Viscosity Measurement and Physical Correlation of Aqueous Blends of Potassium Carbonate and Ammonium-Based Ionic Liquid

By

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(14778)

Dissertation submitted in partial fulfilment of
the requirement for the
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(CHEMICAL ENGINEERING)

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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 acknowledgments, that the original work contained herein have not been undertaken or done by unspecified sources or persons.

ANWAR FARID BIN SHAHUDIN

ABSTRACT

Release of carbon dioxide into atmosphere by sources such as chemical industries, open burning and motorised vehicles poses various detriments towards people, animals and environment alike. The effort of absorbing carbon dioxide has been implemented and ways to improve the performance is continuously discovered. The know-how of physical and thermal property such as viscosity solvent systems are prerequisite in analysing and evaluating mass transfer and CO_2 capture capacity for the rational design and optimization of acid gas treatment processes. Viscosity data of these aqueous solutions are also vital for the separation of acid gases using microporous membranes as a gas-liquid contactor. However, the data for the blends of potassium carbonate (K_2CO_3) + tetrabutylammonium hydroxide (TBAOH) is still unavailable, which creates a knowledge gap to use these types of IL and their blends for various purposes. The study will be done by measuring the viscosity of blends in temperatures 303.15 K to 333.15 K (30^{o}C – 60^{o}C) and varying concentrations. The viscosity will be calculated using Hoppler's principle and least-square method to calculate deviation between values.

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CHAPTER 1: INTRODUCTION

1.1 Background of Study

It is now scientifically proven that human activities have caused the increase significantly in concentrations of greenhouse gases (GHGs) over the last 2 centuries, contributing to the global warming. Therefore, the necessity to relieve this problem has resulted in serious environmental concerns deriving from the need to reduce GHG emissions from industrial sources. Global and national emission reduction targets were set, signed, and approved or consented by 124 countries under the 1997 Kyoto Protocol [1]. Davis and Cornwell [2] stated that the major GHGs that contribute to the problem are methane (CH4), carbon dioxide (CO2), nitrous oxide (N2O) and halogens such as chlorofluorocarbons (CFCs) and hydrofluorocarbons (HFCs). Among these, CO2 is the primary contributor to the problem due to its abundance, and is thus a major target for reduction. 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 [3], [4], [5], [6-8]. The thermophysical data OF blends of potassium carbonate (K_2CO_3) and tetrabutylammounium hydroxide (TBAOH) is elusive, which creates gap of knowledge regarding usage of these types of IL and their blends for different applications. In the present work, viscosity of TBAOH (aq) and K_2CO_3 (aq) as blended solutions over a wide range of temperatures (303.15-333.15) K and concentrations are being studied and reported systematically.

1.2 Problem Statement

The know-how of physical and thermal property such as viscosity solvent systems are prerequisite in analysing and evaluating mass transfer and CO_2 capture capacity for the rational design and optimization of acid gas treatment processes. Viscosity data of these aqueous solutions are also vital for the separation of acid gases using microporous membranes as a gas-liquid contactor. However, viscosity data for blends of potassium carbonate (K_2CO_3) + tetrabutylammonium hydroxide (TBAOH) remains elusive, which creates a knowledge gap to use these types of IL and their blends for various purposes.

1.3 Objectives

The objectives of this study are:

1) To measure and present new data of the viscosity of aqueous solution of K_2CO_3 and TBAOH blends.
2) To study the influence of temperature and concentration change upon the physical property of aqueous solution of K_2CO_3 and TBAOH blends.
3) To execute correlation study on the measured values of physical property as a function of temperature by using standard equations of the least-squares method.

1.4 Scope of Study

Viscosity 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 physical property of the aqueous blends of are to be measured for temperatures ranging from (303.15 to 333.15) K and various concentrations.

CHAPTER 2: LITERATURE REVIEW

2.1 Viscosity of Fluid and Newton's Law

Fluid when subjected to stress will deform, that is, flow at velocity proportional to stress and shows resistance towards this stress. Presence of force that resists relative movement of adjacent layers in fluid is referred as viscosity [9].

