Recovery of Ionic Liquid [Bmim][$FeCl_4$] After Sulfur Extraction From Model Oil Using Liquid-Liquid Extraction Process

By

Nur Hafizah Binti Beserdin

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Chemical Engineering)

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Universiti Teknologi PETRONAS
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Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(Nur Hafizah Binti Beserdin)

ABSTRACT

Ionic Liquid (IL) is proven to be able to extract sulphur compound from petroleum distillates such as diesel and gasoline providing an alternative to replace the conventional sulphur removing method that is the Catalytic Hydrodesulphurization (HDS), which requires both high temperature and pressure. Many researchers have pointed out the major concerns in the use of IL is their relatively high cost, which makes their recycling an important issue for further study. In addition, currently, quite limited studies are being conducted on the recovery, regeneration or recycling of IL after extractive desulphurization. Therefore, this research project is conducted where attention are given to the recycling and recovery of the chosen IL that is, 1-butyl-3-methylimidazolium tetrachloroferrate [Bmim][$FeCl_4$] using Liquid-liquid Extraction (LLE) method with a co-solvent, hexane. The aim of this research is to study the recyclability of the IL and to investigate on the effect of three manipulated variables which are, the contact time of the regeneration process, the ratio between sulphur-loaded IL to the co-solvent and the recovery process temperature on the recovery efficiency in term of sulphur removal. Project scope are divided into four stages; synthesis of the [Bmim][$FeCl_4$] IL, extraction of benzothiophene from model oil Dodecane, recovery of the sulphur-loaded [Bmim][$FeCl_4$] using hexane as solvent and lastly to re-use the recovered [Bmim][$FeCl_4$]. The characterization is done using Fourier Transform InfraRed (FT-IR) and Carbon, Hydrogen, Nitrogen and Sulfur Analyzer (CHNS). The approach for this project is mainly LLE. Extractive desulfurization between [Bmim][$FeCl_4$] and model oil containing sulphur is done to obtained sulphur-loaded [Bmim][$FeCl_4$] and extraction using hexane is conducted to recover the IL. The recovered [Bmim][$FeCl_4$] is then reused for extractive desulfurization and the process is repeated. Recyclability is evaluated without recovery. The result shows the chemical composition of [Bmim][$FeCl_4$] does not change meaning that the recpvery process is a successful and optimizations on IL to solvent ratio prove that ratio of 1:2 is the best option for regeneration. 80 minutes contact time and higher temperature ranging between 45°C to 65°C gives the highest efficiency of recovery.

ACKNOWLEDGEMENT

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Nur Hafizah Beserdin

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABBREVIATION AND NOMENCLUTURES

Biodesulfurization	BDS
Dibenzothiophene	DBT
Fourier Transform Infrared	FT-IR
Hydrodesulfurization	HDS
Hydrogen Sulfide	H_2S
Ionic Liquids	ILs
Liquid-liquid Extraction	LLE
PETRONAS Ionic Liquid Centre	PILC
Sulfur Dioxide	SO_2
Universiti Teknologi PETRONAS	UTP
Volatile Organic Compound	VOC

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Since early 1990s, there has been a growing interest in expanding Ionic Liquids (ILs) to widespread applications in various field including organic and physical chemistry, electrochemistry, engineering and chemical processing. ILs are basically organic salts composed of entirely ions and are in liquid form at ambient temperature. IL is recognized as solvents for "Green Chemistry" and therefore it promises an environmental friendly reaction [2,3]. A part from the mentioned applications of ILs above, great attention is currently given to its usage in Petroleum Industry where ILs may be use for desulfurization of crude oil [4].

Due to atmospheric pollution concerns, toxicity and high tendency to damage engines and equipments by corrosion, the sulfur in crude oil and fuel need to be reduced to allowable and safe limit. Conventional method to remove sulfur compound is by Hydrodesulfurization (HDS) process, which requires hydrogen and high operating pressure and temperature [4,5]. However, some aromatic sulfur compounds such as thiophene and dibenzothiophene (DBT) that usually present in middle distillate such as diesel oil are less reactive to HDS process catalyst [6].

ILs is used as a selective extraction agent of sulfur compounds via Liquid-Liquid Extraction (LLE) technique. ILs not only have the potential to increase the chemical reactivity and lead to efficiency of the process, but also non-flammable and less toxic than the common solvents due to their low vapour pressure. The application of mild process conditions in the sulfur extraction process, which is at ambient temperature and pressure and the fact that no hydrogen and catalysis is needed are additional advantages of using ILs compared to HDS. Studies also show that by using ILs, the sulfur compound that is hard to remove by HDS can be extracted easily [3, 4, 7].

Regeneration or recovery of used ILs is crucial because for industrial uses, large amount of ILs is needed and they are expensive in comparison with other solvent. The ILs used to extract the sulfur compound can be recovered using several methods such as distillation or stripping of solute from ILs, extraction with Volatile Organic Compound (VOC) solvent, supercritical CO2 extraction and heating or evaporation of volatiles under vacuum [4, 8]. The method chosen is closely dependent on the types and how the ILs is being used. In this report, $[Bmim][FeCl_4]$ is used to run the recovery experiment process after it has extracted the sulfur compound from the model oil which is basically the mixture of Dodecane with DBT. $[Bmim][FeCl_4]$ is selected because of it is insoluble in oil, can be synthesis in-house with relatively cheap starting materials and it has been proven that this particular IL can be recovered [7].

The recovery method selected in this report is using a simple solvent extraction by using low boiling point hydrocarbons such as pentane or hexane basically because they are not miscible with $[Bmim][FeCl_4]$. In the recovery point of view, the extraction mechanism with imidazolium IL is caused by the formation of liquid clathrate due to the π–π interaction between the unsaturated bonds of sulfur-compound and IL-imidazolium ring [9]. π–π interaction here means that the sulfur compound is bonded to the IL physically and separation process (recovery) can be done easily using LLE.

1.2 Problem Statement

It is known that ILs are expensive in the market, therefore, recycling and recovering them are very important. When we can reuse the IL, we can save on the cost of purchasing the fresh stock of IL. Among the published literature, it is generally recognized that ILs are easily recovered or regenerated. The recovery of ILs are largely a result of their lack of solubility in some organic solvent which make LLE method possible [9].

In this project, LLE method to recover [Bmim][$FeCl_4$] by using co-solvent hexane is used to extract the sulfur from sulfur-loaded [Bmim][$FeCl_4$] (after desulfurization process on model oil). Research have been done on the LLE recovery ability in term of sulfur removal and it has been proven that [Bmim][$FeCl_4$] can be recovered [10]. However, the problem is that, there are no sufficient data on the recovery method using LLE for [Bmim][$FeCl_4$] and its recyclability with or without recovery.

This project is conducted with the aim to study and obtain valuable result on the recovery efficiency of chosen IL, [Bmim][$FeCl_4$] because there are very limited information or proven result and data available regarding the efficiency of [Bmim][$FeCl_4$] recovery process in term of sulfur removal on how we can further optimized the recovery process to obtained highest percentage of sulfur removal specifically by using LLE method. This project can provide a new perspective towards the optimization of [Bmim][$FeCl_4$] recovery so that it can be recycle and re-use with maximum capability.

1.3 Objective

The focus subjects in this project are generally to investigate the recyclability of [Bmim][$FeCl_4$] without recovery process and also the factors that are affecting the performance of the recovery process using LLE method. Thus, the main objectives that have been identified to ensure a complete analysis and promising results for this project are:

1) To study the recyclability of [Bmim][$FeCl_4$] by re-using it without recovery in order to determine how many times the it can be recycle and re-use before it loses its purity and ability to extract sulfur from model oil.
2) To study the recovery of [Bmim][$FeCl_4$] after extractive desulfurization by using LLE method with co-solvent hexane and to investigate the efficiency of [Bmim][$FeCl_4$] recovery process in terms of:-
    - Contact time
    - Different ratio of sulfur-loaded [Bmim][$FeCl_4$] to Hexane
    - Different process temperature

1.4 Scope of Study and Feasibility of Project

This experiment will be conducted at standard condition that is at room temperature (25°C) and pressure (1 atm). The LLE process will be done in batch using common lab apparatus. The experiments are designed and will be conducted in UTP, PETRONAS Ionic Liquid Lab where all the chemicals and advanced equipments are available and in good condition. This research project can be completed within the time frame given that is approximately 4 months starting from May 2012.

This research project will be able to provide ideas to support a great deal of future research on [Bmim][$FeCl_4$] recovery and recyclability. It will also give new perspective towards the optimization of [Bmim][$FeCl_4$] recovery process and finally it will provide valuable recyclability data in evaluating [Bmim][$FeCl_4$] viability in commercial application.

CHAPTER 2

LITERATURE REVIEW

2.1 Ionic Liquid An Introduction

Ionic Liquids (ILs) are organic salts in the form of liquid at room temperature and composed of entirely ions, typically large organic cations and small inorganic anions [3, 10]. Within the last twenty years, research involving these unique liquids has grown exponentially due to their notable characteristics. ILs have no detectable vapour pressure, extremely low volatility, low combustibility, corrosion resistance to plastic and carbon steels, excellent thermal stability, high solvating capability and available in a wide liquid range [3, 11].

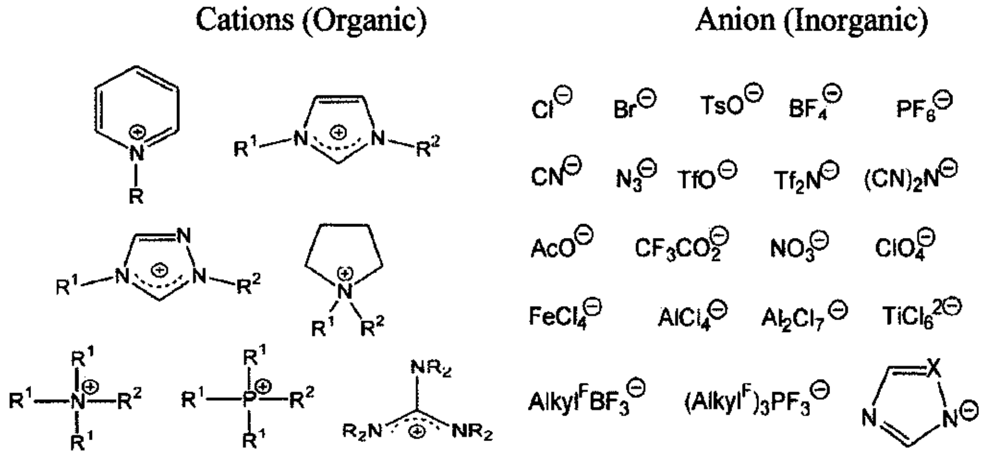


Figure 2.1: Example of cations and anions in ILs [3]

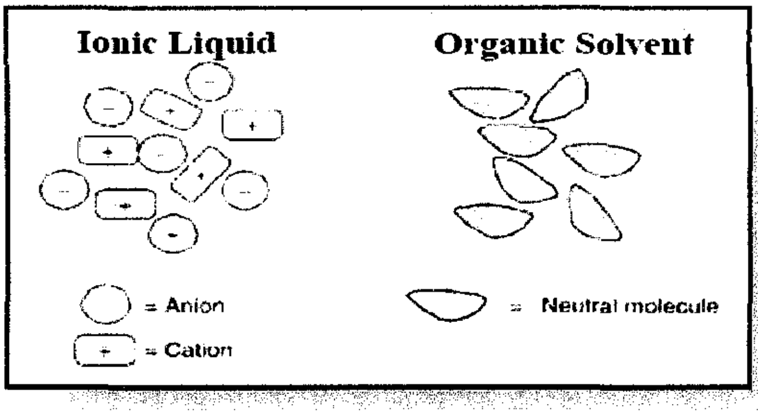


Figure 2.2: Chemical structure for ILs versus organic solvent

Large number of ILs can be produced with the variations in cations and anions. The properties of ILs depend on their ions structure. With the enormous diversity of substituent available to make useful ILs, they can be designed with the foresight of having desired physical properties and less toxic chemical properties [3, 12]. The solubility of ILs depends on the nature of the anion, temperature and the length of the alkyl chain on the organic cations. Tetrafluoroborates, chlorides, nitrates and trifluoroacetates display complete miscibility in water while, hexafluorophosphates, triflimides and other perfluorinated anions shows very low solubility in water [13]. The hydrophilic or hydrophobic behaviour is important for the salvation properties of ILs as it is necessary to dissolve reactants and it is also important for the recovery of products by solvent extraction [3, 13].

