# Refractive Indices of Aqueous Blends of Ammonium-Based Ionic Liquids and Potassium Carbonate

by

Muhammad Rusydi bin Jamil

Dissertation submitted to
the Chemical Engineering
Programme Universiti
Teknologi PETRONAS
in partial fulfilment of the requirement
for the BACHELOR OF
ENGINEERING (Hons) (CHEMICAL
ENGINEERING)

JAN 2015

Supervisor:
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Perak Darul Ridzuan.

# CERTIFICATION OF APPROVAL

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PERAK DARUL RIDZUAN

# 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.

(MUHAMMAD RUSYDI BIN JAMIL)

# ABSTRACT

Carbon dioxide ($CO_2$) capture via chemical absorption to capture excess $CO_2$ in a variety of gas streams has emerged as an important operation in industrial process. This project concerns the thermophysical properties study of aqueous blends of ammonium-based ionic liquids (AILs), a new class of unique solvents that has emerged as cheaper and greener alternatives due the possibility of tuning its properties by combining between cations, anions and/or possibly the introduction of functional groups. The specific AIL of particular interest in this study is tetrabutylammonium hydroxide (TBAOH), blended with the common solvent, potassium carbonate ($K_2CO_3$). Experimental data of refractive indices of aqueous blends of TBAOH and $K_2CO_3$ are measured in the temperature range between 303.15 and 333.15 K and at varying degrees of concentration. Based on the gathered results as well as comparative study with previous literature, the influence of varying temperature and concentrations on the refractive indices of these blends are discussed. Derived correlational properties such as polarizabilities, molar refractions and free volumes are also predicted by utilizing techniques from past studies.

# ACKNOWLEDGEMENT

I would like to take this opportunity to thank everyone whom had given their support and help throughout the whole period of completing this project.

First and foremost, I would like acknowledge the endless support received from my supervisor, Dr. Bhajan Lal throughout the whole period of completing this final year project. His guidance has really been the main source of motivation and has driven me in completing this project successfully, which has not been without adversity. My gratitude also goes to the staff at the Research Centre of $CO_2$ Capture ($RCCO_2C$), Mr. Adli and Mr. Shafiq as well as PhD student, Mr. Rezwan for their constant assistance in ensuring the success of this project. Also, not to forget, the dedicated staff and technologists at the Chemical Engineering Department for their cooperation.

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# CHAPTER 1: PROJECT BACKGROUND

## 1.1 Background of Study

The continuous emission of major greenhouse gases (GHGs) from various human activities have contributed to the ongoing global warming issue that has been causing significant environmental problems. The Intergovernmental Panel on Climate Change (IPCC) predicted that the average global temperature will rise by 1.1 to 6.4 °C by the end of the 21$^{st}$ century. Among the greenhouse gases released to the atmosphere, carbon dioxide ($CO_2$) has been identified as the largest component and responsible for up to 60% of the enhanced greenhouse effect. The primary sources of $CO_2$ emissions are power plants, industries, transportation, residential buildings, and energy sectors. Meanwhile in industries, the presence of $CO_2$ in natural gas reduces the heating value and can cause corrosion in process equipment due to its acidic nature. $CO_2$ also threatens to poison the catalysts in the ammonia synthesis process. Thus, $CO_2$ capture to retain excess $CO_2$ from a variety of gas streams has emerged as an increasingly important operation in industrial processes.

Whilst various techniques have been developed over the years including absorption, adsorption, membrane and cryogenic processes, the most commonly applied method is absorption by chemical solvents due to the many advancements in the selection of solvents that have been developed by numerous researchers. Initially, aqueous alkanolamines like monoethanolamine (MEA), diethanolamine (DEA), and methyldiethanolamine (MDEA) were considered as the most widely used and preferred solvents due to their supposed favourable attributes such as high $CO_2$ absorption capacity and high water solubility. Formation of solvents by blending of different alkanolamines is also favorable as small addition of secondary alkanolamines to primary alkanol amines increases mass transfer coefficient of $CO_2$ two to three times without any change in properties of blends. Although the absorption of $CO_2$ with the accelerated alkonolamines has been established as a proven technology, several drawbacks of their uses have been identified along the

way which include a relatively short life span due to amine oxidation degradation and the occurrence of corrosion in pipelines and equipment. These may result in significant consequences especially in terms of higher energy requirement and capital costs due to unplanned downtime, reduced equipment life, production losses and other management expenses. As such, the challenge is to combat these pivotal factors with the development of more economic and greener solvents.