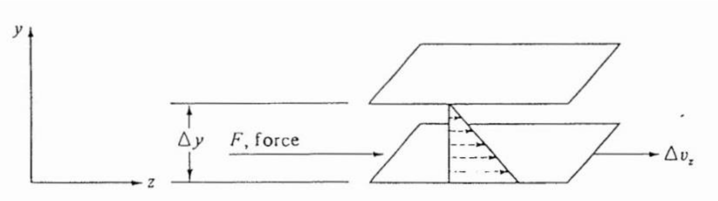


Figure 1: Fluid shear between two parallel plates

In Figure 1 above, a fluid in laminar flow is contained between two long parallel plates. Bottom plate is moving in parallel to top plate and at relatively greater velocity than top plate. Bottom plate moves at constant velocity Δv_z m/s due to application of steady force as much as F Newton. The plates are Δy m apart and each layer of fluid moves in z direction. Layer immediately adjacent to bottom plate is carried along at the velocity of this plate. Each successive layer above one another move at slightly lower velocity as we go up in y direction. The velocity profile is linear in y direction.

$$\frac{F}{A} = -\mu\frac{\Delta v_z}{\Delta y}$$

Figure 2: Newton's law of viscosity

For laminar flow, it has been discovered that force, $F$is directly proportional to area, A and to velocity, Δv_z as well as inversely proportional to distance Δy as shown in Newton's Law of Viscosity (Figure 2) where μ is a proportionality constant referred as viscosity in Pa.s or kg/m.s . Fluid that obeys Newton's Law of Viscosity are called Newtonian fluid and exhibit linear relation between shear stress, (F/A) and velocity gradient (dv_z/dy) while viscosity is constant and independent of rate of shear (velocity gradient).

2.2 Significance of CO_2 Removal

CO_2 removal from gas streams is an important operation in industrial processes. In industries, the acidic nature of CO2 in natural gas causes reduction in heating value and can cause corrosion in process equipment due to its presence. CO2 also threatens to poison the catalysts in the ammonia synthesis process [10]. From the environmental point of view; the alarming rate of CO_2 emissions to the atmosphere has it fair share in the global warming issue, responsible for almost 60% of the enhanced greenhouse effect [11]. 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 attracted the spotlight, with numerous technologies having been developed and applied over the years in an effort to continuously improve and find better solutions.

2.3 Chemical Absorption and Industrial Absorbents for CO_2 Removal

Currently, there are few technologies 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 regarded as one of the reliable and effective methods for CO_2 capture. The most widely used absorbents in carbon removal from process gas streams are mixtures of aqueous alkanolamine solutions such as monoethanolamine (MEA), diethanolamine (DEA), and methyldiethanolamine (MDEA). These solvents have sealed their position in the market and possess commercial importance due to its cheap price, high CO_2 absorption capacity and high water solubility, among other attributes [12]. As time goes by, the disadvantages of these solutions managed to cancel out their merits. These include low CO_2 loading, high energy consumption, high viscosity as well as operational factors such as corrosion and fouling of process equipment. Consequently, the 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 valuable as they combine the higher CO_2 loading of the tertiary amine with the higher reaction rate of the primary or secondary amine [13]. Achieving this would put the best of both worlds while at the same time suppressing the more undesirable characteristics, thus producing excellent blends of absorbents. Meanwhile, sterically hindered amines such as 2-amino-2-methyl-1-propanol (AMP) have been proved to have even higher absorption capacity, absorption rate and selectivity compared to the conventional amines [14].

2.4 Potassium Carbonate

Potassium carbonate's potential as a CO_2 absorbent has been renowned since the early 1900s. The process evolved throughout the years into a viable commercial process, often used in treating synthesis gas [15]. The ideal example is a 40 wt% K_2CO_3 solution in an isothermal absorber/stripper at 100^{o}C and 15 to 20 atm.

There are two studies [16], [17] on commercial validation that show essential pilot plant characterization of hot potassium carbonate (hotpot) versus aqueous MEA and concluded that, under specific configurations, hotpot is an efficient CO2 absorbent. The absorption of CO_2 into aqueous K_2CO_3 is commonly represented by the overall reaction

$$CO_3{}^{2-} + H_2O + CO_2(aq) \leftrightarrow 2HCO_3{}^{-} \qquad (1)$$

though the reaction is usually described in terms of two parallel, reversible reactions.

$$CO_2(aq) + OH^- \leftrightarrow HCO_3{}^{-} \qquad (2)$$

$$HCO_3{}^{-} + OH^- \leftrightarrow H_2O + CO_3{}^{2-} \qquad (3)$$

Since the reaction with hydroxide is the rate-limiting step, the reaction rate is represented as a second order rate expression.

$$r_{CO_2} = k_{OH^-}[OH^-][CO_2] \qquad (4)$$

This reaction, though significant to the solution equilibrium, is generally much sluggish than aqueous amines thus, limiting its application in processes high percentage of removal is needed. To increase the absorption rate, it is often advantageous to add a promoter. The energy required to reverse the reaction is typically less than that required for amine solvents.