The most commonly used cations in ILs are imidazolium, pyridinium, pyrolidium and phosphonium base [7, 10, 12]. In this project, imidazolium based IL is being used for the experiments. The solubility of different species in imidazolium ILs depends mainly on polarity and hydrogen (H) bonding ability [14, 15]. Werner *et al.* stated that many ILs have unusual solubility and miscibility properties, attractive electricity conductivity and interesting polarity nucleophilicity for catalysis [16].

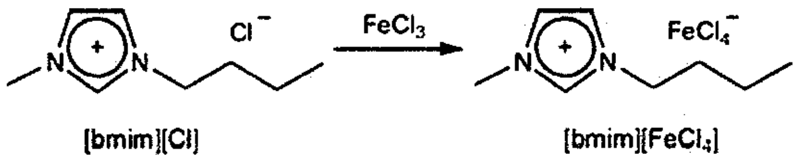


Figure 2.3: Structure of [Bmim][FeCl$_4$]

[Bmim][FeCl$_4$] is chosen in this project since it is insoluble in model oil and stable which means the experiment can be easily conducted at ambient temperature and pressure based on previous research by Jasmari, N. F. [7]. [Bmim][FeCl$_4$] is a kind of IL that has special magnetic properties since they show strong response to magnetic field. Based on the literature, recently, the FeCl$_4^-$ moiety could play roles as synthetic catalyst as well as dopant when polymerization of π-conjugated polymer was conducted in [Bmim][FeCl$_4$] [17,18].

The thermodynamic and kinetics of processes carried out in ILs media are different from those carried out in conventional media. Therefore, it creates new opportunities for catalytic reactions, separations, electrochemistry and combined reaction-separation processes [8, 11]. ILs have a wide range of various application in the chemical industry such as metal ions removal from waste water, absorption of gases for storage and delivery of highly toxic, flammable and reactive gases, hydroformilation, hydrogenation and a very important application which is the main highlight in this report is in extractive desulfurization of crude petroleum, liquefied petroleum gas (LPG), diesel fuel and jet fuel [6, 8, 16]

2.2 Desulfurization

2.2.1 Sulfur content in petroleum crude and products

Sulfur bonded chemically to hydrocarbon molecules making it hard to be separated. That is, not until it is burned, where the exhaust gases containing sulfur dioxide (SO_2) and sulfuric acid [4, 6]. This corrosive sulfur compounds have an obnoxious odour causing air pollution and acid rain [5]. Each crude oil has different amounts and types of sulfur compound, but as a standard rule, the proportion, stability and complexity of it increase in heavier crude oil fraction such as gasoline and diesel [9].

Since 2010, the total sulfur content cannot be higher than 10ppm in many countries based on the European Union; therefore, considerable attention has been given to deep desulfurization of gasoline and diesel [5]. This means that the degree of sulfur-conversion needs to meet its maximum capacity. Many research groups carry out investigations of environmentally friendly, energy saving and effective desulfurization methods [20].

2.2.2 Current technology for Desulfurization process in the industry

Hydro-desulfurization (HDS) is the conventional desulfurization technology used in many refineries around the world [4, 5, 6]. In HDS, organic sulfur compound are reacted under elevated temperatures (300℃ – 400℃) as well as elevated pressure (20 – 100 atm) with the use of catalyst which makes the method very costly and energy consumptive [5, 21, 22, 23]. In HDS, the bonds between the carbon and sulfur atoms are broken and it combines with additional hydrogen to form the toxic gas that is the hydrogen sulphide (H_2S) [4, 21, 23]. However, the reactivity of sulfur compounds in HDS strongly depends on the molecular structure. The aromatic sulfur compounds including thiophenes, benzothiophenes and their alkylated derivatives, which are present in heavier crude oil fraction, are difficult to convert over HDS catalyst [5, 6]. Thus, new alternative process needs to be investigated in order to abide with the tight regulation on sulfur content in fuel. Other method available are summarized in the table below:

Table 2.1: Summary of Available Conventional Desulfurization Method

Technologies	Advantages	Disadvantages
Solvent Reaction	• Lower extraction duration. • Reduce natural oil losses. • Lower Operating Cost.	• Perform under high pressure condition.
Biodesulfurization (BDS)	• Lower operation and energy cost. • Require less energy and hydrogen. • High selectivity. • No generation of undesirable side products. • Non-invasive approach.	• Dependant on the extreme conditions encountered in petroleum refining. • Efficiency related to oil droplet size. • Isolating new strains for desulfurizing a broader range of sulfur compounds.
Oxy-Desulfurization (ODS)	• Can be carried out in the liquid phase under very mild conditions. • Sulfur can be easily removed using distillation, SE, adsorption, and decomposition.	• Forced to take more drastic reaction condition. • High amount of oxidants. • Low oil recovery efficiency.

2.2.3 Desulfurization Using Ionic Liquid

Many investigations have been directed towards expanding a new original approached to deep desulfurization of fuels such as a processes based on distillation, adsorption, precipitation, photochemical, extraction and combinations of mentioned methods [8]. The use of IL using LLE method shows a great potential in comparison with the traditional organic solvent to extract sulfur from crude oil due to their unique physicochemical properties as stated earlier [5]. The first investigation of fuels extractive desulfurization using IL has been reported by Bossman *et al.* in the year 2001 [24]. This extractive desulfurization is carried out at ambient condition without the need of hydrogen is the main advantages compared to HDS [4, 5, 6].

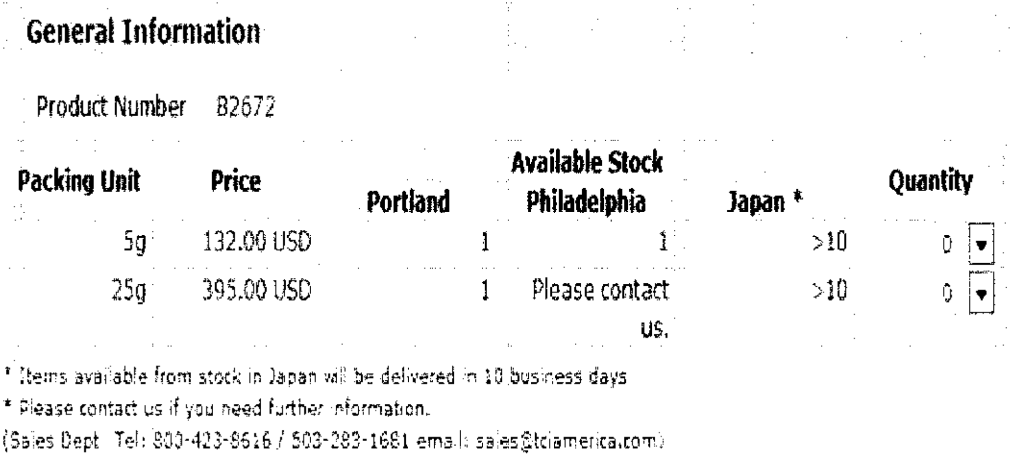
The reason that IL can be used in desulfurization is that molecules with highly polarisable π-electron density (such as thiophene) preferably insert into the dynamic structure of the IL and the driving force for the molecular insertion is the favourable electronic interaction of polarized aromatic molecules with the charged ion pairs of IL [25]. According to Nan Hee Ko and co-workers (2008), [Bmim][$FeCl_4$] are highly effective for extraction of sulfur at ambient conditions [26].

Table 2.2: Desulfurization of Model Oil using various types of ILs [26]

Ionic Liquids (ILs)	Degree of Desulfurization (%)
$FeCl_3$ –[HHIm]Cl	9.6
$FeCl_3$ –[HMIm]Cl	11.7
$FeCl_3$ –[HDMIm]Cl	18.5
$FeCl_3$ –[BMIm]Cl	42.2
$FeCl_3$ –[BMIm]Cl^z	100
$FeCl_3$ –[BDMIm]Cl	44.8
$FeCl_3$ –[BDMIm]Cl^z	100
$AlCl_3$ –[BMIm]Cl^z	23.5
CuCl –[BMIm]Cl^z	17.2
$FeCl_3$ –[HHIm]$Cl^{z,d}$	100

2.3 Recovery of Ionic Liquid

Currently ILs are quite expensive in comparison with conventional molecular solvents and somehow, large amount of ILs are needed in various application. Thus, efficient recycling of ILs is important for their economic use, especially in large-scale [27]. However, Khodadoust and co-workers (2006) estimates that the cost for ILs is expected to decrease due to their higher production a rate, which is expected to render ILs economically competitive with organic solvents [28]. To overcome the cost problem, some low cost and simple synthesis of IL are important for recycling and re-use. Below is the latest pricing of [Bmim][$FeCl_4$] by TCI America:-



General Information

Product Number B2672

Packing Unit	Price	Available Stock Portland	 Philadelphia	 Japan *	Quantity
5g	132.00 USD	1	1	>10	0 ▾
25g	395.00 USD	1	Please contact us.	>10	0 ▾

* Items available from stock in Japan will be delivered in 10 business days

* Please contact us if you need further information.

(Sales Dept Tel: 800-423-8616 / 503-283-1681 email: sales@tciamerica.com)

Figure 2.4: Current price of [Bmim][$FeCl_4$] by TCI America

In general, ILs has higher density than organic liquids and water. Therefore, many ILs existed as a separated phase when in contact with organic aqueous phase. This feature makes it possible to recycle the ILs for multiple extractions without additional environmental concerns [6]. The recycling rules usually make use of the low solubility of the IL in organic solvents. This allows products and residual organics to be extracted using an organic solvent while the salt by-product present in water-immiscible ILs can be washed out with water. For water-miscible ILs, it is hard to recycle because inorganic by-products cannot be removed directly [8, 27]. Appropriate separation techniques should be selected according to the different system.

In most published literature, lab scale ILs are easily recycled or recovered and various successful techniques have been proven such as heating or evaporation of volatiles under vacuum, re-extraction with supercritical carbon dioxide ($ScCO_2$), precipitating sulfur compound by water dilution, LLE using VOC and lastly due to ILs not having a detectable vapour pressure distillation or stripping method can be use where the IL will remain in the liquid phase when distilled [4, 8, 29, 30].

Due to simple operation option, mild process conditions and economical advantages, the separation of mixtures by LLE is often applied. High efficiency of this technique depends largely on precisely selected suitable solvent for specific separation process [5]. In this report, sulfur-loaded [Bmim][$FeCl_4$] is recovered using LLE method with the use of hexane as the co-solvent because it is known as the simplest and easiest solvent that can be used. Water is not being used because [Bmim][$FeCl_4$] is miscible in it while hexane is insoluble in [Bmim][$FeCl_4$]. This makes the separation process easier and more feasible.

2.4 Qualitative and Quantitative Analysis

Qualitative Analysis: Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared spectroscopy, FT-IR can provide us three major information about the samples. It can identify the unknown materials, it can determine the quality or consistency of a sample and it can also determine the amount of components in the mixture. In infrared spectroscopy, infrared (IR) radiation is passed through a sample. Some of the infrared radiation is absorbed, some of it is passed through or transmitted and this will create a molecular fingerprint for the sample.

FT-IR in this experiment serves as a qualitative analysis. Sulfur compound has a small peak on FT-IR. Tables below show some of the wave numbers for certain compound.

