid, linoleic (9,12-octadecadienoic, C18:2) acid, stearic (Octadecanoic, C18:0) acid, myristic (Tetradecanoic, C14:0) acid, lauric (C12:0) acid, palmitoleic (C16:1) acid, margaric (C17:0) acid, linolenic (C18:3) acid, arachidic (C20:0) acid and gadoleic (C20:1) acid. The fatty acids present in some VOs are summarized in Table 1.2.

There are many available methods to convert VO into biodiesel, such as pyrolysis, micro-emulsion, catalytic cracking, and transesterification. Among these, transesterification process is found to be most suitable [4, 15]. The main purposes of the transesterification process are to lower the viscosity of the VO, decrease the carbon deposition in the engine, and decrease air pollution [16, 17].

Table 1.2 Fatty acid compositions of different VO's [7]

Oil	Fatty acid composition					
	Palmitic	Palmitoleic	Stearic	Oleic	Linoleic	Linolenic
Cottonseed	28.7	0	0.9	13.0	57.4	0
Poppyseed	12.6	0.1	4.0	22.3	60.2	0.5
Rapeseed	3.5	0	0.9	64.1	22.3	8.2
Safflowerseed	7.3	0	1.9	13.6	77.2	0
Sunflowerseed	6.4	0.1	2.9	17.7	72.9	0
Seasameseed	13.1	0	3.9	52.8	30.2	0
Linseed	5.1	0.3	2.5	18.9	18.1	55.1
Wheat grain	20.6	1.0	1.1	16.6	56.0	2.9
Corn marrow	11.8	0	2.0	24.8	61.3	0
Castor	1.1	0	3.1	4.9	1.3	0
Tallow	23.3	0.1	19.3	42.4	2.9	0.9
Soybean	13.9	0.3	2.1	23.2	56.2	4.3
Bay laurel leaf	25.9	0.3	3.1	10.8	11.3	17.6
Peanut kernel	11.4	0	2.4	48.3	32.0	0.9
Hazelnut kernel	4.9	0.2	2.6	83.6	8.5	0.2
Walnut kernel	7.2	0.2	1.9	18.5	56.0	16.2
Almond kernel	6.5	0.5	1.4	70.7	20.0	0
Olive kernel	5.0	0.3	1.6	74.7	17.6	0
Coconut	7.8	0.1	3.0	4.4	0.8	0
Palm	42.6	0.3	4.4	40.5	10.1	0.2

Transesterification, also called alcoholysis, is a chemical process between the TGs and alcohol in the presence of a suitable catalyst to form three molecules of alkyl esters and one molecule of glycerol. A typical transesterification reaction is shown in Figure 1.1.

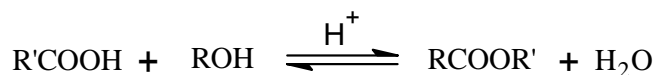


Where: R<sub>1</sub>, R<sub>2</sub> and R<sub>3</sub> are carbon chain of fatty acid; R is an alkyl group of alcohol.

Figure 1.1 Transesterification of TGs with alcohol in the presence of catalyst [15].

The homogeneous alkali catalyzed process, using NaOH, KOH and alkoxides have been used commercially and found to be most effective for biodiesel synthesis [18]. However, for VOs that contain high FFA, acid catalyst is required. The esterification of FFA with alcohol in the presence of an acid catalyst is as shown in Figure 1.2.

FFA catalyzed esterification reaction using acid catalyst (e.g., sulfuric, phosphoric, hydrochloric, and organic sulfonic acids) is more suitable for low quality feedstocks having significantly high amounts of FFA, such as unrefined oils, restaurant waste oils, and trap greases [18-20].



Where: R' is carbon chain of fatty acid; R is an alkyl group of alcohol.

Figure 1.2 Esterification of FFA with alcohol in presence of acidic catalyst.

