Density Measurements of Aqueous Solution of Potassium Carbonate and ammonium based ionic liquids and their blends

By

Lukman Hakim bin Noor Azmi

15002

Dissertation submitted in partial fulfillment of

the requirement for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

January 2015

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Approved by,

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(Dr. Bhajan Lal)

FYP Supervisor

UNIVERSITI TEKNOLOGI PETRONAS

BANDAR SERI ISKANDAR, PERAK

CERTIFICATION OF ORIGINALITY

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

_____

Lukman Hakim bin Noor Azmi

Acknowledgement

I would like to thank my supervisor, Dr Bhajan Lal for guiding me through this project from day one up until the day I submit this dissertation. Not to forget Rizwan, a master student under the supervision of Dr. Bhajan Lal that help guide me through the experimental procedures. I could not have done this without the help of the lecturers in Chemical Engineering department of UTP that provide insight whenever problems occurred throughout this project.

Abstract

The application of ammonium based ionic liquids (ILs) in carbon dioxide absorption has never been thoroughly researched and the data available is limited. Ammonium based ILs have unique characteristic which makes it a good substance as a CO2 capture agent because of its negligible vapor pressure, non-flammability and thermal stability. In depth research must be conducted to understand the thermophysical properties of ILs. In this project, the student will focus more on how density changes with respect to the changes in temperature and concentration of ILs and Potassium carbonate as well as their blends. The concentration will be altered slightly with each reading and the temperature will be incremented accordingly. The data recorded from this experiment will be analyzed and further justifications will be put forward to clarify the data collected.

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CHAPTER 1 INTRODUCTION

1.1 Background

Over the last 200 years unwanted changes occurred in climate because of increase in concentration of greenhouse gases causing global warming of the earth. According to Spasojevic(2013), the disturbed heat balance of the earth became a serious environmental concern causing negative effects on the human life, agriculture and global economy. CO_2 gas is regarded as major greenhouse gas and its emission to atmosphere increases global warming. The major sources of CO_2 emissions are chemical and fertilizer industries, coal power plants and energy sectors. CO_2 capture and storage is particularly promising to reduce the emission of CO_2 to atmosphere to maintain the heat balance of the earth, on the other hand stored CO_2 can be used for manufacturing of urea and as fire extinguishers due to its incombustible nature (Shaikh, M. S.; Shariff, A. M.; Bustam, M. A.; Murshid, G..2013). In this regard different techniques have been developed including absorption, adsorption, membrane and cryogenic processes. Among these techniques most mature scientific and industrially adapted technique is absorption by liquid solvent. Due to Researcher's efforts many advancements and alterations became possible in selection of a solvent to enhance CO_2 absorption. There is a need to find an effective way to absorb carbon dioxide using a cheaper and greener process. The most widely used solvents are aqueous solution of amines like monoethanolamine (MEA), diethanolamine (DEA) and Methyldiethanolamine (MDEA). Some researchers found that sterically hindered amines have potential to be used for acid gas removal because of their high carbon dioxide loading capacity and easy regeneration of solvent (Samanta & Bandyopadhyay, 2007). Therefore it should be noted that under certain conditions these sterically hindered amines have low CO_2 loading capacity than conventional alkanol amines. Formation of solvents by blending of different alkanolamines