Table 2.3: Functional Groups Easily Spotted in a Spectrum

Band Position in cm^{-1}	Functional Group
3500 – 3200	O-H, N-H
3200 – 2800	C-H
2250 – 2000	C=N, C=C
1800 - 1600	C=O
< 1000	C=C

Table 2.4: The Group Wavenumbers for SO_2 Containing Molecules

Functional Group	Asymm. SO_2 Stretch	Sym. SO_2 Stretch
Sulphone	1340 - 1310	1165 - 1135
Sulphonate	1430 - 1330	1200 - 1150
Sulphate	1450 - 1350	1230 – 1150

Based on the result, for the Dodecane (model oil) with DBT, small peak of sulfur is detected and after the desulfurization the peak is no longer on the graph.

Quantitative Analysis: Carbon, Hydrogen, Nitrogen and Sulfur Analyzer

For quantitative analysis, CHNS Analyzer is used. CHNS Analyzer measures the amount of elemental compound specifically for carbon, hydrogen, nitrogen and sulfur in a sample. Sample preparation need to be done before analysis can be conducted. The CHNS will give the result in terms of percentage. The standard used is Sulfamethazime which contains 51.78 % of Carbon, 5.07 % of Hydrogen, 20.13 % of Nitrogen and 11.52 % of Sulfur. Each sample is run 3 times and the average was calculated.

CHAPTER 3

METHODOLOGY

3.1 Preparation of [Bmim][FeCl$_4$] and Model Oil

[Bmim][FeCl$_4$] are obtained from the PETRONAS Ionic Liquid Centre where it has been synthesized in-house by the research officer. Model oil used in this experiment is dodecane provided by Sigma-Aldrich and the sulfur species used is dibenzothiophenes (DBT). 1.0 liter of the model oil is prepared by mixing the DBT into dodecane. The amount on both [Bmim][FeCl$_4$] and model oil is measured by weight for the experiment. Next is to conduct the extractive desulfurization experiment. A batch of ratio 1 to 1 that is 50 grams [Bmim][FeCl$_4$] is used to desulfurize 50 grams of model oil. Later, the sulfur-loaded [Bmim][FeCl$_4$] is divided into two parts, part A of 20 grams and part B of 30 grams. Part A will be use for recycling experiment and part B is for the recovery experiment. The recovery using LLE method requires n-Hexane provided by Merck. Samples for each experiment are taken for qualitative using FT-IR spectroscopy and quantitative analysis using CHNS analyzer.

3.2 Part A: Recycling of [Bmim][FeCl$_4$] Strategy

Sulfur-loaded [Bmim][FeCl$_4$] will be use to extract sulfur from the fresh stock of model oil for every cycle.

To study the recyclability of [Bmim][FeCl$_4$] in Extractive Desulfurization

Material: 20 grams of [Bmim][FeCl$_4$], Dodecane 2wt% DBT

Preparation: Mix 20 grams of [Bmim][FeCl4] with 20 grams of model oil (1 to 1 ratio) in a 3-neck flask. Stir it with 400rpm speed for 30 minutes at T= 30°C. Let the mixture settled for 10 minutes and then separate the sulfur-loaded [Bmim][FeCl$_4$] from the model oil. Reuse the sulfur-loaded [Bmim][FeCl$_4$] for extractive desulfurization using a fresh stock of model oil with appropriate ampunt of 1 to 1 ratio. Repeat the procedure more than 5 times (7 times) and for each cycle, prepare a small sample of about 1 gram model oil and [Bmim][$FeCl_4$] for analyisis.

Analysis: The samples are sent for analysis. For FT-IR, the data obtained is re-plotted using excel. Comparison is done between the pure [Bmim][$FeCl_4$] and recycled [Bmim][$FeCl_4$] for any change in chemical structure. CHNS Analyzer is used to determined the sulfur content in the model oil and also [Bmim][$FeCl_4$]. The amount of sulfur is obtained in percentage.

3.3 Part B: Recovery of Sulfur-Loaded [Bmim][$FeCl_4$] Strategy

To study the Recovery Performance of the Sulfur-loaded [Bmim][$FeCl_4$] based on Different Time Variation

Material: 10 grams of sulfur-loaded [Bmim][$FeCl_4$], 10 grams of n-hexane

Preparation: Mix 2 grams of sulfur-loaded [Bmim][$FeCl_4$] with 2 grams of n-hexane into 4 different vials. Using a small size stirrer, the mixtures are stirred at 400rpm speed at room temperatures, 25 degree Celcius. The first vials is stirred for 30 minutes, next is 60 minutes, 90 minutes and lastly 120 minutes. The mixture is leave to settle for 10 minutes and then the recovered [Bmim][$FeCl_4$] is separated from the hexane using small tip dropper. This is possible due to the immiscibility of the two liquid. About 1 grams of sulfur-loaded [Bmim][$FeCl_4$] and recovered [Bmim][$FeCl_4$] for all the four different contact time is taken out for analysis.

Analysis: All the samples are sent for FT-IR spectroscopy and CHNS Analyzer. For FT-IR, the data obtained is re-plotted using excel. Comparison is done between the pure [Bmim][$FeCl_4$] and recovered [Bmim][$FeCl_4$] for any change in chemical structure. CHNS Analyzer is used to determined the sulfur content in the sulfur-loaded [Bmim][$FeCl_4$] and recovered [Bmim][$FeCl_4$]. The amount of sulfur is obtained in percentage. Calculation on the efficiency of the recovery process for different contact time is done based on the ability of sulfur removal.

To study the Recovery Performance of the Sulfur-loaded [Bmim][FeCl$_4$] based on Different Solvent Ratio

Material: 10 grams of sulfur-loaded [Bmim][FeCl$_4$], 10 grams of n-hexane

Preparation: Mix sulfur-loaded [Bmim][FeCl$_4$] with n-hexane into 3 different vials. For the first vial use 1 to 1 ratio, that is 2 grams of sulfur-loaded [Bmim][FeCl$_4$] and 2 grams of hexane. The second vial is for 1 to 2 ratio, that is 2 grams of sulfur-loaded [Bmim][FeCl$_4$] and 4 grams of hexane and lastly in the third vial is for 2 to 1 ratio, that is 4 grams of sulfur-loaded [Bmim][FeCl$_4$] and 2 grams of hexane. Using a small size stirrer, the mixtures are stirred at 400rpm speed at room temperatures, 25 degree Celcius for 30 minutes. The mixture is leave to settle for 10 minutes and then the recovered [Bmim][FeCl$_4$] is separated from the hexane using small tip dropper. This is possible due to the immiscibility of the two liquid. About 1 grams of sulfur-loaded [Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$] for all the 3 different ratio is taken out for analysis.

Analysis: All the samples are sent for FT-IR spectroscopy and CHNS Analyzer. For FT-IR, the data obtained is re-plotted using excel. Comparison is done between the pure [Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$] for any change in chemical structure. CHNS Analyzer is used to determined the sulfur content in the sulfur-loaded [Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$]. The amount of sulfur is obtained in percentage. Calculation on the efficiency of the recovery process for different solvent ratio is done based on the ability of sulfur removal.

To study the Recovery Performance of the Sulfur-loaded [Bmim][FeCl$_4$] based on Different Process Temperature

Material: 10 grams of sulfur-loaded [Bmim][FeCl$_4$], 10 grams of n-hexane

Preparation: Mix 2 grams of sulfur-loaded [Bmim][FeCl$_4$] with 2 grams of n-hexane into 3 different vials for three different process temperatures. Using a small size stirrer, the mixtures are stirred at 400rpm speed for 30 minutes. The first vials is stirred for 30 minutes at 25 degree Celsius, next is 45 degree Celsius and lastly at 65 degree Celcius. The mixture is leave to settle for 10 minutes and then the recovered [Bmim][FeCl$_4$] is separated from the hexane using small tip dropper. This is possible due to the immiscibility of the two liquid. About 1 grams of sulfur-loaded

[Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$] for all the 3 different contact time is taken out for analysis.

Analysis: All the samples are sent for FT-IR spectroscopy and CHNS Analyzer. For FT-IR, the data obtained is re-plotted using excel. Comparison is done between the pure [Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$] for any change in chemical structure. CHNS Analyzer is used to determined the sulfur content in the sulfur-loaded [Bmim][FeCl$_4$] and recovered [Bmim][FeCl$_4$]. The amount of sulfur is obtained in percentage. Calculation on the efficiency of the recovery process for different process temperature is done based on the ability of sulfur removal.

3.4 Calculation

- Degree of Desulfurization (in term of sulfur removal in ***Dodecane***)

$$= \frac{\text{Initial Sulfur Content} - \text{After Extraction Sulfur Content}}{\text{Initial Sulfur Content}} \times 100\%$$

Note: For degree of desulfurization, we take the amount of sulfur in the model oil that is the mixture of dodecane and DBT prepared earlier. This is the amount of the initial sulfur content.

- Recovery Performance (in term of sulfur removal in ***[Bmim][FeCl4]***)

$$= \frac{\text{Sulfur-Loaded [Bmim][FeCl}_4\text{]} - \text{After recovery Sulfur Content}}{\text{Sulfur-Loaded [Bmim][FeCl}_4\text{] Sulfur Content}} \times 100\%$$

Note: For recovery efficiency, we look at the sulfur content in [Bmim][FeCl$_4$]. The sulfur-loaded [Bmim][FeCl$_4$] here is the amount of sulfur in it after the first extractive desulfurization. It is the same for all the three different sections (contact time variation, different solvent ratio and different process temperature) of Part B.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Recyclability of [Bmim][FeCl$_4$]

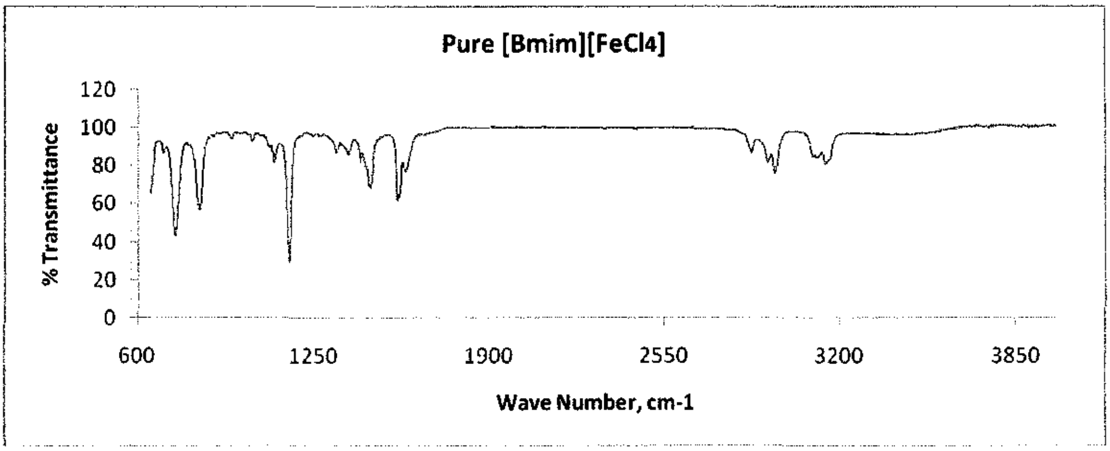


Figure 4.1: FT-IR Spectroscopy for Pure [Bmim][FeCl$_4$]