Ionic liquids (ILs) as an alternative class of solvents appear as a promising choice to be used as absorbent for $CO_2$ separation because of its ability to be "tuned" for specific applications. Due to unique chemical and physical features such as negligible vapour pressure, non-flammability, thermal stability, high ionic conductivity, high solvation capability, hydrophobic and hydrophilic nature, excellent properties of recycling and contamination free functionality, a number of ILs from different groups including imidazolium, phosphonium, pyridinium and ammonium based have been synthesized and used for $CO_2$ separation. Despite these promising characteristics, there are some issues including high viscosity, high price and water instability at high temperatures as most of task specific imidazolium, phosphonium and pyridinium based ILs are used in pure form for $CO_2$ separation process. Among the ILs, ammonium based ILs appear as most promising class as they are water soluble, hydrolytically stable and nontoxic in nature. They can be used alone or with other solvents as a blend in aqueous solution for $CO_2$ absorption like conventional alkanolamines. Studies have shown that the hydroxyl ammonium based ILs have potential to be used as corrosion inhibitors for CS material in acidic media. Meanwhile, tetra-alkyl ammonium ILs have the ability to form the clathrate hydrate crystals below ambient temperature without applying high pressures, making them one of the key compounds in clarifying the structure of aqueous solutions. $Tf_2N^-$ anion-based ILs, on the other hand, when formed with quaternary ammonium cations could be optimized to achieve lower viscosity and high electrochemical stability.

Knowledge of thermophysical properties of ILs are essential for designing the acid gas removal systems. In the project, the refractive indices and surface tension of the ammonium-based ionic liquid TBAOH (aq), $K_2CO_3$ (aq) and their blended solutions over a wide range of temperatures (303.15-333.15) K and concentrations are to be studied and reported systematically. The study also seeks to analyze the influence of different temperatures and concentrations upon the refractive indices of TBAOH + $K_2CO_3$ blends.

## 1.2 Problem Statement

Ammonium-based ionic liquids (ILs) have increasingly been considered as green alternative solvents to replace their volatile traditional counterparts due to the unique features that they possess. Understanding the thermophysical properties of solvent systems, particularly ILs, is required to allow for the analysis and evaluation of mass transfer and $CO_2$ capture capacity for the rational design and optimization of acid gas treatment processes. However, the thermophysical data for the tetrabutylammonium hydroxide (TBAOH) and blends of TBAOH + $K_2CO_3$ is still unavailable, which creates a knowledge gap to use these types of IL and their blends for various purposes.

## 1.3 Objectives

The objectives of this project are:

1) To measure and present new data of the refractive indices of aqueous solution of TBAOH and $K_2CO_3$ and their blends.
2) To study the influence of temperature and concentration change upon the refractive indices of aqueous solution of TBAOH and $K_2CO_3$ and their blends.
3) To analyze derivative properties such as the polarizabilities and free volumes of TBAOH and $K_2CO_3$ and their blends based on the measured values.

## 1.4 Scope of Study

The project is an experiment-based study whereby the refractive indices of the aqueous solution of tetrabutylammonium hydroxide (TBAOH) and potassium carbonate ($K_2CO_3$) and their blends at varying temperatures and concentrations are to be determined. The measurements are to be recorded for temperatures ranging from (303.15 to 333.15) K and varying concentrations ($K_2CO_3$: 10 wt %, 20 wt %, and 30 wt %; TBAOH: 1 wt %, 2 wt %, 3 wt %, 4 wt %, and 5 wt %. The author is to compare the experimental data with related previous work and derive additional correlation properties based on the measurements obtained.

# CHAPTER 2: LITERATURE REVIEW

The capture of $CO_2$ from gas streams is an important operation in industrial processes. In industries, the presence of CO2 in natural gas reduces the heating value and can cause corrosion in process equipment due to its acidic nature. CO2 also threatens to poison the catalysts in the ammonia synthesis process (Mazinani et al., 2011). From the environmental aspect, the alarming rate of $CO_2$ emissions to the atmosphere has contributed greatly to the global warming issue, responsible for almost 60% of the enhanced greenhouse effect (Mondal et al., 2009). The Intergovernmental Panel on Climate Change (IPCC) predicted that the average global temperature will rise by 1.1 to 6.4 °C by the end of the 21st century due to the increasing emissions of GHGs. Thus, the reduction of $CO_2$ emissions from industries has become an urgent area of concern, with numerous technologies having been developed and applied over the years in an effort to continuously improve and find newer solutions.