Currently, the biodiesel production is more expensive than the production of petroleum based diesel fuel. The high production cost is associated with the relative high price of the virgin oil and refined oil used. The refined VOs account from 70 to 95% of the total cost of biodiesel production. This meaning that the biodiesel price is highly dependent on the raw material cost [21, 22]. Thus, other feedstock options are being evaluated as potential substitutes of the refined oils; among others include crude VO, soapstocks and waste oil. These oils can be potential alternatives as low-cost biodiesel feedstock.

However, these cheap feedstocks, as mentioned above, always contain high FFA content varying from 3–40% [21, 23]. Due to this high FFA content, the transesterification process requires acidic catalyst such as  $\text{H}_2\text{SO}_4$ , which has many drawbacks related to its acidic nature. Moreover, these catalysts will add to the production cost since the process will require high cost stainless steel equipment. In addition, it will necessitate an extensive and expansive conditioning separation, and purification steps [11, 24, 25].

Replacement of the acidic homogeneous catalysts with a more environmental friendly and reusable catalyst such as ionic liquid may solve these problems and permit a continuous operation in a more economical way.

## 1.2 Ionic liquids

Ionic liquids (ILs) are generally defined as salts of organic cations with low melting temperature below the boiling point of water. These compounds have been found to exhibit a variety of useful properties [26-28]. Thus, they have been accepted as a new green chemical which have stimulated the chemical industries to reduce the use of hazardous and polluting organics [29].

The world of ILs started with the first report of the synthesis of ethylammonium nitrate  $[\text{EtNH}_3][\text{NO}_3]$ , with melting point of 12 °C, in 1914 by Walden and co-workers. This compound was formed by the reaction of concentrated nitric acid with ethylamine. Following that, a highly reactive tetrachloroaluminate ( $\text{AlCl}_4^-$ ) based IL was synthesized in 1948 for electrochemical applications [26, 30]. Soon after, in 1970s, Wilkes and his colleagues synthesized a series of ILs based on the mixture of N-alkylpyridinium, 1,3-dialkylimidazolium halides and trihalogenoaluminate [31]. These ILs were used first as battery electrolytes for nuclear warheads and space probes as they required molten salts to be operated, and then later developed as solvents as well as catalysts [29].

In the early 1990's, Zaworotko and Wilkes reported the first example of air and moisture sensitive ILs, such as 1-ethyl-3-methylimidazolium hexafluorophosphate ( $\text{EMIMPF}_6$ ) and tetrafluoroborate ( $\text{EMIMBF}_4$ ). A wide range of cations and anions

sources were coupled to yield a new class of air and moisture stable solvents with diminutive vapor pressure, chemically stable and excellent solvents for a wide range of materials including organic, inorganic, and polymeric materials [31, 32]. In addition, they were thought to have usability in wider range of applications, such as oligomerisation [33], metal ion extraction [34], Friedel-Crafts acylation and alkylation [35], Diels-Alder reactions [36], and some other applications.

Recently, several researchers have revealed that ILs are more than just green chemicals. They have found the way to several applications such as making new materials, conducting heat effectively, hosting a variety of catalysts such as homogeneous, heterogeneous catalysis and supporting enzyme-catalyzed reactions, purification of gases, biological reactions media and removal of metal ions [29].

### **1.3 Structure and Properties of ILs**

Generally, the cations are bulk organic structure with low symmetry. Most of the described ILs based cations are shown in Figure. 1.3.

The most common cations consist of imidazolium, pyrrolidinium, pyridinium, tetraalkylammonium, and tetraalkylphosphonium. Most of the researches have been focused on room temperature ionic liquids (RTILs), in particular the asymmetric N,N'-dialkylimidazolium based cation, with a variety of anions, because of the wide spectrum of physical and chemical properties revealed by this class [29, 37].

The combination of anions and cations is expected to reveal billions of different structures of ILs. In fact, it is estimated that  $10^{18}$  ILs can be designed [38]. Some cations and anions combination comprising the main roots of ILs are shown in Figure 1.3.