is also a favorable approach 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. Samantha & Bandyopadhyay added that the use of promoters and activators has also shown a potential by enhancing the rate of reaction. Potassium carbonate (K_2CO_3) has emerged as a good promoter than any other conventional accelerator for CO_2 absorption when used with MDEA and MEA due to rapid formation of carbonate and theoretically absorb more CO_2 moles than any other amines. After many years of using these solvents for CO_2 absorption many drawbacks have been found. The identified drawbacks include corrosion of pipe lines and equipments causing shutdowns of system, minimizing production, additional cost to restore the system, significant amount of energy requirement for regeneration of solvent, foaming and degradation of solvent. In the light of these drawbacks, ionic liquids (ILs) as an alternative class of solvents appear as a promising choice to be used as absorbent for CO_2 separation. Due to unique chemical and physical features such as negligible vapor pressure, non-flammability, thermal stability, hydrophobic and hydrophilic nature, excellent properties of recycling and contamination free functionality, a number of ionic liquids from different groups including imidazolium, phosphonium, Pyridinium and ammonium based have been synthesized and used for CO_2 separation. With all these promising characteristics of ionic liquids there are some issues of high viscosity, high price and water instability at high temperatures as most of task specific Imidazolium, phosphonium and Pyridinium based ionic liquids are used in pure form for CO_2 separation process. Among the ionic liquids, ammonium based ionic liquids 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 ionic liquids have potential to be used as corrosion inhibitors for CS material in acidic media. Knowledge of physical properties like density, viscosity and

refractive index are essential for design the acid gas removal systems. The thermophysical data for the tetrabutyl ammonium hydroxide and blends of TBAOH + Potassium carbonate is unavailable, which makes a knowledge gap to use this types of IL and their blends for different applications. In the present work physicochemical properties including density of TBAOH (aq), K_2CO_3 (aq) and their blended solutions over a wide range of temperatures (303.15-333.15) K and concentrations have been studied and reported systematically. The current proposal includes the analysis of the influence of temperatures and concentrations upon density.

1.2 Problem Statement

In today's era, the world has undergone an industrial revolution and advancement of technologies. This resulted in an increase in greenhouse gases being release to the atmosphere, more commonly known gases are carbon dioxide. There have been several critical questions arise on how are we going to cope with the increase in number of carbon dioxide content in our atmosphere.

Up till now, researchers have published work in the thermodynamics of polyamines or salt amines mixtures. One of the most interesting part of the studies have been the relation between ammonium based ionic liquids and potassium carbonate with carbon dioxide. The ammonium based ionic liquid that we are interested in for this project will be Tetra-butylammonium hydroxide, TBAOH. An effective thermodynamic representation of OH^-/K_2CO_3 will improve the fundamental understanding of other amine solutions and mixtures.

1.3 Objective

The objectives of this paper are:

- To determine the effect of temperature on density of TBAOH and K_2CO_3
- To determine the effect of concentration on density of TBAOH and K_2CO_3
- Provide these data to enable further study and design of CO2 capture models.

1.4 Scope of Study

The scope of this paper will cover the physical properties of individual solvents (K_2CO_3, TBAOH and H_2O) and their blends. Laboratory experiments will be conducted to measure the density of aqueous solutions of TBAOH (1-5wt%) and K_2CO_3 (10-30wt%) and their blends at a range temperature of T=(303.15 to 333.15) K.

CHAPTER 2 LITERATURE REVIEW

2.1 Physical / Chemical Properties of TBAOH

TBAOH, also known as Tetra-butylammonium hydroxide is the chemical compound with the formula $(C_4H_9)_4$NOH or Bu_4NOH. This substance is not readily obtained as a pure component, but is available as solution in water.

Table 1: Physical/ Chemical properties of TBAOH

Physical state	Solid at 25℃
Density	$0.995 g/cm^3$
Melting Temperature	$27 - 30$℃
Boiling Temperature	100℃
Molecular Weight	259.47 g/mol

2.2 Physical / Chemical Properties of Potassium carbonate

Potassium carbonate is a white salt, which is soluble in water. It forms a strong alkaline solution. The appearance of potassium carbonate is usually damp, and it is mainly used in the production of soap and glass.