## 2.1 Chemical Absorption and Industrial Absorbents for $CO_2$ Capture

There are several technologies readily available for removing $CO_2$ from industrial gas streams such as physical absorption, cryogenic separation, physical adsorption, membrane separation, and biological fixation. Among these techniques, chemical absorption by the use of solvents is considered as one of the reliable and effective methods for $CO_2$ capture (Mazinani et al., 2011). It deploys a system consisting of an absorber and a desorber. CO2 is separated from the flue gas by passing the flue gas through a continuous scrubbing system. The process utilizes the reversible chemical reaction of CO2 with an aqueous alkaline solvent (Kothandaraman, 2010). The most widely used absorbents being utilized for carbon removal from process gas streams are mixtures of aqueous alkanolamine solutions such as monoethanolamine (MEA), diethanolamine (DEA), and methyldiethanolamine (MDEA). These solvents have been established in the market and possess commercial importance due to its cheap price, high $CO_2$ absorption

capacity and high water solubility, among other attributes (Lin et al., 2014). In recent times, some disadvantages of these solutions have emerged to cancel out their more favourable attributes. These include low $CO_2$ loading, high energy consumption, high viscosity as well as operational factors such as corrosion and fouling of process equipment (Mazinani et al., 2011). Consequently, of increasing interest nowadays is the use of mixed amine solvents in gas treating processes in order to cater these problems. Some examples of mixed solvents are blends of primary and tertiary amines (MEA + MDEA) or blends of secondary and tertiary amines (DEA + MDEA). These blends are deemed advantageous as they combine the higher $CO_2$ loading of the tertiary amine with the higher reaction rate of the primary or secondary amine (Gomez et al, 2012). As such, the favourable features of different chemical absorbents could be merged while at the same time suppressing the more undesirable characteristics, thus producing excellent blends of absorbents.

## 2.2 Emergence of Ammonium-Based Ionic Liquids as 'Green Solvents'

Ionic liquids (ILs) are essentially a class of neoteric solvents composed of an organic cation and an organic/inorganic anion, with a melting point below 150°C, which do not easily form an ordered crystal and thus remain in a liquid state at or near room temperature (Ghatee et al, 2013). Dubbed as 'designer solvents' due to the possibility of "tuning" their physiochemical properties through a wise selection of cations and ions to cater for specific applications, an increasingly large amount of effort has been devoted to the study upon these ILs and they have emerged as an attractive alternative to the more volatile-natured conventional organic solvents in an effort to support more environmentally friendly processes (Verevkin et al., 2012). The unique properties possessed by the ILs - such as negligible vapor pressure, non-flammability, stability at high temperatures, high solvation capacity, and high ionic conductivity – make them suitable to be used as green solvents in a wide range of novel applications including gas separation (Bhattacharjee et al., 2014). Most reports on investigated ILs have been focused mainly on the imidazolium cation, such as the study conducted by Fredlake et al. (2004) on thermophysical properties of

imidazolium-based ILs including 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim][$BF_4$]) and 1-butyl-3-methylimidazolium hexafluorophosphate ([bmim][$PF_6$]). However, such experimental data of most other classes of ILs have remained scarce and largely unavailable, with only a minimal amount of study having been conducted as of yet (Li et al., 2013). Among the various types of ILs being developed, tetrabutylammonium hydroxide (TBAOH) is one particular IL that has attracted interest for potential use as a new solvent for $CO_2$ capture. TBAOH [linear formula: $(CH_3CH_2CH_2CH_2)_4N(OH)$] has a molecular weight of 259.47. It is a strong base and has excellent solubility in organic solvents. Extensive study shall be made on the physiochemical properties of this IL for further validation and development of its use and whether it could become a mainstay as a new class of greener solvent in the area of $CO_2$ removal.

***Figure 2.1:*** *Organic structure of tetrabutylammonium hydroxide (TBAOH)*

## 2.3 Physicochemical Properties of Potassium Carbonate

Pure potassium carbonate is a solid at room temperature. Potassium carbonate dissociates completely in water into potassium (K+) and carbonate ions ($CO_3^{2-}$). The dissolution in water is exothermic, so vigorous reaction can occur when potassium carbonate is added to water. The vapour pressure of the substance is very low and a melting point cannot be determined, as the substance decomposes at high temperature. The physicochemical properties of the compound are mentioned in the Table 3.