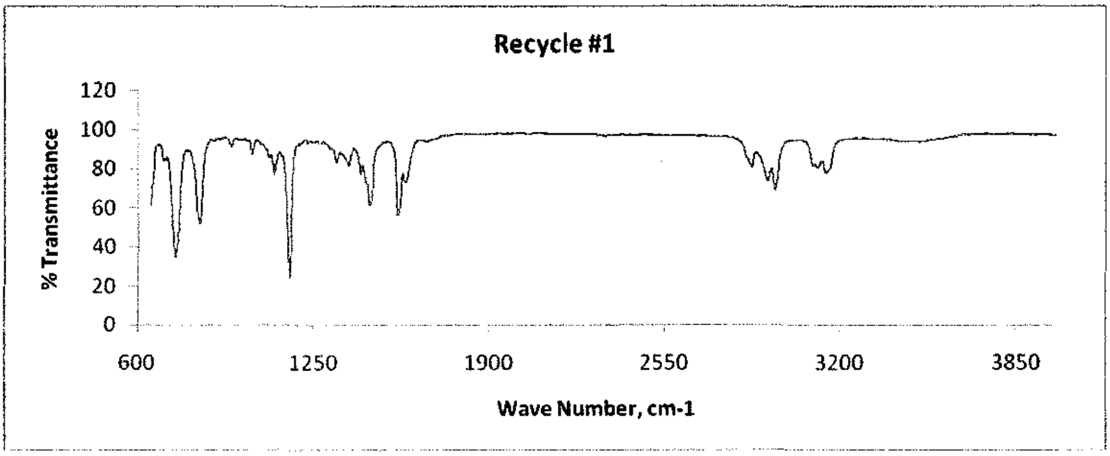


Figure 4.2: FT-IR Spectroscopy for [Bmim][FeCl$_4$] Recycle #1

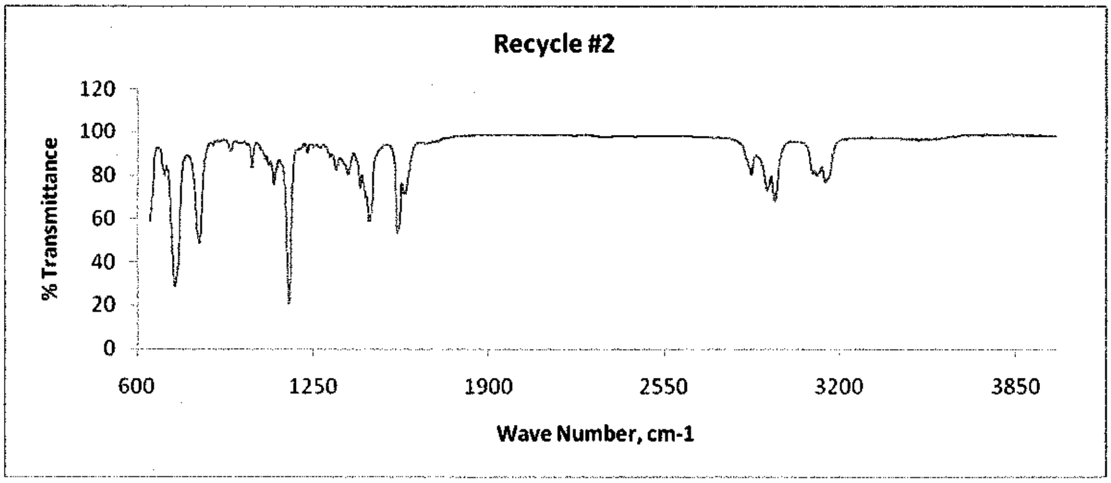


Figure 4.3: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #2

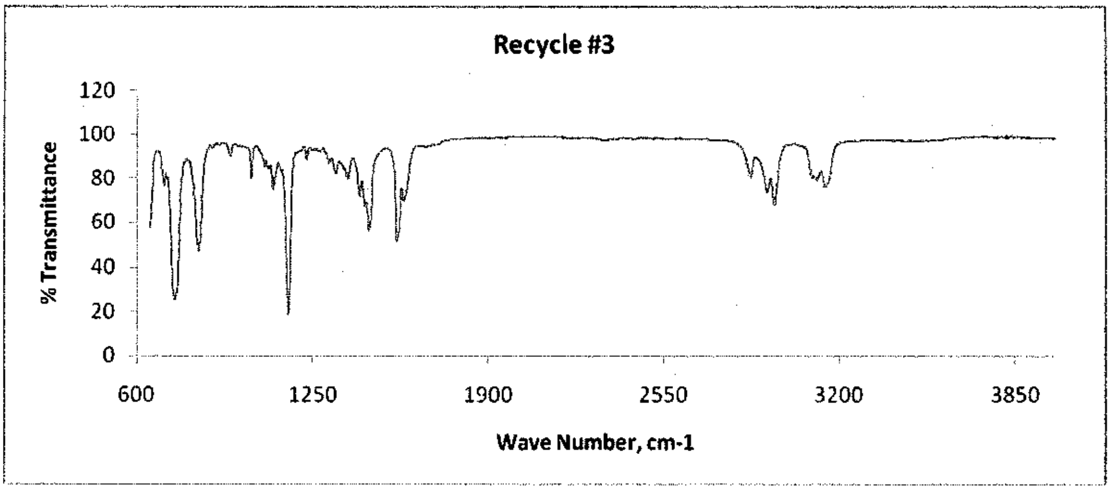


Figure 4.4: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #3

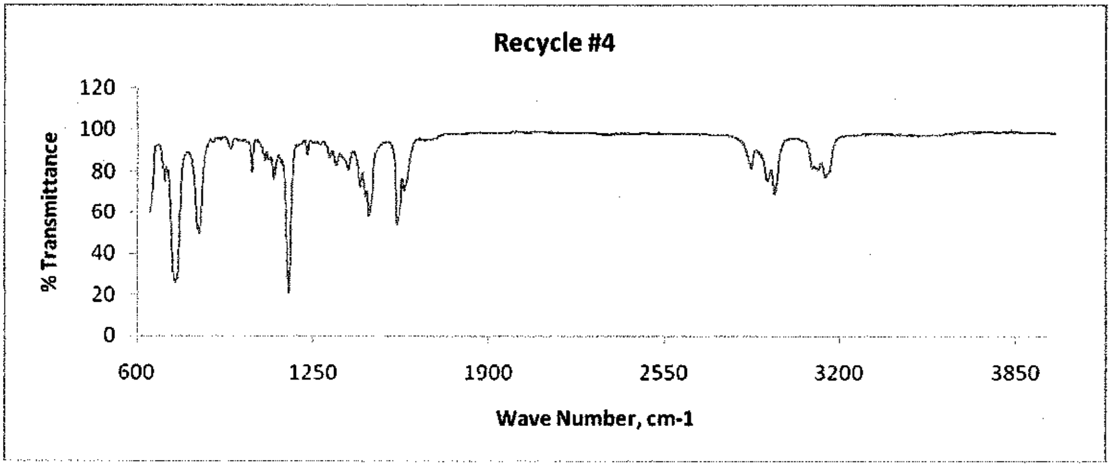


Figure 4.5: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #4

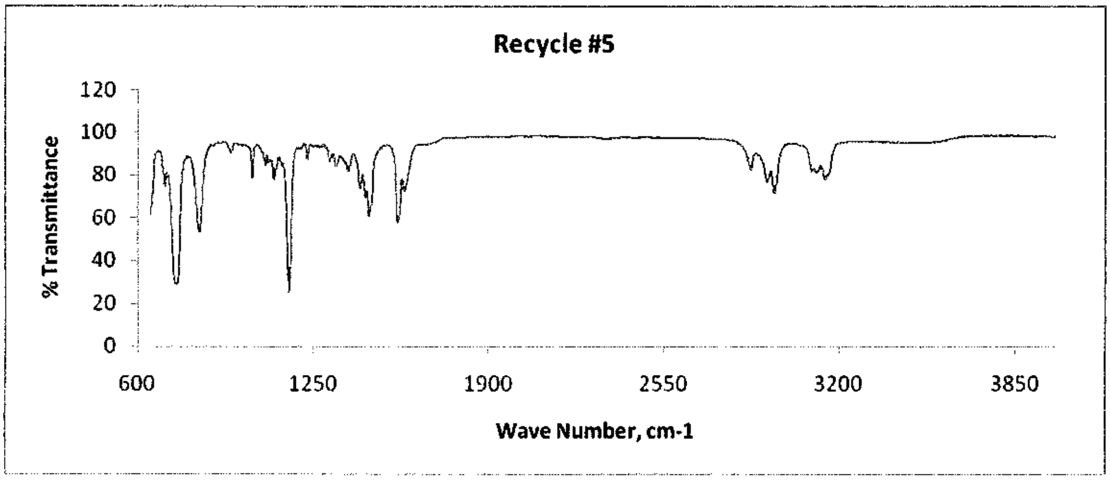


Figure 4.6: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #5

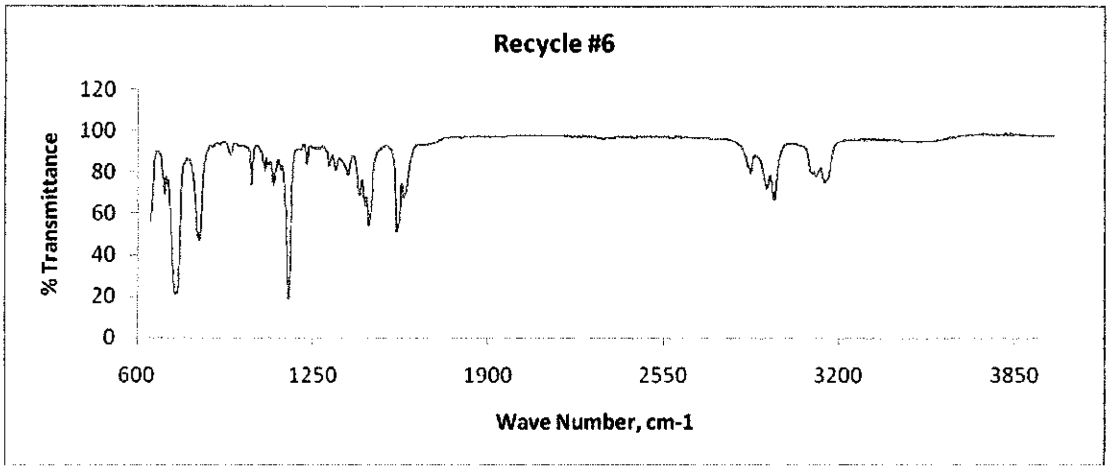


Figure 4.7: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #6

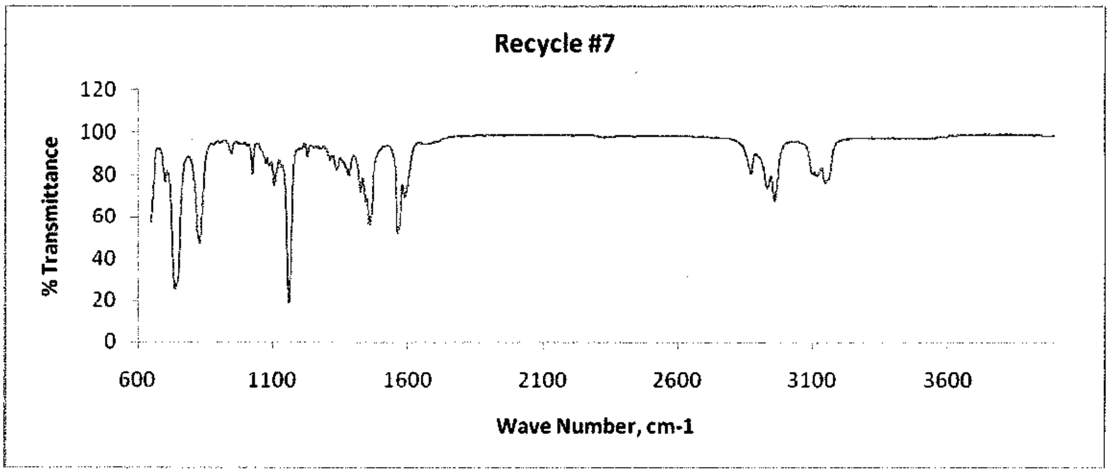


Figure 4.8: FT-IR Spectroscopy for [Bmim][$FeCl_4$] Recycle #7

Based on the FT-IR Spectroscopy result above, when we compare pure [Bmim][$FeCl_4$] with the recycled [Bmim][$FeCl_4$], their chemical structures remain the same even after 7 cycles. It is clear that [Bmim][$FeCl_4$] can be recycle few times without being decompose or experiencing any change in its bonding and structure. However, in order to look deeply into its performance in removing sulfur from the model oil, we need to look at the CHNS result.

We can divide it into two parts first, the degree of desulfurization based on the sulfur content in the model oil (dodecane +DBT) and also by looking at the sulfur content in [Bmim][$FeCl_4$] in order to observed its ability to remove sulfur as the number of cycle increase.

