CERTIFICATION OF APPROVAL

Development of Ionic Liquids for CO₂ Absorption

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 work except as specified in the reference and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABBREVIATION & NOTATIONS

[bmim][PF ₆]	Binaphtyldiimine Hexafluorophosphate
CO ₂	Carbon Dioxide
DEA	Diethanol Amine
FAB-MS	Fast Atom Bombardment Mass Spectroscopy
FT-IR	Fourier-transform Infra Red
H_2CO_3	Carbonic Acid
H_2S	Hydrogen Sulfide
КОН	Potassium Hydroxide
MDEA	Methyldiethanol Amine
MEA	Monoethanol Amine
NaOH	Sodium Hydroxide
NMR	Nuclear Magnetic Resonance

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ABSTRACT

The increasing manmade carbon dioxide (CO₂) emission to the atmosphere is expected to rise tremendously in the next 100 years, and this will impact the environment seriously in the future. Thus, a substantial focus has been brought forward on improving techniques for CO₂ sequestration. Accordingly, the basic objective of this project is to develop several ionic liquids for more optimum carbon dioxide gas absorption and to compare the synthesized liquids versus three commercial ionic liquids, through laboratory experiments and research. Currently used absorbents for CO₂ absorption such as amines have been found to be effective in the removal CO₂; however, they have notable flaws i.e. high energy requirement for reactivation, corrosion due to formation of acid, and amine degradation and loss. On the other hand, ionic liquids are in liquid state under atmospheric conditions at room temperature with specific properties that make them extremely effective as CO_2 absorbents. The ionic liquid that is to be developed through this project involves amines functionality group attached to imidazolium ions. Imidazolium based ionic liquids will be prepared through the variation of cation and anion species. The liquids produced; together with some commercially used absorbents; were tested for CO₂ absorption. Tests using FT-IR spectroscopy were conducted in the end to gauge the success of the experiments. Through this project, a total of six liquids have been produced and tested for CO₂ absorption. Comparisons were made with three commercial ionic liquids. All tests yielded positive results where IMI-01 absorbed the most CO₂ (1.55 g CO₂ absorbed/mole liquid) among the synthesized liquids while C-01 was the best CO_2 absorbent (1.80 g CO_2 absorbed/mole liquid) among all the ionic liquids tested. FT-IR results also show that amine (Wave Number: 3400-3250 cm⁻¹) was present in the pre-absorbed liquids tested and CO_2 (Wave Number: 1820-1650 cm⁻¹) was present in the post-absorbed liquids tested. As a conclusion, the ionic liquids that have been produced in this experiment are all capable of CO_2 absorption.

CHAPTER 1: INTRODUCTION

It has been estimated that man-made emissions of CO_2 into the atmosphere have resulted in an increase in CO_2 concentration of at least 30% over pre-industrial age concentrations. CO_2 emissions over the next 100 years could see an increase of 3 to 4 times from current levels (University of Wyoming, 2004). While there is considerable debate over the environmental impact of CO_2 emissions on climate change and global warming, there is a general consensus that tripling or quadrupling CO_2 emissions will have a serious environmental impact in the future. Accordingly, there has been a substantial focus on improving techniques for CO_2 sequestration or capture. Today, companies are required to remove or substantially reduce CO_2 levels in exhaust streams before they are vented to the atmosphere.

Fossil fuel is one of the most important sources of energy in the world right now. This includes petroleum, coal and natural gas. In the oil and gas industry, natural gas exists in two forms, which are sweet gas (lowly contaminated) and sour gas (highly contaminated, usually by CO_2 and H_2S). To make use of this sour gas which is available in abundance, separation processes are required to first purify them.

1.1 BACKGROUND

Currently, a commonly used CO_2 separation process involves amine absorption. In brief, flue gas streams and natural gas streams are bubbled through an amine solution and the CO_2 in these streams becomes bound to the amine groups in the solution. Consequently, the CO_2 content in the resulting gas stream is significantly reduced. Amine absorption is an effective technique to remove CO_2 . In a commercial process, concentrations of up to 30 wt% of MEA have been employed successfully to remove 80-90% of CO_2 from a feed gas (Mardziah, 2005).