***Table 2.1:*** *Physicochemical Properties of Potassium Carbonate*

| | |
|---|---|
| **Physical State** | Solid (Powder) |
| **Colour** | White |
| **Density** | 2.43 g/cm$^3$(19˚C) |
| **Melting Temperature** | 891˚C |
| **Boiling Temperature** | Very high temperatures |
| **Molecular Weight** | 138.2 g/mol |
| **Water Solubility** | Very Soluble |

## 2.4 Refractive index (RI) Analysis

Refractometry is a technique that measures how light is refracted when it passes through a given substance, in this case, an unknown compound. The amount by which the light is refracted determines the refractive index (RI). Refractive index can be used to identify an unknown liquid compound, or it can be used as a means of measuring the purity of a liquid compound by comparing it to literature values (Chem 211: Refractrometry). The closer the refractive index is to the literature values, the purer the sample. Refractive index is very important since it is related to such fundamental thermodynamic properties as solvent density, phase composition, solute concentration, and interfacial tension. Refractive index is defined as the ratio of the velocity of light in air to the velocity of light in the medium being measured: $\eta_D$ = [V air]/ (V liquid). The refractive index of a substance is strongly influenced by temperature and the wavelength of light used to measure it, therefore, care must be taken to control or compensate for temperature differences and wavelength.

## 2.5 Previous Studies on the Thermophysical Properties of Ammonium-Based Ionic Liquids

In recent times, researches have conducted studies on the thermophysical properties of ammonium-based ionic liquids (ILs). Bhattacharjee et al conducted a study on the thermophysical properties of similar ammonium-based ILs with the common bis(trifluoromethylsulfonyl)imide anion (Bhattacharjee et al., 2014). The study covered measurement of refractive indices of the ammonium-based ILs, performed at 589.3 nm using an automated Abbemat 500 Anton Paar refractometer in the temperature range from T = (283.15-353.15) K and at atmospheric pressure. The result showed a decrease in refractive indices with increasing temperature, fitted with the group contribution method proposed by Gardas and Coutinho. The figure below illustrates the measured refractive indices, with the solid lines representing the Gardas and Coutinho group contribution method:

***Figure 2.2:*** *Refractive index data for the studied quarternary ammonium ILs (Bhattacharjee, 2014)*

Another study was also done which included refractive index measurement for ammonium-based ILs with $MF_m$ anions (Li et al., 2013). The measurement was done using an Abbemat 2W Anton Paar refractometer at T = 20°C, P = 0.1 Mpa for different mass fractions, where [w = mass of ionic liquid/(mass of ionic liquid + mass of acid)]. The results showed that the refractive index increases pseudo-linearly with increasing mass fraction. It was also observed that the elongation of alkyl chain has positive effect on refractive index of binary mixtures. From the data, the derived molar refractions, $R_m$, free volumes, $f_m$, and polarizabilities were determined using the following equations:

# CHAPTER 3: METHODOLOGY

## 3.1 Research Methodology

**Problem Definition**

**Definition of Objectives of Study**

**Literature Review**

**Experiment Methodology** **No**

**Is properties required obtained?**

**Yes**

**Results Obtained**

**Data analysis and validation (AAD)**

***Figure 3.1:*** *Research Methodology*

## 3.2 Specification of Chemicals

Tetrabutylammonium (TBAOH) and potassium carbonate ($K_2CO_3$) with a purity ≥ 99.00% and 99% respectively are to be purchased from Sigma Aldrich/Merck. The specifications provided by the supplier for TBAOH contain halide (bromide) ≤ 0.01 % and sulphate ≤ 500 mg/kg as impurities. All the chemicals are used without any further purification. The aqueous solutions of TBAOH and $K_2CO_3$ and their aqueous blends of TBAOH + $K_2CO_3$ are prepared in Ultrapure water (model Lab Tower EDI 15). The concentrations of the prepared solutions are measured by a weighing balance with an accuracy of ± 0.003g. All prepared aqueous solutions are kept in airtight bottles before use.