Table 4.1: CHNS Result on Sulfur Content in Dodecane After Desulfurization

Dodecane (Model oil)

Number of Recycle	Sulfur Content (%)	Degree of Desulfurization (%)
0	0.013	97.96
1	0.068	89.36
2	0.127	80.13
3	0.214	66.51
4	0.268	58.06
5	0.287	55.09
6	0.289	54.77
7	0.290	54.62

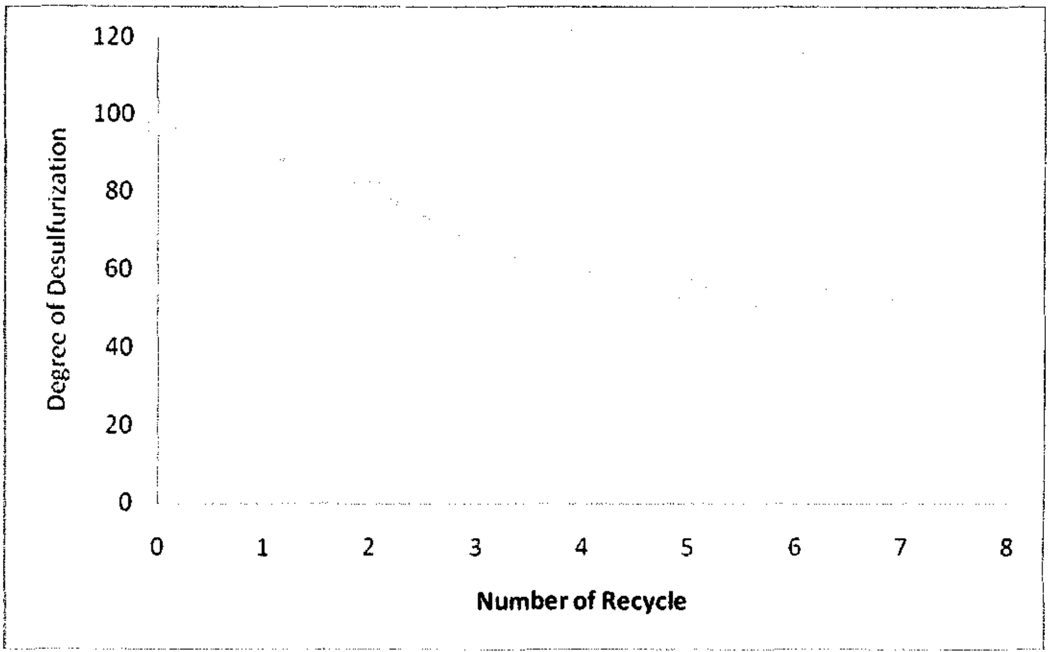


Figure 4.9: Graph of degree of desulfurization of Dodecane versus Number of recycle

Table 4.2: CHNS Result on Sulfur Content in [Bmim][FeCl$_4$]

[Bmim][FeCl$_4$] (Ionic Liquid)

Number of Recycle	Sulfur Content (%)
0	0.328
1	0.818
2	1.074
3	1.491
4	1.724
5	1.727
6	1.730
7	1.731

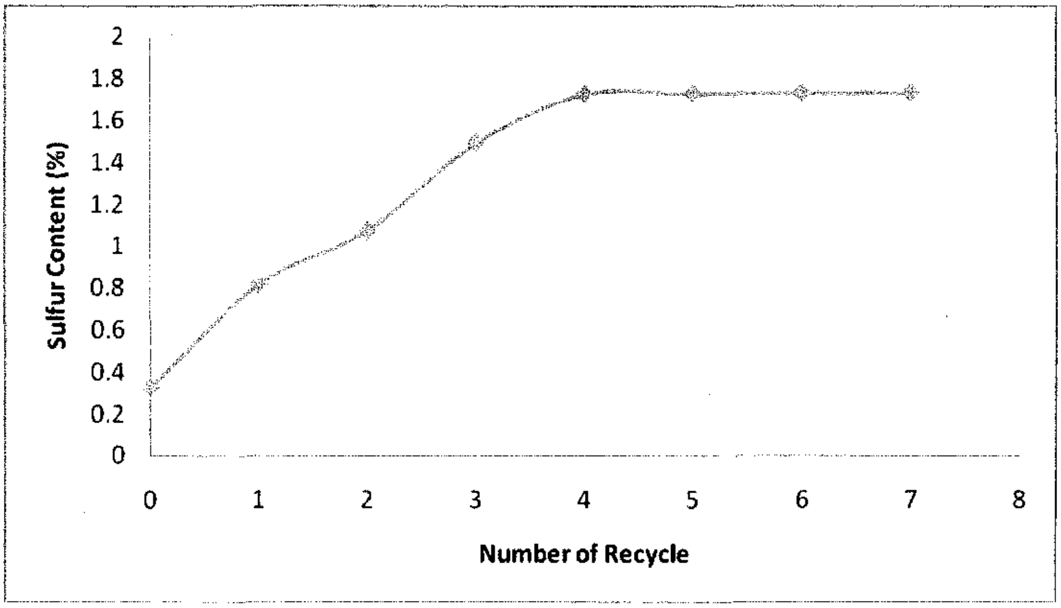


Figure 4.10: Graph of Sulfur Content in [Bmim][FeCl$_4$] versus Number of recycle

Based on the CHNS results obtained, sulfur content in fresh dodecane stock increases as the number of cycle increase. Based on the graph in Figure 4.2, we can see that the degree of desulfurization decreases and become almost constant after 5 times [Bmim][FeCl$_4$] is recycled and vice versa in Figure 4.3, Sulfur content in [Bmim][FeCl$_4$] increases and become constant after about 5 cycles. This is due to maybe the Ionic Liquid has become saturated with sulfur and cannot extract anymore sulfur from the model oil. At this point, in order to increase the degree of desulfurization, we need to regenerate [Bmim][FeCl$_4$] or use a fresh Ionic Liquid.

4.2 Recovery Performance – Contact Time Variation

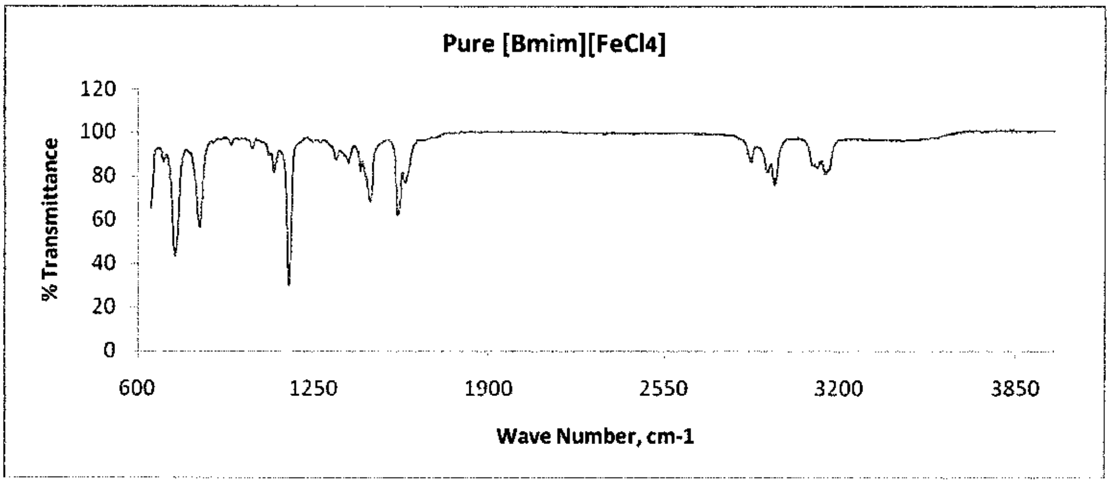


Figure 4.11: FT-IR Spectroscopy for Pure [Bmim][FeCl$_4$]

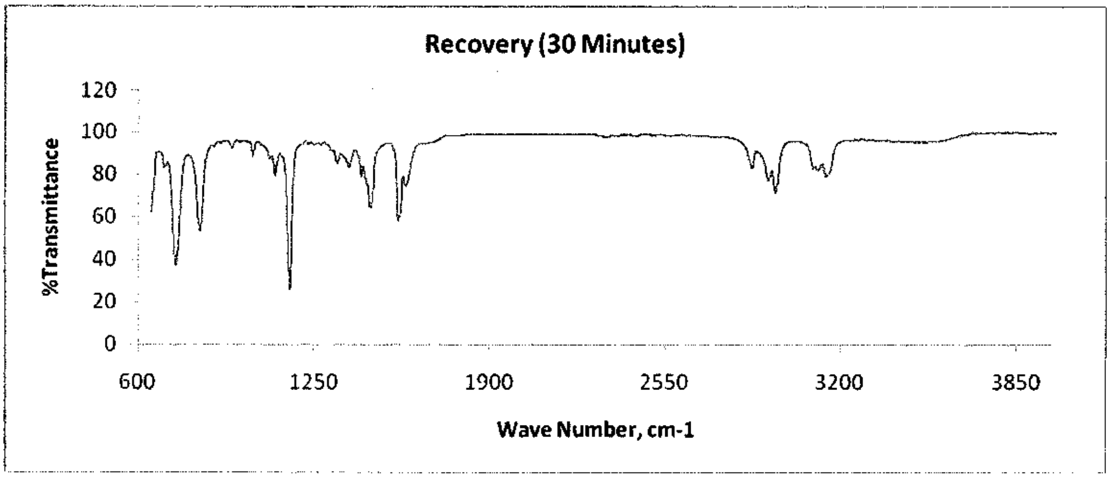


Figure 4.12: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (30min)

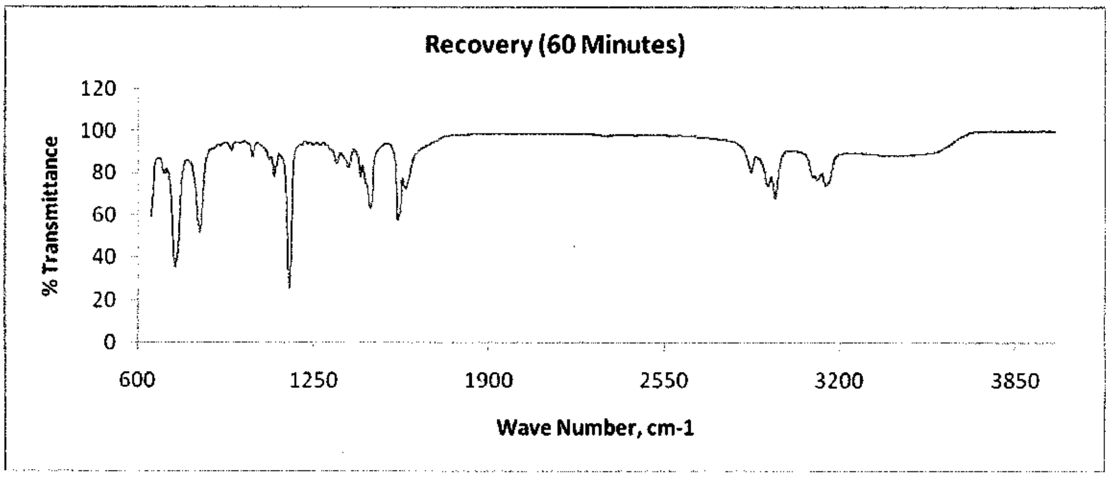


Figure 4.13: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (60min)