However, this technique has notable flaws. First, once the amine solution is saturated, the process to reactivate the solution (i.e., remove the bound CO_2 from the amine groups in the solution) for reuse requires a high amount of energy. Second, this process has a tendency to corrode equipment due to the

formation of H_2CO_3 , which is an acid, through the reaction of CO_2 with the water molecules. Third, over a short period of time, the amine solution loses viability through amine degradation and loss due to its dependence on water molecules for the formation of carbamate makes the solution less effective in the absorption of CO_2 .

1.2 PROBLEM STATEMENT

Amine solutions and other common absorbents have notable flaws in terms of absorbing CO₂, namely:

- High energy requirement for reactivation
- Corrosion due to formation of H₂CO₃
- Amine degradation and loss due to dependence on water

1.3 OBJECTIVES & SCOPE OF DUTY

Thus it is the objective of this project to develop ionic liquids for a more optimum CO_2 gas absorption and to compare commercial ionic liquids versus the ionic liquids developed through laboratory experiments and research.

CHAPTER 2: LITERATURE REVIEW & THEORY

2.1 ABSORPTION THEORY

Absorption is a separation process where the two contacting phases are a gas and a liquid. The process involves molecular and turbulent diffusion or mass transfer of a solute in the gas phase into a stagnant absorbent in liquid. The reverse of absorption is called stripping or desorption, and the same theories and basic principles hold (Geankoplis, 2003).

2.2 AMINE SOLUTIONS

The amine solutions primarily used in industries for natural gas purification are Monoethanol Amine (MEA), Methyldiethanolamine (MDEA) and Diethanolamine (DEA). They are considered to be good in removing CO_2 and H_2S through scrubbing but are water dependent and not cost effective due to the gradual loss of the solution and corrosion.

Amines can be considered as derivatives of ammonia where one or more hydrogen(s) have been replaced by alkyl or aryl groups. Amines can be divided into four groups which are; **Primary amines** which have one alkyl or aryl group bonded to the nitrogen, for example MEA and butylamine, **Secondary amines** which have two groups on the nitrogen, for example DEA, **Tertiary amines** which have three groups, for example MDEA and dimethylcyclopentylamine, and **Quaternary ammonium salts** which have four groups attached to the nitrogen, for example tetramethylammonium hydroxide (Hornback, 1998).



Figure 1: Tertiary amine & Quaternary ammonium salt examples

2.3 IONIC LIQUIDS

Ionic liquids are typically organic salts that, in their pure state, are liquid under atmospheric conditions at room temperature. They have unusual properties that suggest they could be extremely effective as carbon dioxide absorbents, possibly replacing current amine-based technology to capture carbon dioxide from power plants stacks. Unlike amines, which are corrosive and costly to operate, organic salts are typically benign, and can be disposed of in landfills. (Techline, 2004)

The ionic liquid which is to be developed is to be "task-specific", in which the ionic liquid will be very specific in removing CO_2 . The cation of this ionic liquid is an imidazolium ion to which a primary amine moiety is covalently tethered. This salt will readily and reversibly absorb CO_2 . (Bates et al., 2002)

Imidazolium ion is used in making this ionic liquid due to the melting point of the salt compound is below room temperature, allowing the salt solution to be in stable liquid form and not volatile. Previous studies on imidazolium ion based ionic liquids show that a wide variety of solutes can be extracted from [bmim][PF₆] with CO₂ with recovery rates greater than 95%. (Blanchard et al., 2001)

Thus, the task focuses on developing or synthesizing such an ionic liquid in the laboratory and testing the effectiveness of the ionic liquid to absorb CO_2 .