Table 2: Physical/ Chemical properties of Potassium Carbonate

Physical state	Solid at room temperature
Density	2.43 g/cm^3
Melting Temperature	891℃
Boiling Temperature	$decomposes$
Molecular Weight	138.205 g/mol

2.3 How density is influenced by temperature and concentration

Blend amine solvents, which consist of a mixture of a primary or secondary amine with a tertiary amine to combine with a higher equilibrium capacity of the tertiary amine with the higher reaction rate of the primary or secondary amines are suggested to be used for industrial treating process. (Samanta & Bandyopadhyay, 2007) The application of amines will deliver a huge improvement in the absorption capacity as well as the absorption rate and in the end will result in great savings in solvent regeneration energy requirement. Samanta and Bandyopadhyay work concluded that density reduces as the temperature increases. As for the effect of concentration on density, as the concentration of mixtures increases in the solvents, the density increases as well, but as the temperature goes up, the density will reduce. (Lu, J.G., & et. Al, 2012) The work that Lu has done is by using Potassium citrate and Piperazine, and the results show that the density of PZ+PC decreases with an increase in mole fraction of PZ. This is because the molecular weight of PZ is smaller as compared to PC. In my experiment later on, instead of using PC, TBAOH will be used. TBAOH molecular weight is still much higher than PZ's thus we can expect a similar trend. Liu, Wang, Hartono, Svendsen and Chen have conducted an experiment using NH3 and PZ blends. From their results, the densities of the binary and tertiary mixtures decrease as the mole fraction of NH3 increases, mole fraction of PZ decreases and temperature increases. Comparing Lu J.G et.al work with Liu's, there is a slight difference where in Lu J.G. et.al, the density decreases as the mole fraction of PZ increase. In Liu's case, the density decreases as the mole fraction of

PZ decreases. This is because in Liu's work, the other substances that are used in the blend have a lower molecular weight as compared to PZ whereas in Lu J.G. et.al's work, PZ has a smaller molecular weight than the other substances.

CHAPTER 3 METHODOLOGY

3.1 Research Methodology

Stock Solution preparation

TBAOH and Potassium carbonate with a purity ≥ 99.00 % are purchased from Benua Sains and Merck respectively. 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 (1,2,3,4 and 5) wt. % and Potassium carbonate at (10, 20 and 30) wt. % are prepared in the lab. 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.

Density Measurements

The densities of binary and ternary aqueous solutions are measured at different temperatures ranging from 298.15-333.15 K using DMA 5000 (with automatic viscosity correction) with measuring accuracy ± 0.0001 g/cm^3. Density measuring cell is designed to measure the density of liquids and gases at high pressures and temperatures. It can operate at temperature ranges from 263.15 - 473.15 K and pressure up to 1000 kPa. The cell temperature is regulated with built-in solid state thermostat, maintaining a temperature within accuracy of ± 0.01 K. The reported densities are the average of three measurements. For better accuracy the equipment is calibrated with standard water of Millipore quality after each measurement.

3.2 List of Equipment

Density Meter DMA 5000

Description – Since the density will be measured with respect to the change in temperatures and concentrations, density meter DMA 5000 will be used to measure the density of TBAOH, K_2CO_3 and their blends.

This experiment will be conducted in Block N.

3.3 Sample Preparation

Preparation of solutions will be done as per table below:

K_2CO_3 (10 wt.%)

Table 3: 10wt% K_2CO_3

	K_2CO_3 (g)		TBAOH + H_20 (g)		H_20 (g)	
SAMPLE	ACTUAL	CALCULATED	ACTUAL	CALCULATED	ACTUAL	CALCULATED
1	3.2498	3.249	0.99	1	28.264	28.241
2	3.249	3.249	1.9927	2	27.2674	27.241
3	3.249	3.249	2.9982	3	26.244	26.241
4	3.2498	3.249	4.0059	4	25.243	25.241
5	3.2496	3.249	5.0041	5	24.253	24.241

K_2CO_3 (20 wt.%)

Table 4: 20wt% K_2CO_3

	K_2CO_3 (g)		TBAOH + H_20(g)		H_20 (g)	
SAMPLE	ACTUAL	CALCULATED	ACTUAL	CALCULATED	ACTUAL	CALCULATED
1	6.498	6.498	1.004	1	25.0168	24.992
2	6.4975	6.498	2.0016	2	23.9904	23.992
3	6.4981	6.498	3.001	3	23.0479	22.992
4	6.499	6.498	4.0083	4	22.0255	21.992
5	6.498	6.498	4.998	5	21.071	20.992