ILs are assemblies of positively and negatively charged ions, whereas water and organic solvents are made of molecules. The assembly of ILs is alike to the table salts such as sodium chloride, which contains crystals created of positive sodium ions and negative chlorine ions, not molecules [29].

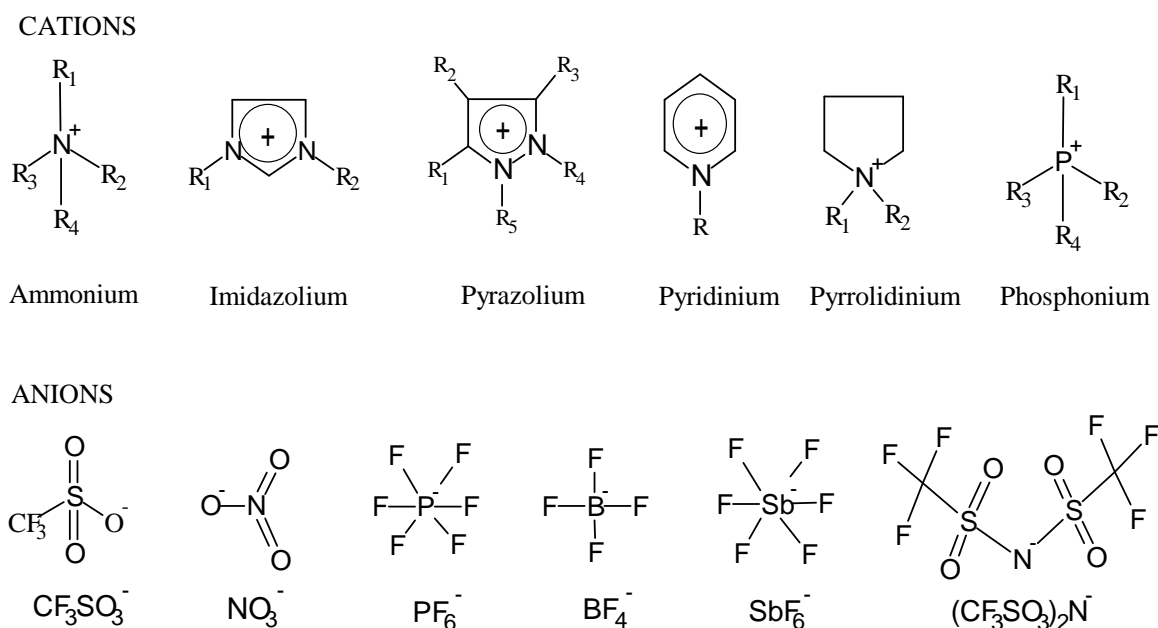


Figure 1.3 Examples of cation and anion pairs used to form the ILs.

Lately, ILs are gaining strong interest from both the academic and industrial fields, for their promising unique properties. Compared to conventional organic solvents, the use of ILs for synthesis and extraction has great advantages; their respective properties are determined by the unique combination of cation-anion properties [32]. Besides, the cation and anion, the size, geometry, and charge distribution of the ILs determine the properties [29], such as:

- Negligible vapor pressure [39].
- High thermal stability [29].
- Very miscible with polar substances, due to the mutual electrostatic or repulsion of charged particles interactions existing in ILs [32]. Also, ILs show high capability to dissolve a wide variety of materials; including salts, fats, proteins, amino acids, surfactants, sugars, and polysaccharides; and organic molecules including crude oil, inks, plastic, and DNA [29].
- A wide liquid range of about 300 °C, with low melting point [32]. Generally, lower melting (<100°C), due to the charge distribution on the ions, H-bonding ability, the



symmetry of ions and Van der Waals interactions [32], or due to the ions not well packed [40].

- Electrochemical stability [41].
- Non-flammability [27, 29].
- Can be tuned for specific purposes [42].
- High polarity [29].
- Excellent and variable Lewis/Brønsted acidity [32].
- Exhibit the behavior of reusability and recyclability [32].