2.4 PROCESS CHEMISTRY

2.4.1 Absorption

The direct chemistry of CO_2 sequestration using absorbents is the carbamatereaction, which results in the formation of carbamate. H₂S and CO₂ are "acid gases" because they dissociate in water or aqueous solutions to form weak acids. Aqueous amines are bases. Thus for CO_2 removal, the basis of the chemistry is a combination of acid-base-reaction and carbamate reaction for primary and secondary amines, while only acid-base-reaction for tertiary amines. Removal of CO_2 using ionic liquids does not involve acid-base-reaction due to the fact that there are no water molecules for CO_2 to dissociate into and behave like a weak acid. The acid-base-reaction is kinetically slow because of the carbonic acid dissociation to the bicarbonate is relatively slow. This is different from the carbamate-reaction where the rate of CO_2 absorption is very rapid.

2.4.2 CO₂ absorption via Acid-Base-Reaction

$$CO_2 + H_2O - H_2CO_3$$

Using Amines:

$$H_2CO_3 + [Amine] \longrightarrow HCO_3 + [Amine]^+$$

2.4.3 CO₂ absorption via Carbamate-Reaction

Primary and Secondary Amines:

$$CO_2 + 2 [Amine] = [Amine]^+ + [Amine] CO_2^-$$

Ionic Liquid:

$$CO_2 + 2 [Anion]^+ = \left[Anion]^+ + [Amine]CO_2 \right]^+$$

2.4.4 Ionic Liquid Synthesis

Basically, there are 2 general reaction processes that are done to produce each liquid i.e. the **Imidazole + Amine** reaction and the **Ion Exchange** reaction. The **imidazoles** are **methylimidazole** and **butylimidazole**; the **amines** are **ethylamine** and **propylamine**, while the **anions** are **methylsulfonate** and **benzenesulfonate**. Reactions involved in the production of each ionic liquid will be further elaborated in the Chapter 4: Results & Discussion.

2.5 INFRARED SPECTROSCOPY

Prior to the introduction of NMR spectroscopy, infrared (IR) spectroscopy was the instrumental method most often used for structure determination of organic compounds. IR still retains an important place in the chemist's inventory because of its usefulness in identifying the presence of certain functional groups within a molecule.

Infrared radiation comprises the portion of the electromagnetic spectrum between microwaves and visible light. Two derived units commonly employed in IR spectroscopy are the micrometer and the wave number. One micrometer (μ m) is 10⁻⁶ m, and IR spectra record the region from 2.5 μ m to 16 μ m. Wave numbers are reciprocal centimetres (cm⁻¹), so that the region 2.5–16 μ m corresponds to 4000–625 cm⁻¹. (Atkins et al., 2002)

Most common functional groups absorb infrared energy at characteristic frequencies in the infrared spectrum. The presence of a peak at a particular frequency can be used to determine whether an unknown molecule contains a certain functional group.

Group	Position (cm ⁻¹)
—О-Н	3550-3200
—_N—Н	3400-3250
≡с−н	3300
 —С—Н	3100-3000
Ċн	3000-2850
О Ш —С-Н	2830-2700
—C≡N	2260-2200
—C=C—	2150-2100
0 	1820-1650
c=c	1660-1640
	1600-1450
NO ₂	1550 and 1380
	1300-1000

Table 1: Important Absorption Bands in the Infrared Spectral Region

(Hornback, 1998)

CHAPTER 3: METHODOLOGY & PROJECT WORK

3.1 SYNTHESIS OF IONIC LIQUIDS

The solutions are produced using the following general procedures. These procedures have been based on the basic procedures outlined in the American Chemical Society Journal entitled CO_2 Capture by a Task-Specific Ionic Liquid (Bates et al., 2002).

A. Imidazole + Amine reaction

The imidazole component and the amine component are reacted in ethanol as shown in Figure 1. The amount of reactants used is 1mmol imidazole to 1.2mmol amine. The reactants will be stirred at 250rpm at temperature of 90° C for 3 days.



Figure 2: Experimental Set-up for Synthesis of Ionic Liquids

B. Removal of ethanol in vacuo using BUCHI Rotavapor

The liquid is put into the set-up with water bath temperature at 80°C for 30 minutes.