2.5 Ammonium-Based Ionic Liquid

Ammonium-based ILs promises high potential since they are water soluble, hydrolytically stable and non-toxic in nature. They can be used alone or blended with other solvents as an aqueous solution for CO_2 absorption like conventional alkanolamines [18–20]. Ammonium-based ILs have shown a wide range of scientific applications in biochemical process [21-23], separation technology [24-26], and chemical synthesis [27], [28]. Numerous studies have been done to variety of ammonium-based ionic liquids, promoters as well as their mixtures. For instances, Umapathi et al. [28] on the other hand studied density, viscosity, density, ultrasonic sound velocity, refractive index and hydrogen bonding of mixtures of diethylammonium acetate, triethylammoniumacetate, diethylammonium hydrogen sulfate, triethylammonium hydrogen sulfate, trimethylammonium acetate and trimethylammonium hydrogen sulfate with water. Bhattacharjee et al. [29] studied thermophysical properties of quarternary ammonium based ionic liquids with (Tf_2N) anions while Chhotaray et al. [30] studied thermophysical properties of ammonium and hydroxylammonium protic ionic liuids with propylammonium, 3-hydroxy propylammonium as cations and formate, acetate, trifluoroacetate as anions. Taib and Murugesan studied the density and excess molar volumes for bis (2-hydroxyethyl) ammonium acetate with water over the entire composition range [31] while Alvaresz et al. tackled density and ultrasonic sound velocity data for 2-hydroxy ethylammonium acetate with water throughout the whole concentration range [32]. Xu et al. [33], [34] reported the thermophysical properties of ethylammonium acetate with water and n-butylammonium acetate or n-butylammonium nitrate with water over the whole concentration range. Zarkewska et al. [35] explored solubility of carbon dioxide into tetrahexylammonium bromide $[(C_6H_{13})_4N][Br]$, tetrabutylammoniumtetrafluoroborate $[(C_4H_9)_4N][BF_4]$, trioctylmethylammoniumtosylate $[(C_8H_{17})_3(CH_3)N][tos]$, trioctylmethylammoniumtrifluoromethanesulfonate $[(C_8H_{17})_3(CH_3)N][CF_3SO_3]$ and didodecyldimethylammonium saccharine $[(C_{12}H_{25})_2(CH_3)_2N][sac]$. Usula et al. studied mixing properties of ethylammonium nitrate (EAN), N-propylammonium nitrate (PAN), N-butylammonium nitrate (BAN) 2-methoxyethylammonium nitrate (MEOEAN) with solvent, N-methyl-2-pyrrolidone (NMP) [36]. However, there are still void on study of viscosity of ammonium-based ionic liquids blended with potassium carbonate and water so, this study intended to do just that. Data on viscosity is vital for designing acid gas removal systems.