## 3.3 Equipment for Experimental Work

***Table 3.1:*** *Equipment for Experimental Work*

| No | Parameter | Equipment & Principle | Location |
|---|---|---|---|
| 1. | Refractive Index<br><br>Figure 3.5: Atago RX-5000 | - Name: Atago RX-5000 Alpha Digital Refractometer.<br>- Temperature range: 20-80 $^oC$.<br>- An automatic digital refractometer which can set measurement temperature internally, it has a thermo-module to control temperature. Thus, a constant temperature water bath is not necessary.<br>- High refractive index ± 0.00004 accuracy can be obtained. | Block 4, Level 2 |

## 3.4 Gantt Chart of FYP II

***Table 3.2:*** *FYP II Gantt Chart*

| No | Detail/Week | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Project Work Continues | | | | | | | | | | | | | | |
| 2 | Submission of Progress Report | | | | | | | | | | | | | | |
| 3 | Pre-SEDEX | | | | | | | | | | | | | | |
| 4 | Submission of Draft Final Report | | | | | | | | | | | | | | |
| 5 | Submission of Dissertation | | | | | | | | | | | | | | |
| 6 | Submission of Technical Paper | | | | | | | | | | | | | | |
| 7 | Viva | | | | | | | | | | | | | | |
| 8 | Submission of Project Dissertation | | | | | | | | | | | | | | |

# CHAPTER 4: RESULTS AND DISCUSSION

## 4.1 Sample Preparation

Initially, sample preparation was conducted beforehand. The aqueous blends of tetrabutylammonium hydroxide (TBAOH) and potassium carbonate ($K_2CO_3$) solutions were prepared gravimetrically using an analytical balance. The table below shows the sample calculations to arrive at the intended concentration (in mass fractions) for the aqueous blends.

***Table 4.1:*** *Sample calculations of $K_2CO_3$ and TBAOH*

| Sample | 10% $K_2CO_3$ Sample | | | | | |
|---|---|---|---|---|---|---|
| | $K_2CO_3$ | | TBAOH + $H_2O$ | | $H_2O$ | |
| | Actual | Calculated | Actual | Calculated | Actual | Calculated |
| 1 | 3.2498 | 3.2490 | 0.9900 | 1.0000 | 28.2640 | 28.2410 |
| 2 | 3.2490 | 3.2490 | 1.9927 | 2.0000 | 27.2674 | 27.2410 |
| 3 | 3.2490 | 3.2490 | 2.9982 | 3.0000 | 26.2440 | 26.2410 |
| 4 | 3.2498 | 3.2490 | 4.0059 | 4.0000 | 25.2430 | 25.2410 |
| 5 | 3.2496 | 3.2490 | 5.0041 | 5.0000 | 24.2530 | 24.2410 |
| Sample | 20% $K_2CO_3$ Sample | | | | | |
| | $K_2CO_3$ | | TBAOH + $H_2O$ | | $H_2O$ | |
| | Actual | Calculated | Actual | Calculated | Actual | Calculated |
| 1 | 6.4980 | 6.4980 | 1.0040 | 1.0000 | 25.0168 | 24.9920 |
| 2 | 6.4975 | 6.4980 | 2.0016 | 2.0000 | 23.9904 | 23.9920 |
| 3 | 6.4981 | 6.4980 | 3.0010 | 3.0000 | 23.0479 | 22.9920 |
| 4 | 6.4990 | 6.4980 | 4.0083 | 4.0000 | 22.0255 | 21.9920 |
| 5 | 6.4980 | 6.4980 | 4.9980 | 5.0000 | 21.0710 | 20.9920 |
| Sample | 30% $K_2CO_3$ Sample | | | | | |
| | $K_2CO_3$ | | TBAOH + $H_2O$ | | $H_2O$ | |
| | Actual | Calculated | Actual | Calculated | Actual | Calculated |
| 1 | 9.7495 | 9.7494 | 1.0040 | 1.0000 | 21.7445 | 21.7406 |
| 2 | 9.7498 | 9.7494 | 2.0100 | 2.0000 | 20.7406 | 20.7406 |
| 3 | 9.7495 | 9.7494 | 3.0085 | 3.0000 | 19.7432 | 19.7406 |
| 4 | 9.7498 | 9.7494 | 4.0011 | 4.0000 | 18.7459 | 18.7406 |
| 5 | 9.4910 | 9.7494 | 5.0094 | 5.0000 | 17.7444 | 17.7406 |

## 4.2 Refractive Index

The refractive indices, $n_d$, of aqueous blends of potassium carbonate and tetrabutylammonium hydroxide ($K_2CO_3$ + TBAOH) were experimentally measured over a wide range of temperatures and concentrations. For refractive index data, it also been compared to the literature review data in ensuring the accuracy of equipment used and the validity of data obtained.