K_2CO_3 (30 wt.%)

Table 5: 30wt% K_2CO_3

	K_2CO_3 (g)		TBAOH + H_20 (g)		H_20(g)	
SAMPLE	ACTUAL	CALCULATED	ACTUAL	CALCULATED	ACTUAL	CALCULATED
1	9.7495	9.7494	1.004	1	21.7445	21.7406
2	9.7498	9.7494	2.01	2	20.7406	20.7406
3	9.7495	9.7494	3.0085	3	19.7432	19.7406
4	9.7498	9.7494	4.0011	4	18.7459	18.7406
5	9.491	9.7494	5.0094	5	17.7444	17.7406

3.4 Gantt Chart and Key Milestones

Table 6: Gantt Chart for FYP 1

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Preliminary Research Work														
3	Submission of Extended Proposal						●								
4	Proposal Defence														
5	Project work continues														
6	Submission of Interim Draft Report													●	
7	Submission of Interim Report														●

Table 7: Gantt Chart for FYP 2

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues	■	■	■	■	■	■	■								
2	Submission of Progress Report							●								
3	Project Work Continues								■	■	■	■	■			
4	Pre-SEDEX										●					
5	Submission of Draft Final Report											●				
6	Submission of Dissertation (soft bound)												●			
7	Submission of Technical Paper												●			
8	Viva													●		
9	Submission of Project Dissertation (Hard Bound)															●

CHAPTER 4 RESULTS AND DISCUSSION

Table 8: Density for 10wt% K_2CO_3 + TBAOH at different concentrations and temperatures

Sample	1	2	3	4	5
K_2CO_3	10%				
TBAOH	1%	2%	3%	4%	5%
Temperature (^oC)	Reading				
30	1.0827	1.0905	1.113	1.1285	1.1356
35	1.0817	1.0889	1.1115	1.1265	1.1346
40	1.0805	1.0878	1.1102	1.1246	1.1335
45	1.0792	1.0857	1.108	1.1232	1.1323
50	1.0781	1.0842	1.106	1.122	1.131
55	1.0769	1.0843	1.1043	1.1208	1.111
60	1.0754	1.0842	1.102	1.1195	1.1075

Table 9: Density for 20wt% K_2CO_3 + TBAOH at different concentrations and temperatures

Sample	1	2	3	4	5
K_2CO_3	20%				
TBAOH	1%	2%	3%	4%	5%
Temperature (^oC)	Reading				
30	1.2476	1.2565	1.2610	1.2675	1.2775
35	1.2453	1.2545	1.2595	1.2666	1.2755
40	1.2442	1.2524	1.257	1.2657	1.2732
45	1.237	1.2506	1.2558	1.2645	1.2722
50	1.234	1.249	1.2539	1.2634	1.2712
55	1.2322	1.2477	1.2526	1.2622	1.2702
60	1.2302	1.2453	1.2508	1.261	1.269

Table 10: Density for 30wt% K_2CO_3 + TBAOH at different concentrations and temperatures

Sample	1	2	3	4	5
K_2CO_3	30%				
TBAOH	1%	2%	3%	4%	5%
Temperature (^oC)	Reading				
30	1.3505	1.3505	1.3505	1.3505	1.3505
35	1.3480	1.3480	1.3480	1.3480	1.3480
40	1.3455	1.3455	1.3455	1.3455	1.3455
45	1.3425	1.3425	1.3425	1.3425	1.3425
50	1.3401	1.3401	1.3401	1.3401	1.3401
55	1.338	1.338	1.338	1.338	1.338
60	1.3367	1.3367	1.3367	1.3367	1.3367