C. Bringing to ~pH 8

KOH dissolved in minimal amount of water (<5ml) is added in small portions till the liquid achieves ~pH 8 which is confirmed using litmus paper.

D. Removal of water with KBr in vacuo using BUCHI Rotavapor

The liquid is put into the set-up with water bath temperature at 95oC for 15 minutes.

E. Extraction of residue

Residues (if any) are separated from the liquid by adding 20ml of ethanol with 20ml of tetrahydofuron to the liquid and leaving the mixture to sit for 30 minutes. The residue (if any) which form the bottom most layer, is drained from the mixture.

F. Ion exchange reaction

1mmol of the anion component is then added to the liquid and then left to stir at 250rpm at ambient conditions for 1 day to yield the ionic liquid.

G. Removal of by-products and solvents in vacuo using BUCHI Rotavapor

The liquid is put into the set-up with water bath temperature at 90°C for 15 minutes.

H. Drying the Ionic Liquids in vacuum oven

The liquids are weighed before they are placed in the oven and sealed. Vacuum is produced by selecting [Vac.], switching on the pump, waiting till P=76cmHg, switching off the pump and selecting [Close.]. Temperature is set at 25°C. The liquid is left in the oven for 15 minutes. Before removing the liquid, [Vent.] is selected to release vacuum. The liquids are weighed again after drying to confirm the loss of water.

3.2 CO₂ ABSORPTION

A. N₂ Purge

Firstly, a few drops of the liquids, i.e. pre-absorbed sample, is taken for FT-IR detection. Nitrogen gas (N_2) is then purged into the vial to remove all other gasses from the system. This is done for at least 15 minutes before proceeding with the CO₂ absorption.

B. CO₂ absorption

Absorption of CO_2 using the ionic liquids produced is performed at room temperature and pressure for 30 minutes each, using the apparatus assembly as shown in Figure 2. The liquids are weighed before and after absorption to calculate percentage of absorption. CO_2 absorption is also done for MEA, aqueous KOH and NaOH, and several commercial ionic liquids. A few drops of the post-absorbed liquids is taken for FT-IR detection.



Figure 3: Experimental Set-up for CO₂ Absorption

C. Detection with SHIMADZU FT-IR Spectroscope

FT-IR is used to detect the structure of the liquids and also the content of CO_2 in the liquids. The structures to detect are:



3.3 CHEMICALS USED

Below are the chemicals used in this experiment:

Chamical	Formula weight	Density (y/m))
Chemica	(g/mol)	
2-Bromoethylamine	204.89	-
3-Bromoethylamine	218.93	-
1-Butylimidazole	124.19	0.950
1-Methylimidazole	82.11	1.030
Sodium Benzenesulfonate	180.16	-
Sodium Methylsulfate	134.09	-
1-Butyl-3-methylimidazoliym hexafluorophosphate	284.18	1.38
1-Butyl-3-methylimidazolium methylsulfate	250.32	1.21
Guanidium trifluoromethylsulfonate	209.15	-

Table 2: Chemicals Used and their details

3.4 HEALTH, SAFETY & ENVIRONMENT

Both imidazoles used in this project are toxic in nature. Also the amines used are irritants and corrosive. The solvents used i.e. THF and ethanol, are both highly volatile and flammable. Thus, the whole experiment is conducted in a specific fume hood and all glassware used is only used for this particular project. This is to avoid contamination of the toxic materials to other users or experiments.

Also while conducting the experiment, personal protective equipment such as goggles, covered shoes and gloves are used to avoid reactants contact with eyes and skin.