### 4.2.1 Refractive Index Measurement

Experimental Procedure:

The refractive indices of varying concentrations of **$K_2CO_3$ (10%, 20%, and 30%)** and **TBAOH (1%, 2%, 3%, 4%, and 5%)** were determined using a **programmable digital refractometer (RX-5000 alpha, Atago)**. All the measurements were performed at **temperatures (303.15 to 333.15) K** with a temperature control accuracy of ± 0.05°C.

Results:

The results obtained are as follows:

***Table 4.2:*** *Data of refractive indices for 10% $K_2CO_3$ and 1-5% TBAOH*

| Temperature, K | 10% $K_2CO_3$ + 1% TBAOH | 10% $K_2CO_3$ + 2% TBAOH | 10% $K_2CO_3$ + 3% TBAOH | 10% $K_2CO_3$ + 4% TBAOH | 10% $K_2CO_3$ + 5% TBAOH |
|---|---|---|---|---|---|
| 303.15 | 1.3482 | 1.3531 | 1.3589 | 1.3632 | 1.3668 |
| 308.15 | 1.3457 | 1.3513 | 1.3561 | 1.3612 | 1.3659 |
| 313.15 | 1.3432 | 1.3482 | 1.3536 | 1.3589 | 1.3639 |
| 318.15 | 1.3416 | 1.3450 | 1.3515 | 1.3565 | 1.3616 |
| 323.15 | 1.3397 | 1.3437 | 1.3496 | 1.3544 | 1.3603 |
| 328.15 | 1.3380 | 1.3410 | 1.3472 | 1.3526 | 1.3591 |
| 333.15 | 1.3356 | 1.3397 | 1.3459 | 1.3512 | 1.3576 |

***Table 4.3:*** *Data of refractive indices for 20% $K_2CO_3$ and 1-5% TBAOH*

| Temperature, K | 20% $K_2CO_3$ + 1% TBAOH | 20% $K_2CO_3$ + 2% TBAOH | 20% $K_2CO_3$ + 3% TBAOH | 20% $K_2CO_3$ + 4% TBAOH | 20% $K_2CO_3$ + 5% TBAOH |
|---|---|---|---|---|---|
| 303.15 | 1.3690 | 1.3721 | 1.3757 | 1.3804 | 1.3841 |
| 308.15 | 1.3674 | 1.3712 | 1.3749 | 1.3787 | 1.3831 |
| 313.15 | 1.3667 | 1.3699 | 1.3737 | 1.3776 | 1.3815 |
| 318.15 | 1.3653 | 1.3684 | 1.3725 | 1.3767 | 1.3806 |
| 323.15 | 1.3642 | 1.3679 | 1.3713 | 1.3754 | 1.3795 |
| 328.15 | 1.3636 | 1.3668 | 1.3702 | 1.3740 | 1.3783 |
| 333.15 | 1.3628 | 1.3653 | 1.3691 | 1.3729 | 1.3771 |

***Table 4.4:*** *Data of refractive indices for 30% $K_2CO_3$ and 1-5% TBAOH*

| Temperature, K | 30% $K_2CO_3$ + 1% TBAOH | 30% $K_2CO_3$ + 2% TBAOH | 30% $K_2CO_3$ + 3% TBAOH | 30% $K_2CO_3$ + 4% TBAOH | 30% $K_2CO_3$ + 5% TBAOH |
|---|---|---|---|---|---|
| 303.15 | 1.3910 | 1.3933 | 1.3959 | 1.3979 | 1.4007 |
| 308.15 | 1.3900 | 1.3928 | 1.3952 | 1.3971 | 1.3999 |
| 313.15 | 1.3893 | 1.3925 | 1.3947 | 1.3963 | 1.3991 |
| 318.15 | 1.3881 | 1.3915 | 1.3933 | 1.3954 | 1.3983 |
| 323.15 | 1.3880 | 1.3901 | 1.3921 | 1.3948 | 1.3974 |
| 328.15 | 1.3866 | 1.3896 | 1.3916 | 1.3943 | 1.3963 |
| 333.15 | 1.3855 | 1.3883 | 1.3911 | 1.3930 | 1.3952 |

## 4.2 Analysis & Discussion

The refractive indices of aqueous solutions of $K_2CO_3$ and TBAOH are plotted versus temperature in Figures 4.1, 4.2, and 4.3 respectively.