ILs are also known as ‘designer solvents’ because of their nature that allows tailoring of their properties for a specific purpose. The task-specific ionic liquids (TSILs) are focus on designing acidic ILs in order to reduce or to replace the usage of polluting homogeneous and heterogeneous acids in some chemical applications. Replacing the organic chemicals with ILs is having more significant concern from the view of green Chemistry as it is one of the ways of making a highly specific process [32].

More recently, researchers have reported that ILs can be used in numerous applications such as making new materials, conducting heat effectively, supporting enzyme-catalyzed reactions, hosting a variety of catalysts, purification of gases, catalyst, biological reactions media and removal of metal ions [43].

The properties of ILs have been extensively studied by a great number of researchers, and they concluded that the IL properties have served to draw exponentially upward increasing interest into using RTILs as replacements for volatile organic compounds. However, there are many rules in chemistry were broken by some of the ILs. Such as one of the known basic rules of chemistry, “Like dissolves like”, which is broken by polar tetrachloroaluminate-based ILs when was found to be up to 50% soluble in non-polar benzene [29].

ILs currently play a role for green catalysis. More than 60% of products and more than 90% of universal processes rely on the rapidly growing field of catalysis. Moreover, the high specificity and efficiency beside the unique properties of ILs gave them a right to be practical for several areas of application including reaction and synthesis media [32].

Among the applications include nucleophilic and electrophilic reactions [37], synthesis of chalcone [44], plasticizer [45]; esterification of aliphatic acids [46], purification of gasses [47], etc. ILs also show active role in chemical reactions such as polymerization [48], carbonylation [49], oxidation [50], and sulfonation [51].

The present applications of ILs in the field of catalysis are diversified into many directions, and one of the main interests is to replace traditional liquid acids in catalytic reactions [32]. The work presented in this thesis is on the synthesis of acidic ILs for use as catalysts in biodiesel production from crude palm oil.

#### **1.4 Task specific ILs**

There is significant interest on the development of “task-specific ionic liquids” (TSILs) [26], where the anion and/or cation can be aimed to impart particular properties to the ionic liquid, such as chirality [52], or improved extraction or separation capability [53]. Hence, the design and selection of ionic liquid is focused on their physical properties and the effects of the chemical properties [54].

The ILs can, however, display significantly different behaviors; meanwhile their properties are still poorly understood. However, according to MacFarlane *et al.* [54], they can be categorized in terms of relative order of anions basicity or acidity.

The majority of ions used in designing ILs can be considered in terms of the Lewis classification of acids and bases. On the other hand, there are some ions that have to be considered according to the Brønsted definition; where they are able to donate or accept a proton. Anions that have the acidity behavior include dihydrogen phosphate and hydrogen sulfate [54].

The simplest examples of slightly acidic ionic liquids come from the routes of some imidazolium, pyrrolidinium, and some ammonium, and they are considered, to some extent, as ions having acidic protons [57-60]. Figure 1.4 shows the classification of some anions and cations in terms of their acidity, basicity, or in the acid/base sense, i.e. neutral.