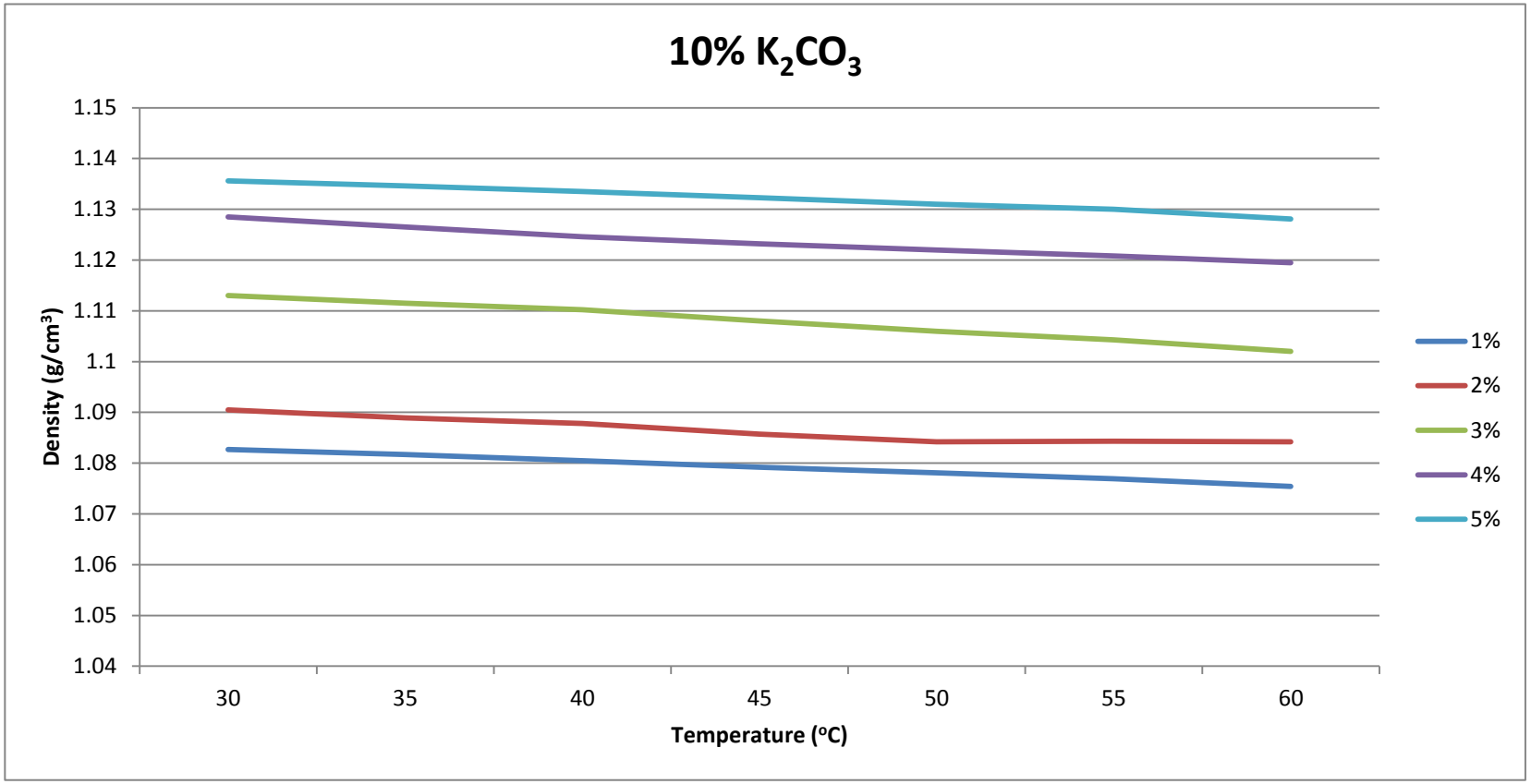

Figure 1: Graph of density for 10wt% K_2CO_3 + TBAOH at different concentrations and temperatures