***Figure 4.1**: Graph of refractive indices for 10% $K_2CO_3$ and 1-5% TBAOH*

***Figure 4.2**: Graph of refractive indices for 20% $K_2CO_3$ and 1-5% TBAOH*

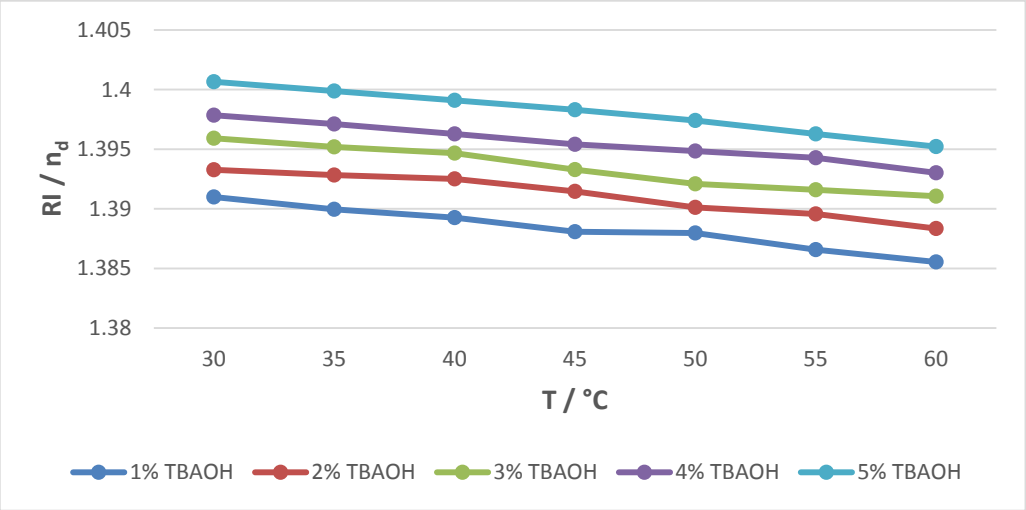


***Figure 4.3**: Graph of refractive indices for 30% $K_2CO_3$ and 1-5% TBAOH*

Based on the graphs in Figures 4.1, 4.2, and 4.3, it can be seen that that with an increase in temperature, the refractive indices values decrease. However, an increase in refractive indices values was observed with an increase of $K_2CO_3$ and/or TBAOH concentration. This observation agrees with the general trend with refractive indices measurement of other ionic liquids, specifically ammonium-based ionic liquids (AILs) as can be witnessed in previous studies.

To verify the accuracy of the experimentation work conducted, the experimental data obtained for the aqueous blends should be compared with existing literature data for similar types of solutions. However, a drawback encountered by the author is the scarcity of previous data for aqueous blends of ($K_2CO_3$ +TBAOH) solution being studied in this project. Fortunately, there exists several studies conducted on other related ammonium-based ionic liquids. As mentioned in the literature review, Bhattacharjee et al. (2014) [61] had conducted a thermophysical properties study on ammonium-based ionic liquids including butyltrimethylammonium bis(trifluoromethylsulfonyl)imide [$N_{4111}$][$NTf_2$] and tributylmethylammonium bis(trifluoromethylsulfonyl)imide [$N_{4441}$][$NTf_2$]. This shall be used as a basis for comparison to indicate the accuracy of the experimental work done by the author in this project, specifically the data for refractive indices.