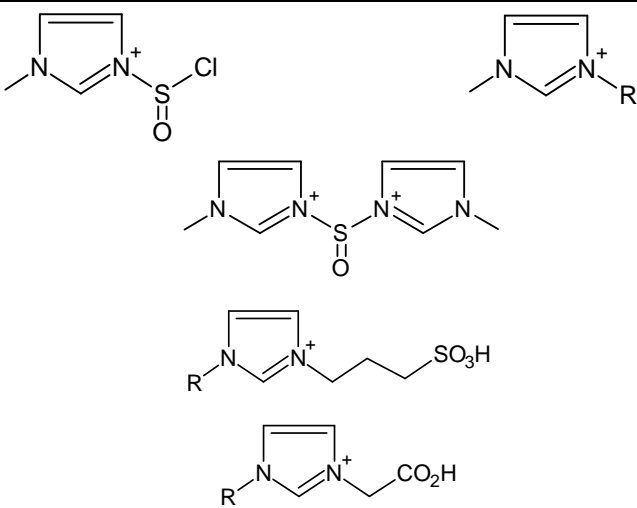
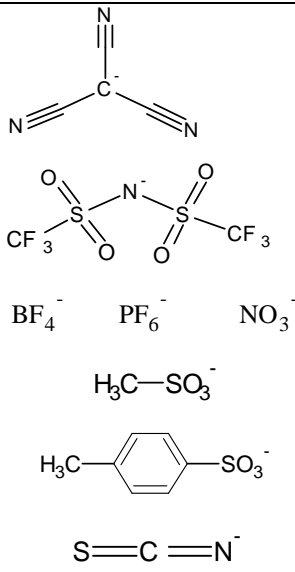
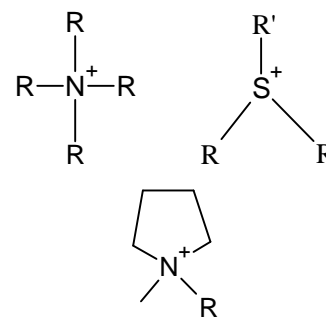
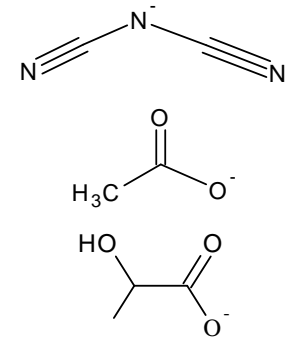
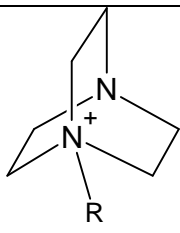
	Anion	Cation
Acidic	$\text{HSO}_4^-$ $\text{H}_2\text{PO}_4^-$ amphoteric	 Protonated cations
Neutral		
Basic		

Figure 1.4 Selected anions and cations categorized according to their acid/base properties [55, 56].

The hydrogen sulfate ( $\text{HSO}_4^-$ ) and dihydrogen phosphate ( $\text{H}_2\text{PO}_4^-$ ) are also classified as “amphoteric anions” with the potential to both accept and donate protons depending on the cations and chains present. Dubreuil *et al.* [61] have reported that the hydrogen sulfate anion is more acidic than dihydrogen phosphate. Zhao *et al.* [58] suggested the use of these two anions to catalyze the Mannich reaction.

In other reported work, Chris [62], in his patent, described the formula of the acidic cation as  $[\text{Cat}^+ - \text{Z} - \text{Acid}]$  (Figure. 1.5), where  $\text{Cat}^+$  may consist of heterocyclic cationic species of imidazolium, pyridinium, pyrazolium, thiazolium, isothiazolium, azathiazolium, oxothiazolium, etc.

Z is a covalent bond linked with  $\text{Cat}^+$  containing from 1–10 carbon atoms; and “A” is an acidic moiety joining Z, and can be selected from  $-\text{SO}_3\text{H}$ ,  $-\text{CO}_2\text{H}$ ,  $-\text{SO}_3-\text{Ph}-\text{R}$ ,  $-\text{SO}_3-\text{R}$ ,  $\text{RPO}(\text{OH})_2$ , or  $\text{R}_2\text{PO}(\text{OH})$ , where R is an alkyl group from  $\text{C}_1-\text{C}_6$  [62].

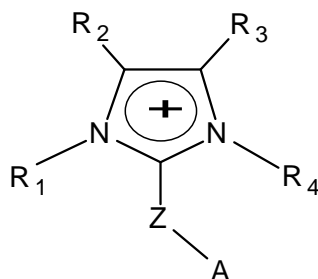


Figure 1.5 Example of acidic ionic liquid with imidazolium cationic species.