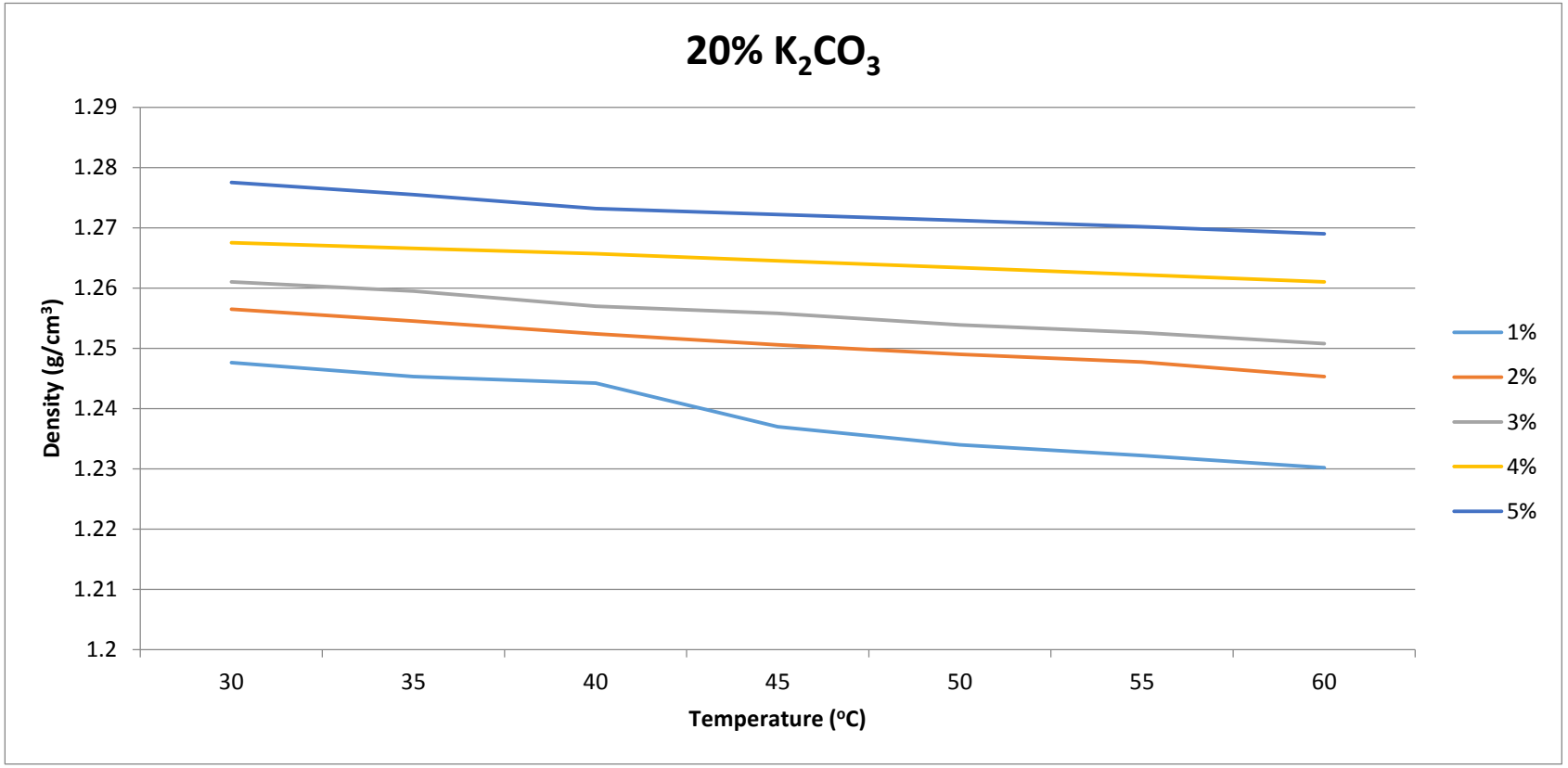

Figure 2: Graph of density for 20wt% K_2CO_3 + TBAOH at different concentrations and temperatures