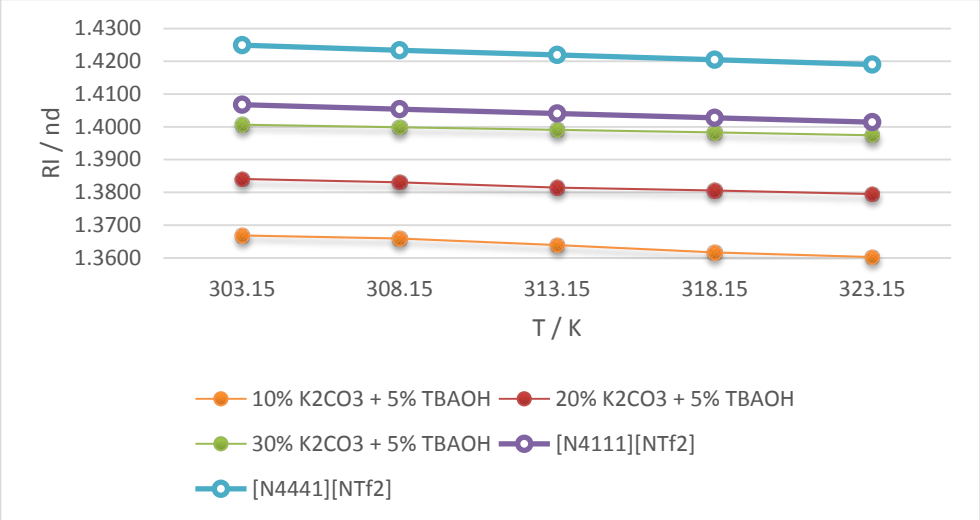


***Figure 4.4**: Comparison between refractive indices data of the current work and those available in literature (Bhattacharjee et al.)*[61]
The filled circles represented the experimental data while the unfilled circles represent data reported in previous works.

Meanwhile, Figure 4.4 illustrates the comparison of the data obtained in the current experimental work with other related ammonium-based ionic liquids available in literature. From the results comparison, we could calculate the average absolute deviation (AAD) between the data involved. The AAD was obtained by using the following equation:

Although it was not possible to have a direct comparison for TBAOH blends due to the scarcity of data, the relatively small AAD of 0.0171% indicates the level of accuracy of the measurement work conducted by the author through the project as it is in good agreement with prior studies conducted by Bhattacharjee et al.[61]

From the experimental data of refractive indices, the author also attempted to derive other correlational properties such as molar refractions, free volumes and polarizabilities by using these equations:

where  is the electronic polarizability,  the vacuum permittivity,  the compound's density, M the molecular weight, and $N_A$ the Avogadro number. The table below summarizes the results from the calculations:

***Table 4.5:*** *Refractive indices, polarizabilities, derived molar refractions, and free volumes for 10-30% $K_2CO_3$ and 5% TBAOH at 303.15 K.*

| $W_{K2CO3}$ | $n_d$ | Polarizability ($bohr^3$) | $R_m$ ($cm^3\ mol^{-1}$) | $f_m$ ($cm^3\ mol^{-1}$) |
|---|---|---|---|---|
| **10% $K_2CO_3$** | 1.3668 | 195.49 | 44.32 | 126.25 |
| **20% $K_2CO_3$** | 1.3841 | 182.54 | 41.39 | 119.75 |
| **30% $K_2CO_3$** | 1.4007 | 173.44 | 39.32 | 115.50 |

It has been suggested that refractive indices are an indication of the dielectric response to an electric field, induced by electromagnetic waves and it could be deliberated as the first order approximation response to electronic polarization within an instantaneous time scale.[61] From the data, the polarizabilities decrease with an increase in refractive index values. This is also possibly influenced by the different densities at varying concentrations of the aqueous blends. Likewise for the derived molar refractions and free volumes, the values show a decrease with increasing refractive index.

# CHAPTER 5: CONCLUSION AND RECOMMENDATION

## 5.1 Conclusion

The objectives set for FYP II has been achieved in the given time frame: The results obtained show a decrease in the refractive indices with an increase in temperature. Conversely, there was an increase of refractive indices with an increase in concentration in for each solution tested.

The comparison between the experimental data obtained and the literature data need to be done in order to measure the accuracy and the validity of the equipment and method used in the project work. The smaller the AAD value calculated, the accurate the data measured from experimental work. In this work, the AAD was found to be 0.0171% which indicated a good degree of accuracy.

Several correlation properties were derived from the measured experimental data of refractive indices such as electronic polarizabilities, derived molar refractions and free volumes. The properties show an overall decrease with an increase in refractive indices.

## 5.2 Recommendations

The recommendations to improve this research project are as follows:

1. To increase the degree of accuracy in the experimentation work by improving the procedural work and taking extra measurements.

2. To analyze methods for predicting the expected results of data that are unavailable in previous literature.

3. To conduct further analysis on correlation properties and its impacts on the effectiveness of the solvent.

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