Where  $\text{R}_1$ ,  $\text{R}_2$ ,  $\text{R}_3$ , and  $\text{R}_4$  can be same or different alkyl group ranging from 1–40 straight or branched [62].

## 1.5 Problem Statement

Alkaline-catalyzed transesterification method is the most effective process for biodiesel production [63] and the use of low quality feedstocks such as unrefined oils (virgin oils) and used cooking oils could potentially make the biodiesel prices more competitive. However, these low cost feedstocks, always containing high amount of FFA ranging from

3–40%. These FFA, presented in feedstock, needs to be esterified to reduce its content because many researchers stated that, to use the alkali catalysts, the TGs should be having lower FFA content.

For instance, Freedman *et al.* [63], Mettelbach *et al.* [64], Liu [65] and Leung *et al.* [66] reported that the VOs used should be having FFA content of less than 1.0 %, equivalent to 2 mg KOH/g oil. Other researchers, Ma and Hanna [13], Di Serio *et al.* [20], Wang *et al.* [22], Canakci and Van Gerpen [67], and He *et al.* [68] stressed the importance of FFA content being below 0.5%. This esterification process will also add to cost of producing biodiesel that might make the production of biodiesel uneconomical despite the cheap feedstocks [21, 23].

As shown in Figure 1.6, the problem of using an alkali catalyst is the formation of soap that resulted from the partial reaction between FFA and the alkali catalyst. The formation of soap reduces the catalytic activity as the catalyst is being consumed in the reaction. Furthermore, the formation of this gel complicated the removal of alkyl ester from the glycerol by gravity separation since it is increasing the viscosity of the product mixture. Another disadvantage of using alkali catalyst is that large amount of water is required to separate the catalyst from the ester phase [69-72]. It is important to ensure complete removal of the catalyst because any trace residue left in the biodiesel can corrode vehicle fuel tanks and injector systems [73]. Thus, the removal of homogeneous base catalyst may add extra cost to the production cost [74, 75].

The transesterification of unrefined oils has been carried out by several researchers using non-green catalysts such as sulfuric acid, hydrochloric acid, phosphoric acid, and organic sulfonic acid [76, 77].

Although, the acidic catalysts were adopted for esterifying the feedstocks having high FFA, the process is required high amount of alcohol used typically in the range of 30:1 of alcohol to oil ratio, and expensive equipment. Whereas, using higher amount of alcohol requires large reactor size and necessitates extensive conditioning and purification steps to recover the alcohol, and to remove the acid catalyst from the reaction products [78].