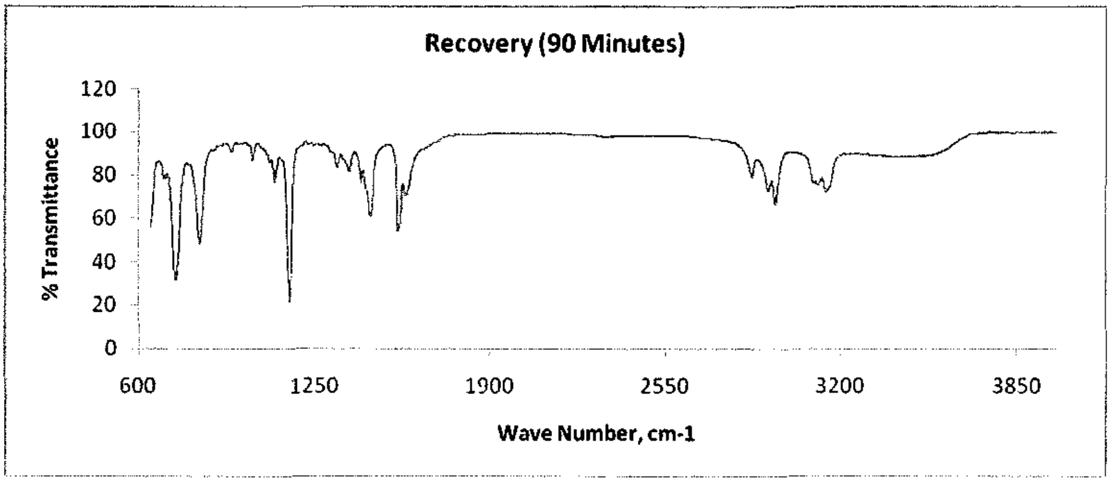


Figure 4.14: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (90min)

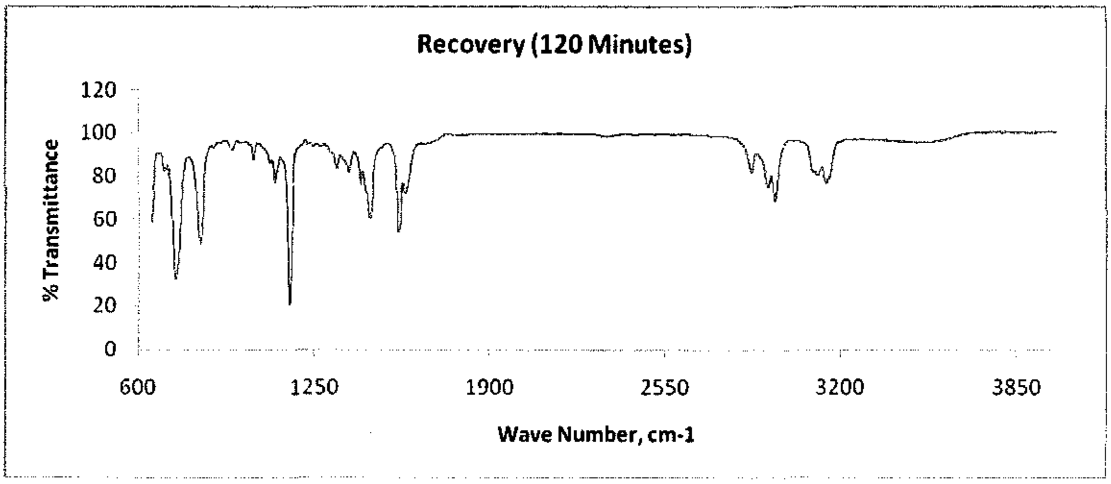


Figure 4.15: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (120min)

Referring to the FT-IR spectroscopy, when we compare pure [Bmim][FeCl$_4$] to the recovered [Bmim][FeCl$_4$] for different contact time the peaks are almost the same. This means that the chemical structure of the [Bmim][FeCl$_4$] does not change after being mixed with the hexane to extract sulfur from it even up to two hours.

For the recovery performance, by using the equation stated in the calculation part earlier, we manage to get as high as 31% of sulfur removal.

Table 4.3: Recovery Efficiency for Different Contact Time

Contact Time (Minutes)	Recovery Efficiency (%)
15	28.05
30	30.53
60	31.42
90	31.58
120	31.56

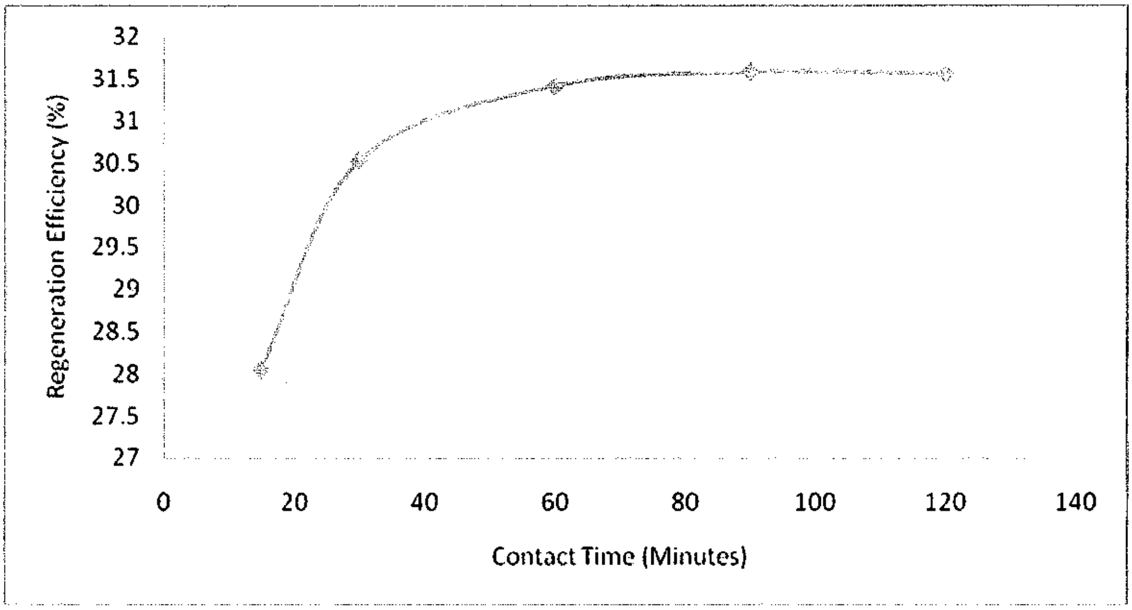


Figure 4.16: Graph of Recovery Efficiency versus Contact Time

Looking at the trending of the recovery performance in Figure 4.4, it shows an increase of efficiency at the beginning around 15 minutes to 70 minutes and become constant after that. We can conclude that the best contact time would be at 80 minutes. The trending become constant as the contact time increases maybe due to saturation of the solvent, Hexane towards the sulfur from the sulfur-loaded [Bmim][$FeCl_4$]. The hexane could not extract more sulfur even we increase the contact time because of the saturation limit.

4.3 Recovery Performance – Different Solvent Ratio

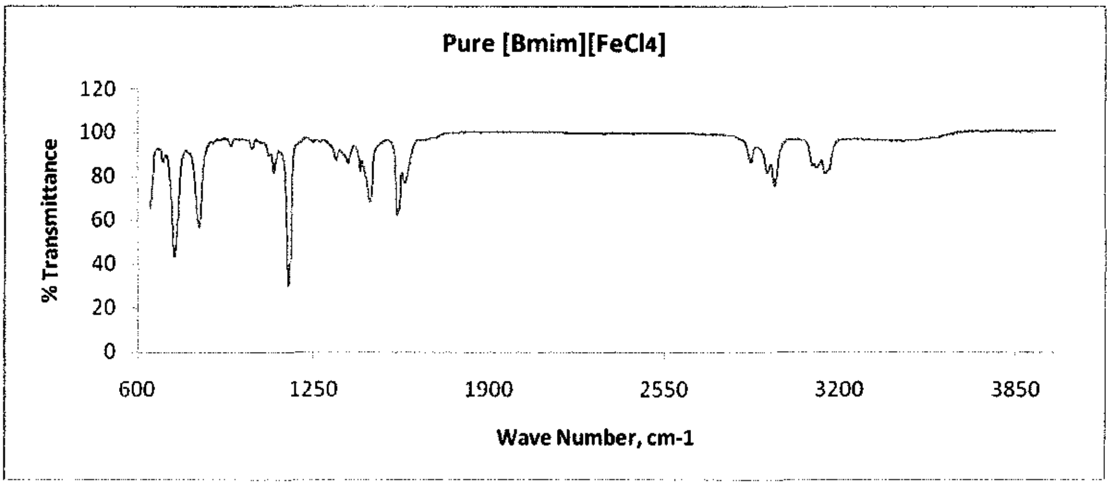


Figure 4.17: FT-IR Spectroscopy for Pure [Bmim][FeCl$_4$]

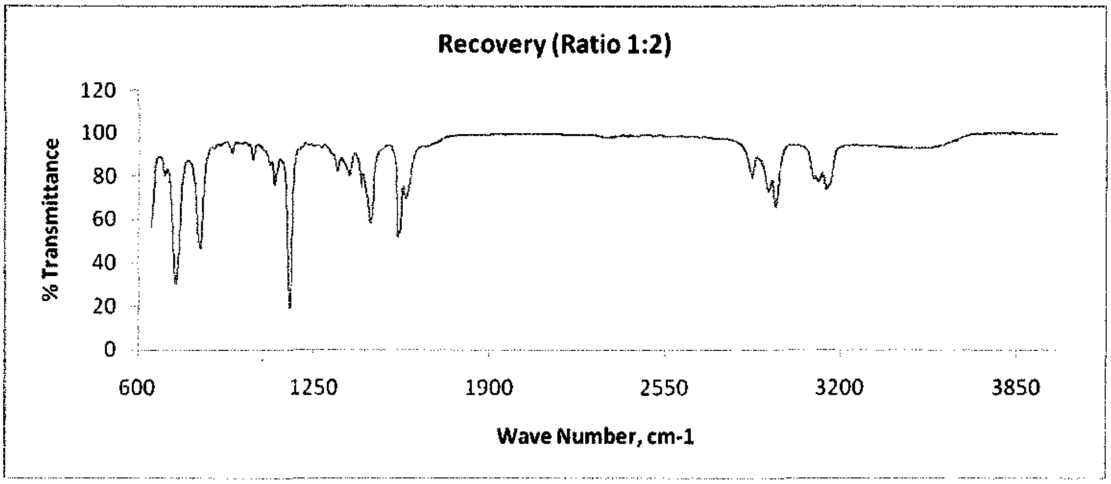


Figure 4.18: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (1:2)

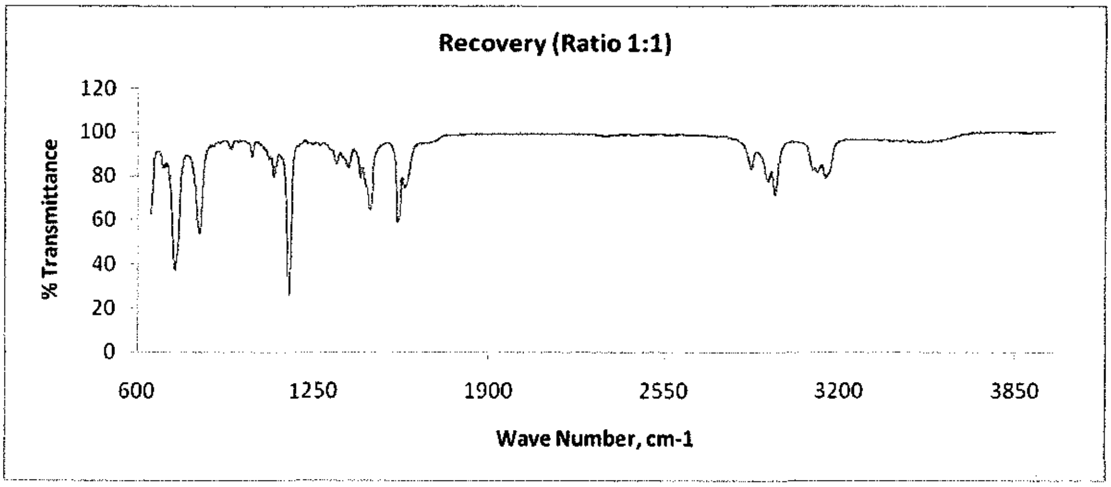


Figure 4.19: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (1:1)