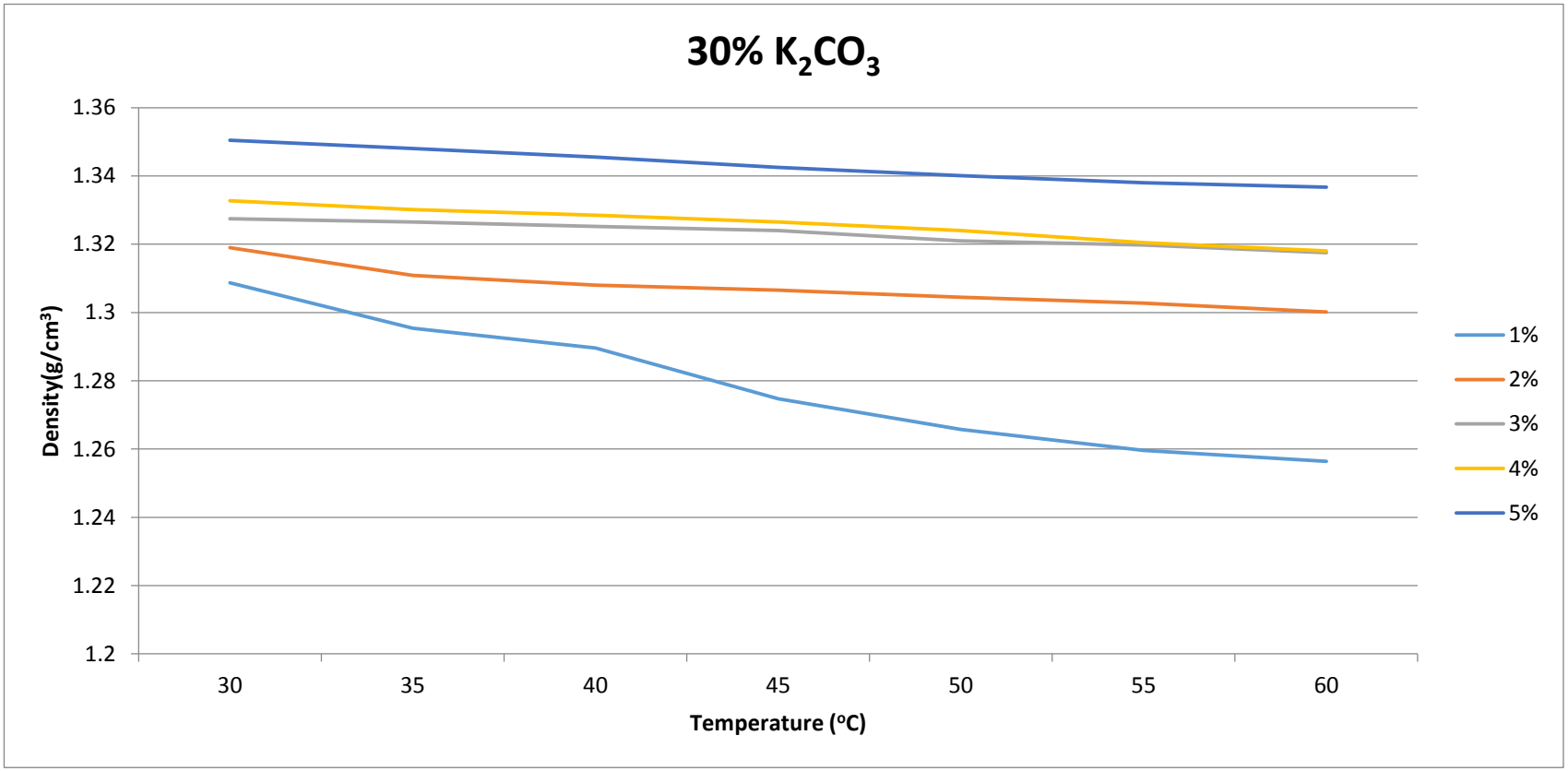

Figure 3: Graph of density for 30wt% K_2CO_3 + TBAOH at different concentrations and temperatures

The graph plotted in figures 1 to 3 shows that the density of the mixtures decreases as the temperature increases for any concentrations. There is also a trend where the density increases as the concentration of TBAOH increases for the same temperature.