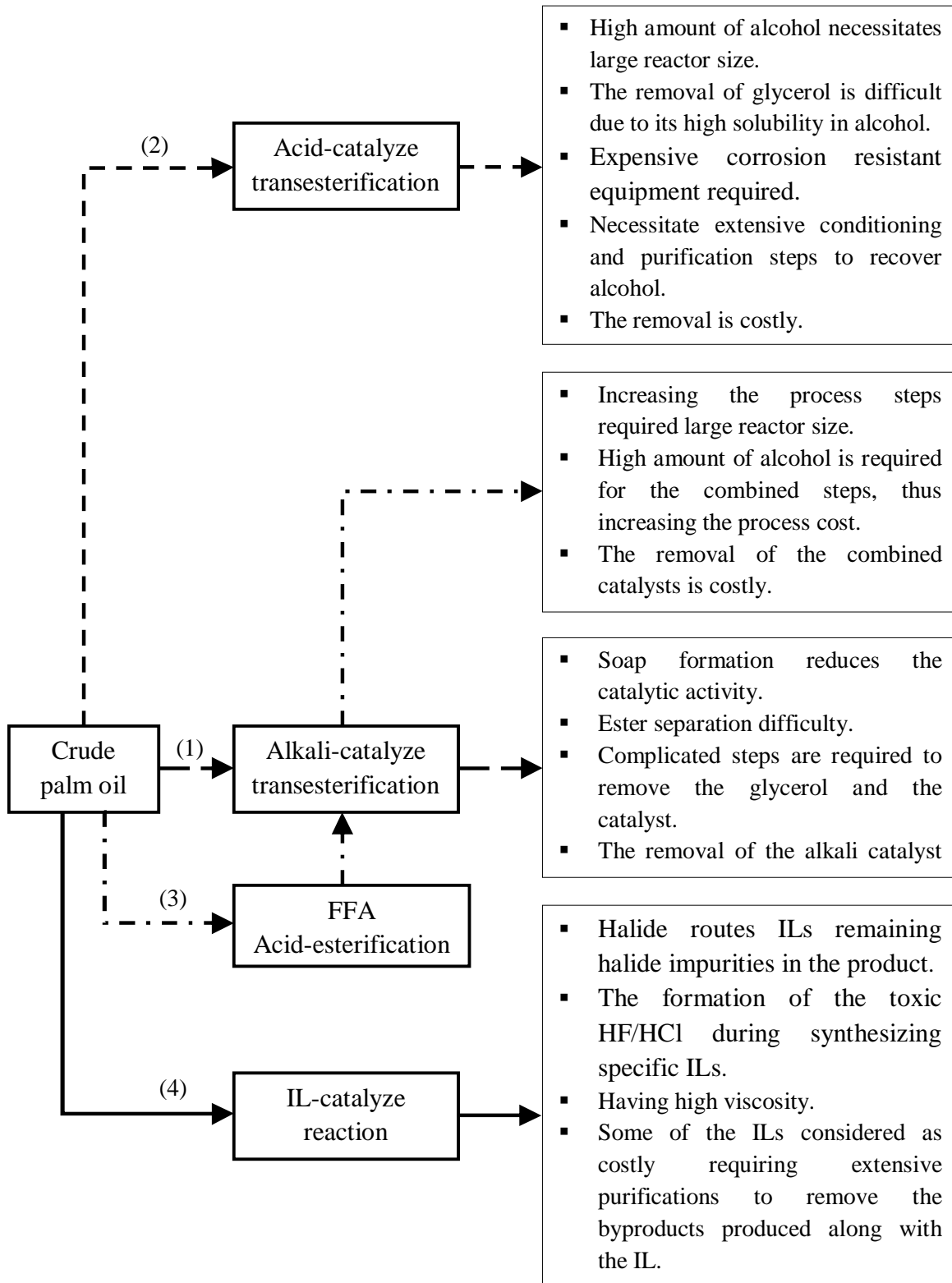


Figure 1.6 Flow chart of transesterification process using various types of catalyst.

Another problem associated with product purification is the complication in the removal of glycerol due to its high solubility in alcohol [11, 25, 76]. A process flow and their characteristics of biodiesel processes with various types of catalyst are shown in Figure 1.6.

In order to overcome the homogeneous acid catalyst drawbacks, heterogeneous catalysts have been used for the transesterification process recently [77, 79-81]. However, there appear to be many disadvantages that make the commercialization of biodiesel still unfeasible.

These disadvantages include the high cost of the heterogeneous catalysts [82], and their unstable activity resulting in incomplete conversion due to that it always started to drop after an hour of reaction. Furthermore, the catalyst leaching leads to phase separation difficulty and complicated the reusability of these catalysts. In addition to the high cost, this type of catalyst is difficult to synthesize, and is sensitive to water and air [82, 83].

## **1.6 Research approach and objectives**

In the processing technology, the challenge is to seek an alternative processes which is not only cost saving without sacrificing the quality and quantity of product, but also to reduce the waste products, which makes the process more environmental friendly. As a new green technology, ionic liquid seems as a promising catalyst that meets all of the criteria.

In addition to environmental concern on pollution from industrial waste, a more urgent need worldwide is to find alternative energy source, due to depleting petroleum reserves. Biodiesel is one of the potential renewable and clean energy sources; however, it is still expensive to produce biodiesel by the traditional homogeneous or heterogeneous catalyzed processes.