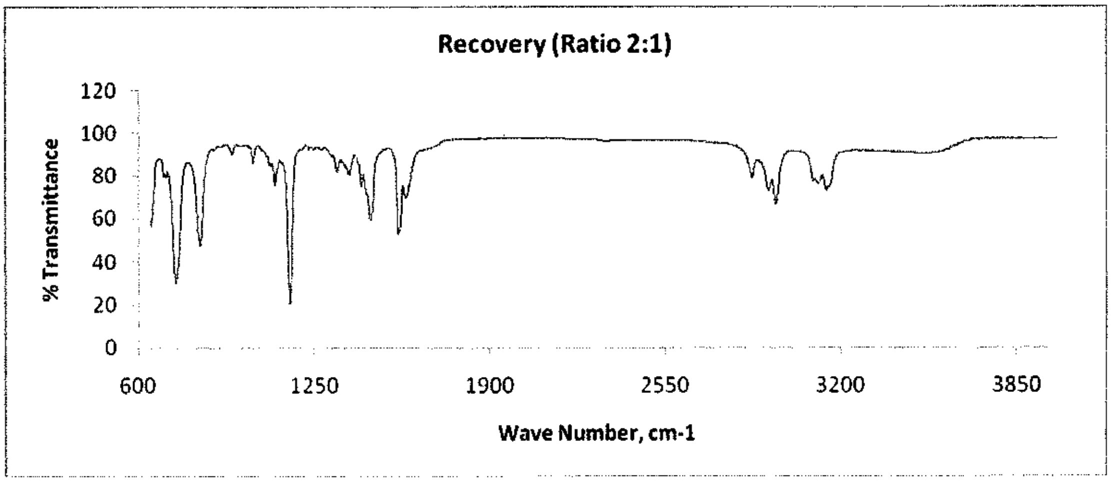


Figure 4.20: FT-IR Spectroscopy for Recovered [Bmim][$FeCl_4$] (2:1)

Based on the FT-IR spectroscopy, the peaks are almost the same for all recovered [Bmim][$FeCl_4$] sample if we compared to the pure [Bmim][$FeCl_4$]. This means that the chemical structure of the [Bmim][$FeCl_4$] does not change after being mixed with the hexane by different ratio. It shows that hexane does not react with the [Bmim][$FeCl_4$] furthermore we know that they are completely immiscible. Any amount of excess solvent, hexane would not affect the structure and bonding of [Bmim][$FeCl_4$].

For the quantitative analysis using CHNS, the same method of calculation is used in order to determine the recovery efficiency. We managed to obtain as high up to 43% of sulfur removal when we use more solvent with the ratio of 1 to 2.

Table 4.4: Recovery Efficiency for Different Solvent Ratio

Sulfur-loaded [BMIM]FeCl4 to Hexane Ratio	Recovery Efficiency (%)
1:2	43.42
1:1	30.53
2:1	7.37

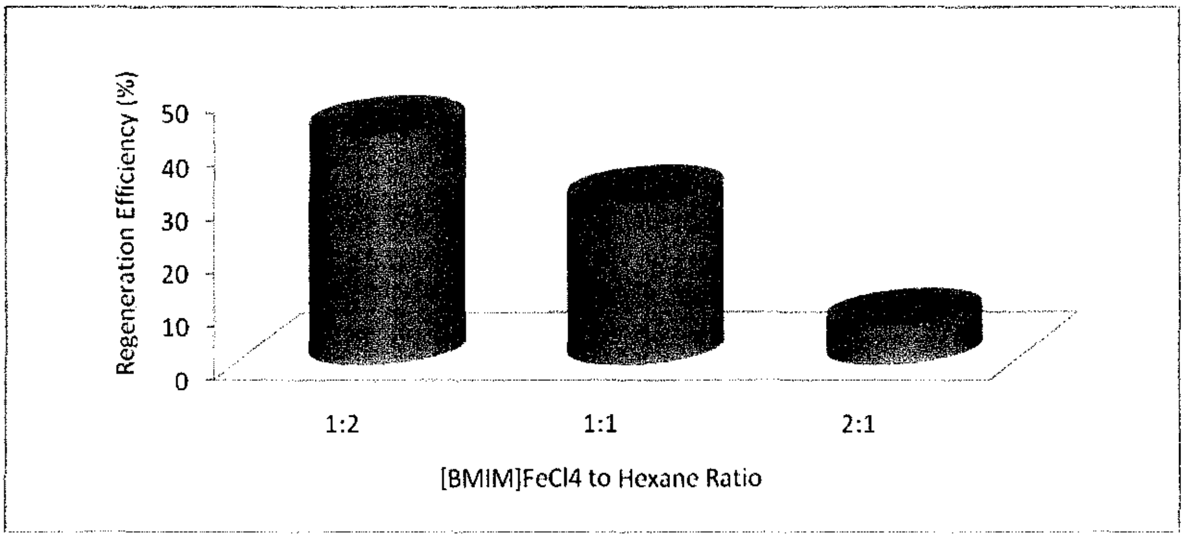


Figure 4.21: Bar chart of Recovery Efficiency versus [Bmim][FeCl$_4$] to Hexane Ratio

Based on the Bar Chart representation above, it is very clear that with the presence of extra solvent, we are able to increase the recovery efficiency quite high compared to the other factors such as contact time. However, it is not economically feasible to use more solvent since it will increase the cost of separation due to chances of cross-solubility occurring are high. Plus there is also a safety and environment issues if we use too much organic solvent in the industry. However, if we use too little solvent, it could not extract excess sulfur and bring down the performance as low as to 7%. Based on the results, with the ratio 1 to 1, we are still able to get a high performance of recovery. Therefore, the best ratio that is economically attractive and safe would be the 1 to 1 ratio of ionic liquid to solvent.

4.3 Recovery Performance – Process Temperature Variation

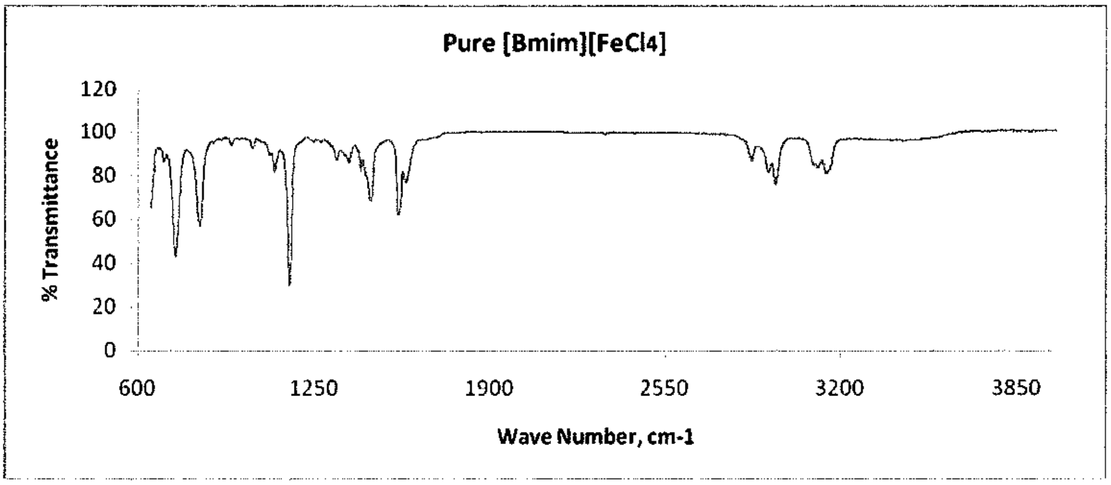


Figure 4.22: FT-IR Spectroscopy for Pure [Bmim][FeCl$_4$]

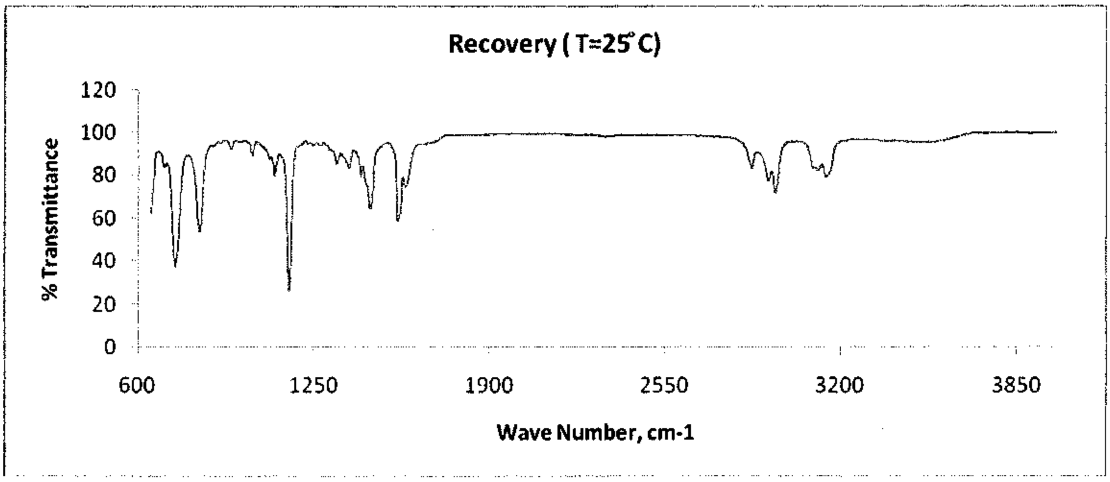


Figure 4.23: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (25°C)

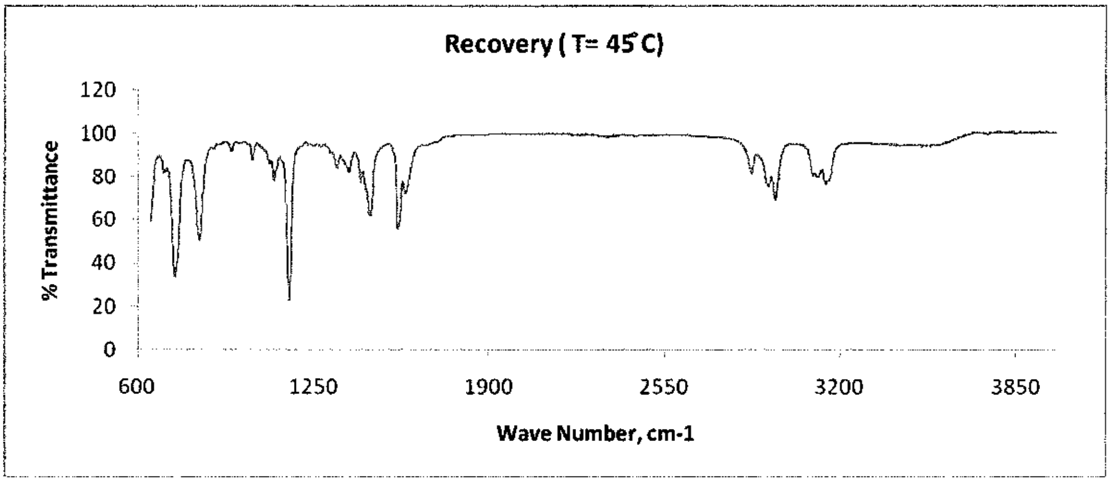


Figure 4.24: FT-IR Spectroscopy for Recovered [Bmim][FeCl$_4$] (45°C)

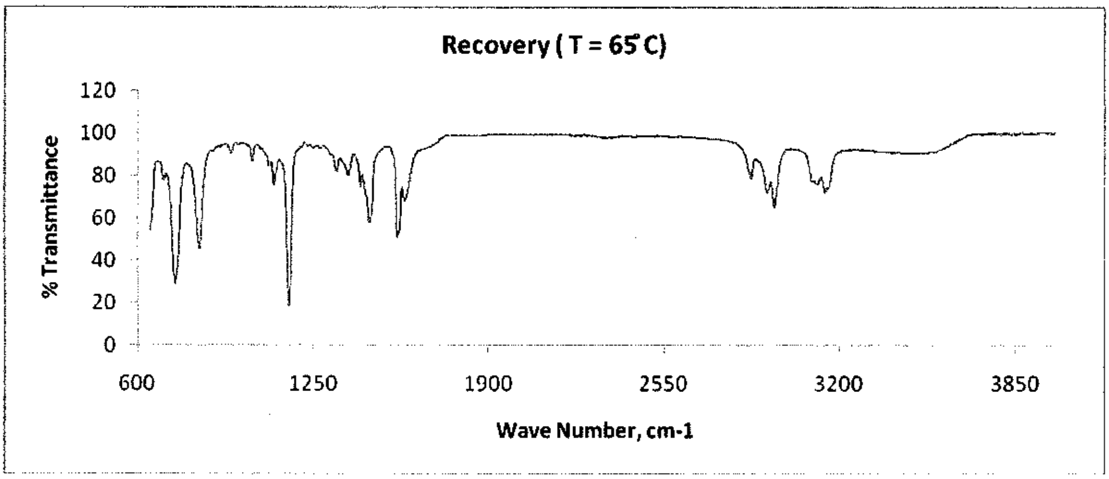


Figure 4.25: FT-IR Spectroscopy for Recovered [Bmim][$FeCl_4$] (65°C)

Based on the FT-IR spectroscopy, if we compare the peaks for the recovered [Bmim][$FeCl_4$] at different temperature with the pure [Bmim][$FeCl_4$], they are almost the same for all. This means that the chemical structure of the [Bmim][$FeCl_4$] does not change even if we increase the temperature and being mixed with the hexane. This is due to the characteristic of the ionic liquid itself were they have an extremely low vapour pressure, high thermal stability and high boiling point. Therefore, by applying heat to the mixture of solvent and [Bmim][$FeCl_4$], it would not affect the chemical structure and interaction.