CHAPTER 4: RESULTS & DISCUSSION

4.1 SYNTHESIS OF IONIC LIQUIDS

Through this project, a total of six ionic liquids have been synthesized using the procedures outlined in the previous chapter. Only IMI-05 and IMI-06 were produced without the ion exchange reaction. Basically ion exchange is carried out to substitute the bromide anion with another anion (methylsulfate and benzenesulfonate) which will allow for better CO_2 solubility in the liquid. The liquids and their characteristics are as follows:

Identifier	Name	Physical State	Colour	Vield (g)
IMI-01	1-ethylamine-3- butylimidazolium benzenesulfonate	Viscous Liquid	Brown	0.2353
IMI-02	1-ethylamine-3- methylimidazolium methylsulfate	Viscous Liquid	Yellow	0.9812
IMI-03	1-ethylamine-3- butylimidazolium methylsulfate	Viscous Liquid	Brown	1.3897
IMI-04	1-ethylamine-3- methylimidazolium benzenesulfonate	Very Viscous Liquid	Yellow	1.3106
IMI-05	1-propylamine-3- butylimidazolium bromide	Viscous Liquid	Yellow	1.2992
IMI-06	1-propylamine-3- methylimidazolium bromide	Viscous Liquid	Yellow	1.6476

Table 3: Final Products i.e. Ionic Liquids Produced

The following sub-sections will discuss the reactions involved in the production of the liquids and their final products.

4.1.1 IMI-01

Imidazole + Amine reaction



Ion exchange reaction



Final Product

1-ethylamine-3-butylimidazolium benzenesulfonate

4.1.2 IMI-02

Imidazole + Amine reaction



Ion exchange reaction



Final Product

1-ethylamine-3-methylimidazolium methylsulfate

4.1.3 IMI-03

Imidazole + Amine reaction



Ion exchange reaction



Final Product

1-ethylamine-3-butylimidazolium methylsulfate

4.1.4 IMI-04

Imidazole + Amine reaction



Ion exchange reaction



Final Product

1-ethylamine-3-methylimidazolium benzenesulfonate

4.1.5 IMI-05

Imidazole + Amine reaction



Final Product

1-propylamine-3-butylimidzolium bromide

4.1.5 IMI-06

Imidazole + Amine reaction



Final Product

1-propylamine-3-methylimidazolium bromide

4.2 FT-IR ANALYSIS PRE-ABSORPTION

The final products tested using FT-IR before going through CO_2 absorption to prove that the synthesis has been a success. To determine this, peaks at around 3250–3400 cm⁻¹ must be obtained to prove that amine is in the liquid. Through the analysis, it is observed that all of the liquids analyzed show peak formations indicating amine presence in the liquid. Repetitions were not able to be done due to limited amount of liquids. FT-IR results for Pre-Absorbed liquids are attached in the Appendix.

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4.3 CO₂ ABSORPTION

The ionic liquids produced were tested for CO_2 absorption using the apparatus as outlined in the previous chapter. Below are the results of the experiment.

 Table 4: CO2 Absorption Results using Ionic Liquids Produced

Absorbents	Initial Mass (g)	A: Moles of Product (moles)	Einal Mass (g)	B: Mass Gained (g)	C: B/A (g/mole)
$\begin{bmatrix} \mathbf{IMI-01} \\ \mathbf{M} \\ $	0.2353	0.00057906	0.2362	0.0009	1.55
$\begin{bmatrix} MI-02 \\ N \\ N \\ N \end{bmatrix} CH_3 SO_4$	0.9812	0.00308363	0.9823	0.0011	0.36
$\begin{bmatrix} & & & \\ & & & \\ & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & $	1.3897	0.00385732	1.3909	0.0012	0.31
$IMI-04$ $\left[\underbrace{N}_{N} \underbrace{N}_{N}^{*} \underbrace{NH_{2}}_{0} C_{0}H_{5}SO_{3}^{*} \right]$	1.3106	0.00359792	1.3086	-0.002	-
IMI-05 $\left[\underbrace{\bigvee}_{N,\bigvee}_{N} \underbrace{\bigvee}_{NH_2}\right] Br^-$	1.2992	0.00378643	1.2971	-0.0021	-
$\begin{bmatrix} \mathbf{MI-06} \\ \mathbf{N} \\ \mathbf{N} \\ \mathbf{N}^{+} \\ \mathbf{N}_{2} \end{bmatrix} \mathbf{Br}^{-}$	1.6476	0.00547303	1.651	0.0034	0.62

The results show that all the ionic liquids produced except for IMI-04 and IMI-05 experience mass gain after CO_2 has been passed through them. The decrease in mass for IMI-04 and IMI-05 is actually due to human error where over-excessive CO_2 inflow at start up causing some of the liquid to leak into the gas outlet through the syringe.