A lot of researches have been conducted in homogeneous and heterogeneous catalyzed transesterification of palm oil. However, none has tested IL as a catalyst for the process. Thus, for the aforementioned reasons, this research has been conducted to

investigate the use of ionic liquids in the field of catalysis as a replacement for homogeneous and heterogeneous acid in the transesterification of palm biodiesel. The research was carried out with the purpose of finding suitable catalyst characteristics, and optimum conditions for biodiesel synthesis.

The main objectives of this work are:

- To synthesize novel ionic liquid, possessing characteristics of easy recovery and recyclability, for biodiesel production from crude palm oil.
- To develop the relationships between the processing parameters; such as reaction temperature, molar ratio of methanol to oil and IL concentration; and product characteristics.
- To study, and compare the physicochemical properties of the produced palm biodiesel with the accepted international standards.
- To study the reaction kinetics and develop the reaction mechanism of the transesterification reaction.

## **1.7 Scope of Research**

This thesis aims to establish the method of producing biodiesel from crude palm oil using ionic liquid as green catalyst for the purpose of establishing green transesterification reaction process. It is also intended to produce new ionic liquids derived from different cations with different side chains and hydrogen sulfate as anion.

The study will start with an investigation into the impact of traditional acidic catalyst of  $\text{H}_2\text{SO}_4$  as preliminary test and evaluation catalyst. Different combinations of the reaction variables will lead to the optimal conditions for acid-catalyzed transesterification of crude palm oil.

Ionic liquids of different cations and couple with  $\text{HSO}_4^-$  anion will be synthesized, and characterized in terms of their purity and thermal stability, besides other important properties. In addition to imidazolium compounds, other cations such as ammonium and pyrazolium based cations will also be used as the transesterification catalysts.



Five different reaction parameters: methanol/CPO molar ratio, IL/CPO molar mass, reaction temperature, reaction time, and agitation rate will be tested and evaluated to determine the optimum reaction parameters. The physical and chemical properties of the produced methyl esters will be determined using standard testing methods.

A kinetic study of CPO transesterification will be conducted in order to investigate the effect of using IL on the reactions rates, and the activation energies will be calculated. The initial reaction rates and kinetic parameters of the transesterification reaction of CPO with methanol catalyzed by imidazolium ILs will be compared. The solubility of IL in both the CPO and the produced ester also tested. With the information obtained from gas chromatography and acidity monitoring, TG and FFA were measured, respectively. In addition, water formation was followed throughout the reaction. This information were analyzed and used to suggest the proposed reaction mechanism.

## **1.8 Thesis Outline**

This thesis consists of several chapters, which comprises:

- Chapter 1 - Biodiesel and ionic liquid introduction and background, including problem statement and current obstacles, objectives and approaches of this project as well as thesis outline.
- Chapter 2 - Literature Review, which discusses the definition of biodiesel, methods used to produce biodiesel, parameters that affect the yield of biodiesel, and reaction kinetics. Included also are recent researches on palm biodiesel production.
- Chapter 3 - Research Methodology, consisting of the methods used for the synthesized-ILs and produced-CPO characterization, transesterified product analysis using gas chromatography (GC), and the important physicochemical properties for all the 12 biodiesel samples.
- Chapter 4 - Identification of the novel designed ILs are presented in this chapter. Their properties, such as thermal decomposition, melting point, density and viscosity are included.

- Chapter 5 - The feasibility of using the synthesized ILs as catalyst for the transesterification reaction is discussed. Moreover, the results obtained from processes using IL and H<sub>2</sub>SO<sub>4</sub> are also detailed out. The discussion includes the yield of biodiesel, parameters that affect the reaction kinetics, and quality of the produced palm biodiesel. The reactions kinetic were discussed besides a new mechanism for the transesterification reaction was proposed.
- Chapter 6 - The conclusion of the research and recommendations for future work are presented in this chapter.