The average absolute deviation (AAD) can be determined by using:

$$\sum \frac{1}{n} \left| \frac{x_{exp} - x_{calculated}}{x_{exp}} \right|$$

Where n is the number of data used.

The value of AAD's are calculated for each concentration and temperature and is summarized in the table below.

Table 11: AAD for 10wt% K_2CO_3 at different concentration and temperatures

	Temperature (C)							AAD
TBAOH	30	35	40	45	50	55	60	
1%	1.0827	1.0817	1.0805	1.0792	1.0781	1.0769	1.0754	0.00207
2%	1.0905	1.0889	1.0878	1.0857	1.0842	1.0843	1.0842	0.00219
3%	1.1130	1.1115	1.1102	1.1080	1.1060	1.1043	1.1020	0.00322
4%	1.1285	1.1265	1.1246	1.1232	1.1220	1.1208	1.1195	0.00253
5%	1.1135	1.1346	1.1335	1.1323	1.1310	1.1300	1.1281	0.00469

Table 12: AAD for 20wt% K_2CO_3 at different concentration and temperatures

	Temperature (C)							AAD
TBAOH	30	35	40	45	50	55	60	
1%	1.2476	1.2453	1.2442	1.2370	1.2340	1.2322	1.2302	0.00605
2%	1.2565	1.2545	1.2524	1.2506	1.2490	1.2477	1.2453	0.00309
3%	1.2610	1.2595	1.2570	1.2558	1.2539	1.2526	1.2508	0.00289
4%	1.2675	1.2666	1.2657	1.2645	1.2634	1.2622	1.2610	0.00190
5%	1.2775	1.2755	1.2732	1.2722	1.2712	1.2702	1.2690	0.00232

Table 13: AAD for 30wt% K_2CO_3 at different concentration and temperatures

	Temperature (C)							AAD
TBAOH	30	35	40	45	50	55	60	
1%	1.3087	1.2954	1.2896	1.2747	1.2657	1.2596	1.2564	0.01655
2%	1.3190	1.3109	1.3080	1.3065	1.3045	1.3027	1.3001	0.00450
3%	1.3275	1.3265	1.3252	1.3240	1.3210	1.3198	1.3175	0.00312
4%	1.3327	1.3301	1.3285	1.3265	1.3240	1.3205	1.3180	0.00422
5%	1.3505	1.3480	1.3455	1.3425	1.3401	1.3380	1.3367	0.00425

The values of AAD above is compared with literature to ensure the validity of the data obtained. The literature used as a comparison was from Journal Chemical Engineering, Data 2006, 5I, 2242-2245. Authors Paul S. and Mandal B.

The AAD's obtained are less than 2% thus the data obtained are in reasonable agreement with the past studies.

CHAPTER 5 RECOMMENDATION AND CONCLUSION

The density of TBAOH, water and K_2CO_3 blends were measured at different temperatures and concentrations. From the experimental data obtained, the densities of the blends tend to decrease as the temperature increases but increases as the concentration of TBAOH and/or K_2CO_3 increases. These data that were obtained were compared with literature's data to ensure that the data obtained during the experiment is within the acceptable limit or range.

Future recommendation for this project would be to conduct CO_2 solubility experiments to test the solubility of these ionic liquids with its promoter. The usage of other promoter to promote CO_2 absorption should be experimentally test to ensure the effectiveness of the blends in capturing CO_2.

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