For the efficiency of sulfur removal, we managed to get about 32% performance of the recovery as when the temperature increase.

Table 4.5: Recovery Efficiency for Different Temperature

Temperature (°C)	Recovery Efficiency (%)
25	30.53
45	31.58
65	31.84

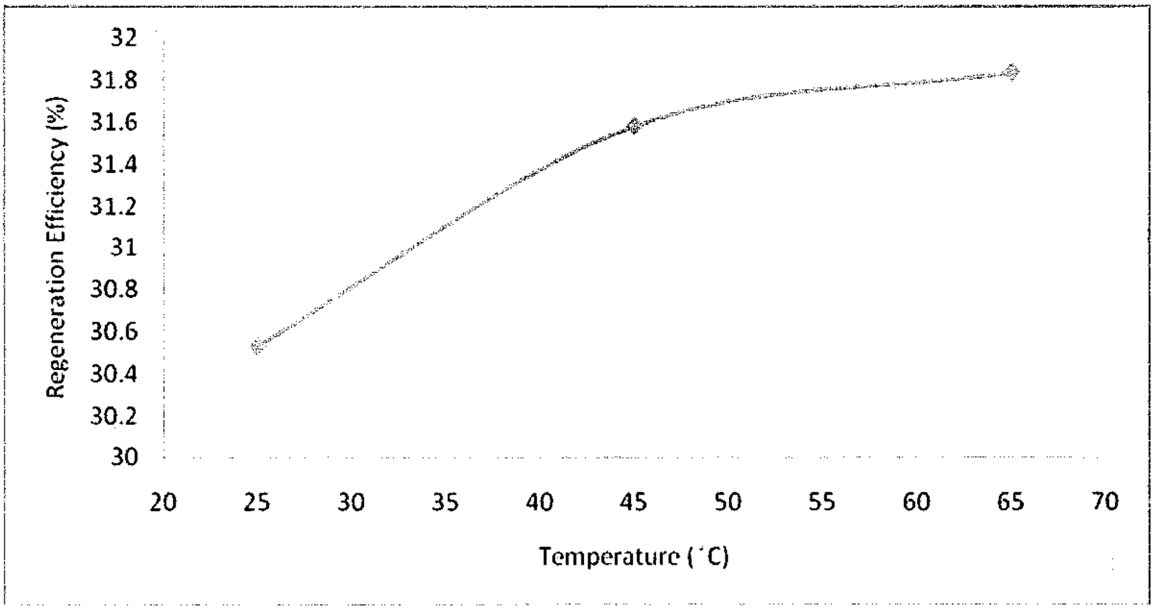


Figure 4.26: Graph of Recovery Efficiency versus Temperature Variation

Based on the figure above, it can be concluded that, as the recovery operating temperature increases, the performance increases as well. This is due to extra kinetic energy provided by the heat to the molecules which make it easier for the hexane to extract the sulfur from the ionic Liquid. However, we need to pay attention to how high the temperature goes. It should be less than 69° C which is the Hexane boiling point because we do not want the hexane to vaporize. When the hexane vaporize, the efficiency to extract sulfur will decrease because the contact time between both liquid mixtures are short. The reason why we are considering conducting the process at high temperature is because in the industry, it is not advisable to bring down the process fluid (petroleum product) temperature to normal ground temperature. It will be costly in to the cooling system and also, will increase the energy consumption when we need to get the temperature back to high for next processes in the plant.

CHAPTER 5

CONCLUSION & RECOMMENDATION

5.1 Conclusion

As a conclusion, all the main deliverables for this semester had been achieved. For this research project, there two main objectives, that is to recover and recycle [Bmim][FeCl$_4$] after serving it purpose as solvent in extractive desulfurization where this will eventually benefits greatly in the field of Ionic Liquid and its application in chemical processes. Based on the scope of work and literature review, this project is completed within the time frame given and it is feasible to be conducted. As the conclusion, [Bmim][FeCl$_4$] are able to be recycle few times up to 4-5 times before its efficiency to extract sulfur decreases and become constant. Furthermore, the chemical structure of the ionic liquid does not change after recycling few times and also after being recovered with co-solvent hexane. The best contact time would be 80 minutes and the best operating temperature is 65 °C. The most preferred ratio is 1 to 1 ratio as it is economically feasible and with satisfying performance.

5.2 Recommendation & Expected Future Work

After completion of this Research project, the aim is to improve certain area in the experiments. Analysis and comparison of result will show how the recovery process can be further optimized. As a recommendation, we should try to manipulate the operating pressure in order to come out with the best and highest performance of the recovery process. Besides that, since there are many solvents in the market and industry, we should use different type of solvent and determine on which would give the highest percentage of recovery. Last but not least, further studies and continuous experiment on recovery and regeneration of other Ionic Liquids with high degree of desulfurization should be conducted to provide useful information and comparison for economical feasibility of different Ionic Liquids can be made.

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APPENDICES

Chemicals, Tools and Equipments

Chemicals

List of Chemicals

No.	Items	Purpose
1	1-butyl-3-methylimidazolium chloride	Synthesis
2	Anhydrous iron chloride	
3	Model oil	Extractive desulphurization
4	Dibenzothiophene	
5	Hexane	Regeneration

Tools and Equipments

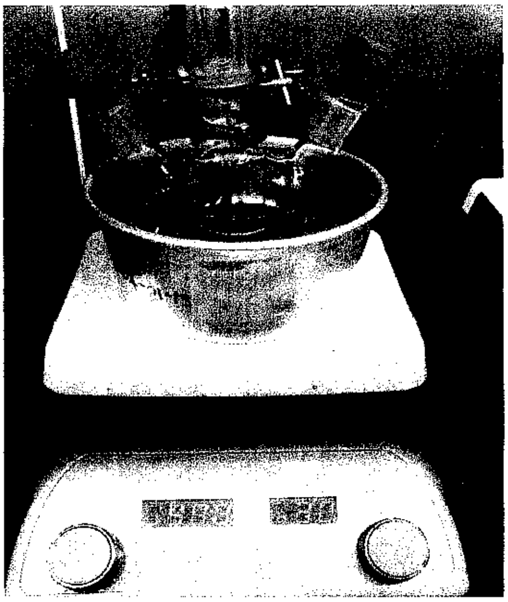
List of Tools and Equipments

No.	Items	Purpose
1	3-neck Flask	• Synthesis • Extractive desulphurization • Storing of ILs and samples • Regeneration • Recycling
2	Beakers	
3	Conical Flask	
4	Vials	
5	Syringes	
6	Retort stand and its Clamp	
7	Magnetic Stirrer	
8	Weigher	
9	Raman Spectroscopy	
10	Total Sulphur Analyzer	

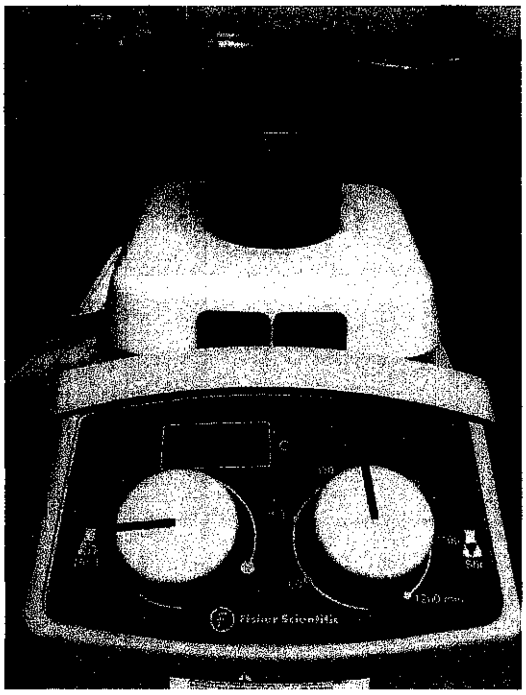
Physical Properties of [Bmim][FeCl$_4$]

[BMIm][FeCl$_4$] Physical Properties	
CAS number	359845-21-9
Molecular Formula	$C_8H_{15}Cl_4FeN_2$
Formula Weight	336.88
Melting Point	17°C
Density	1.30 g/cm^3
Refractive Index	Not available
Appearance	Brown
Risk Statements	Irritating to eyes. respiratory system and skin
	Harmful in contact with skin and if swallowed
Safety Statements	Wear suitable protective clothing. gloves and eye/face protection
	In case of contact with eyes. rinse immediately with plenty of water and seek medical advice

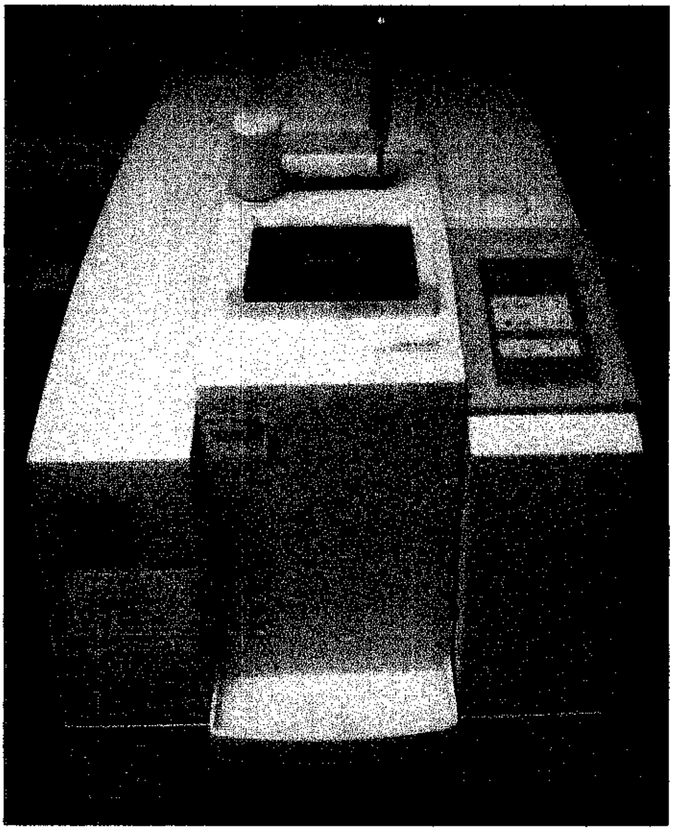
Experimental Work



Extractive Desulfurization Process



Recovery Process (LLE)



Fourier Transform Infrared Spectrometer (FT-IR)

Carbon, Hydrogen, Nitrogen & Sulfur Analyzer (CHNS)