 CO_2 absorption has also been done using three commercial Ionic Liquids. The principles and reactions behind these absorption processes have been elaborated in section 2.4: Process Chemistry of Chapter 2: Literature Review & Theory. The followings are the results of the experiment.

Table 5: CO ₂	Absorption	Results	commercial	Ionic Liquids
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Absorbents	Initial Mass (g)	A: Moles of Product (moles)	Final Mass (c)	B: Mass Gained (2)	C: B/A (g/mole)
1-Butyl-3-methylimidazole hexafluorophosphate (C-01)	0.9998	0.00351819	1.006	0.0062	1.76
1-Butyl-3-methylimidazolium methylsulfate (C-02)	1.2357	0.00493648	1.2438	0.0081	1.64
Guanidium trifluoromethylsulfate (C-03)	0.9969	0.00476644	1.0055	0.0086	1.80

These results (positive mass gain) show that commercial ionic liquids are also capable of CO_2 absorption.

The value of **C** actually reflects the amount of CO_2 absorbed per mole of ionic liquid used. The values show that most of the ionic liquids analyzed are capable of absorbing a significant amount of CO_2 per mole of liquid used. IMI-01 has the highest value of 1.55 g CO_2 absorbed/moles of liquid among the ionic liquids synthesized while C-03 has the highest value of 1.80 g CO_2 absorbed/moles of liquid among all the ionic liquid tested.

4.4 FT-IR ANALYSIS POST-ABSORPTION

IMI-03 – IMI-06 and C-01 – C-02 have been characterized using FTIR. FTIR analysis shows that CO_2 is contained in the all the liquids characterized. This is seen with the peaks of in between 1820-1650 cm⁻¹. Thus this proves that the ionic liquids produced are capable of absorbing CO_2 . FT-IR results for Post-Absorbed are attached in the Appendix.

5.1 CONCLUSIONS

Amine solutions that are used in industries demonstrate a limited efficiency in terms of CO_2 absorption due to its high volatility and their dependence on water.

Thus, the main objective of this project is to develop a new ionic liquid for the absorption of CO_2 . The ionic liquid is to be task specific i.e. CO_2 absorbent and also have low melting point at room temperature. The suggested ionic liquid that was produced consists of a cation built of an imidazolium ion which is covalently bonded to an amine.

Through this project, 6 ionic liquids i.e. IMI-01 - IMI-06 have been produced. All of the ionic liquids produced have been tested for CO₂ absorption and all of them have been successful in absorbing an amount of CO₂. IMI-01 gave the best result if compared to the 5 other liquids synthesized with 1.55g CO₂ absorbed per mole of IMI-01. Three commercial ionic liquids have also been tested for CO₂ absorption and the results were all positive. Guanidium trifluoromethylsulfate gave the best result if compared with all of the ionic liquids with 1.80g CO₂ absorbed per mole of liquid. FT-IR characterization also shows positive results with NH₂ peaks found in Pre-Absorbed Ionic Liquid proving the reaction was a success while CO₂ peaks found in Post-Absorbed Ionic Liquid proving CO₂ has been absorbed by the liquids.

5.2 **RECOMMENDATIONS**

Due to the time constraint imposed on this project, no repetitions were able to be done. Thus it is recommended that less number of liquids be produced with more quantity and repetition. This will allow for more accurate analysis and comparison for efficiency. The use of a CO_2 micro-regulator is also recommended as this would also enhance the efficiency of the experiments conducted, avoid human errors and provide better control over the CO_2 flowrate during the absorption experiment.

And lastly, to further enhance the study of this project, it is recommended that the ionic liquids produced to be tested for CO_2 absorption at different temperature and/or pressure conditions.

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APPENDIX: PRE-ABSORPTION FT-IR CHARACTERIZATION RESULTS



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