CHAPTER 3: METHODOLOGY

3.1 Chemicals & Equipment Required

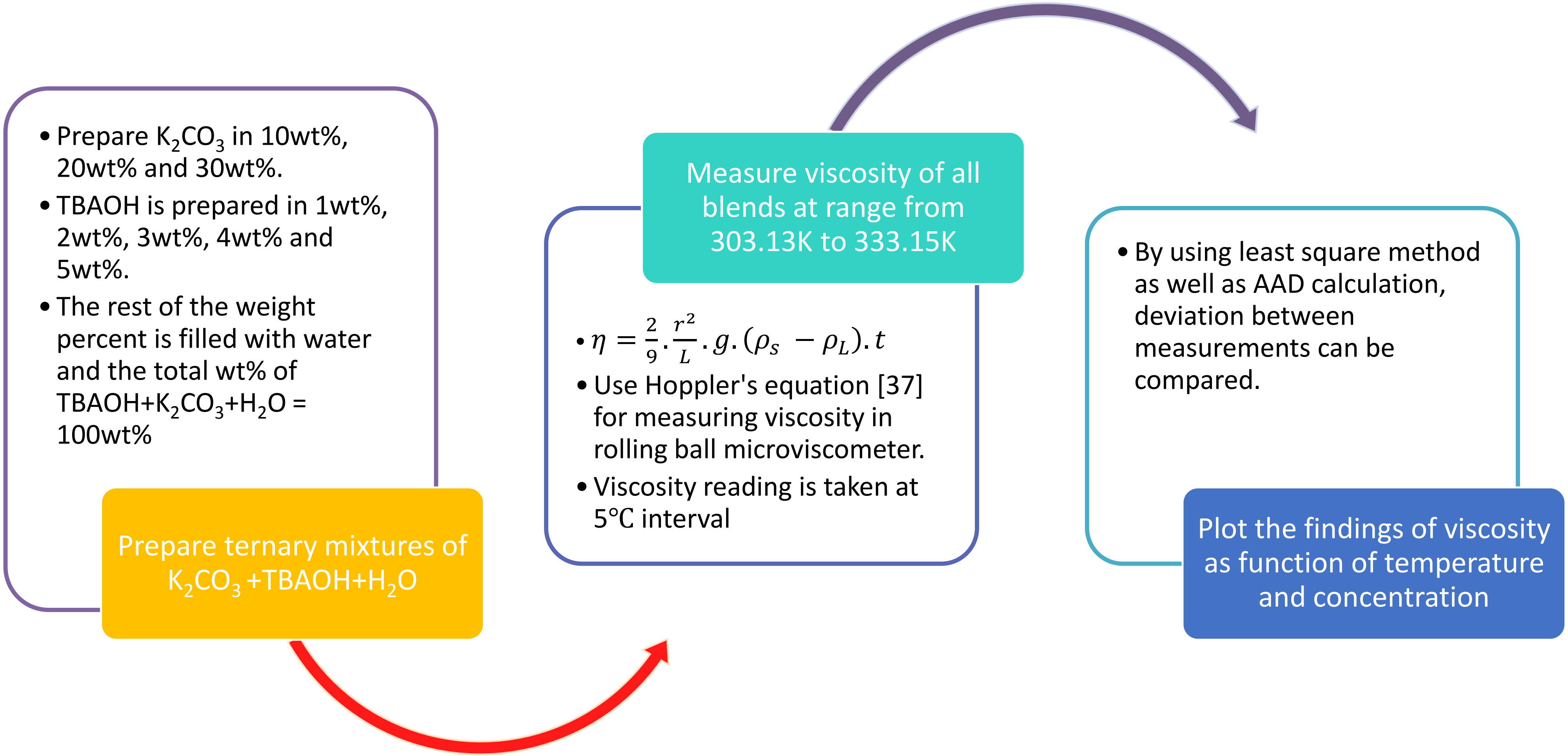
Table 1: List of Chemicals Used

Chemicals	Purity (%)	Supplier
Tetrabutylammonium hydroxide	99.9	Benua Sains Sdn. Bhd.
Potassium carbonate	99.9	Merck Malaysia
Deionised Water	99.9	Merck Malaysia

Table 2: Equipment used

<table>
<tr><td colspan="2">Equipment</td><td>Microviscometer</td></tr>
<tr><td colspan="2">Model</td><td>Anton Paar (LOVIS 2000M)
</td></tr>
<tr><td colspan="2">Measuring Range</td><td>0.3 mPa.s to 10000 mPa.s</td></tr>
<tr><td rowspan="2">Viscosity</td><td>Accuracy</td><td>± 0.5 %</td></tr>
<tr><td>Repeatability</td><td>± 0.1 %</td></tr>
<tr><td rowspan="2">Temperature</td><td>Accuracy</td><td>± 0.02 K</td></tr>
<tr><td>Repeatability</td><td>± 0.005 K</td></tr>
</table>

3.2 Flow of Work



3.3 Gantt Chart and Key Milestone

Table 3: Gantt chart of FYP I September 2014

		Week													
No	**Detail**	**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 resumes														
6	Submission of Interim Draft Report														
7	Submission of Interim Report														

Table 4: Gantt chart of FYP II January 2015

		Week													
No	**Detail**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
1	Project Work Continues														
2	Progress Report Submission														
3	Project Continues														
4	Pre-SEDEX														
5	Draft Final Report Submission														
6	Dissertation Submission (Soft Bound)														
7	Technical Paper Submission														
8	Viva														
9	Dissertation Submission (Hard Bound)														

Process	
Milestone	

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Viscosity Measurements

Viscosity of aqueous solution of potassium carbonate and tetrabutylammonium hydroxide (PC+TBAOH) were experimentally measured for 15 different concentrations over a wide range of temperature. The acquired results are shown as follows:

Table 5: Viscosity of 10wt% K_2CO_3 + wt% TBAOH

		Viscosity (mPa.s)				
K_2CO_3 (wt%)		10				
TBAOH (wt%)		1	2	3	4	5
Temp (°C)	Temp (K)					
30	303.15	1.2852	1.2955	1.3075	1.3135	1.3195
35	308.15	1.283	1.2945	1.3065	1.3125	1.3180
40	313.15	1.2801	1.2932	1.3054	1.3114	1.3165
45	318.15	1.278	1.292	1.3044	1.3101	1.3151
50	323.15	1.2752	1.2906	1.3031	1.309	1.314
55	328.15	1.273	1.2894	1.3024	1.3075	1.3126
60	333.15	1.2701	1.2878	1.3003	1.3061	1.3116

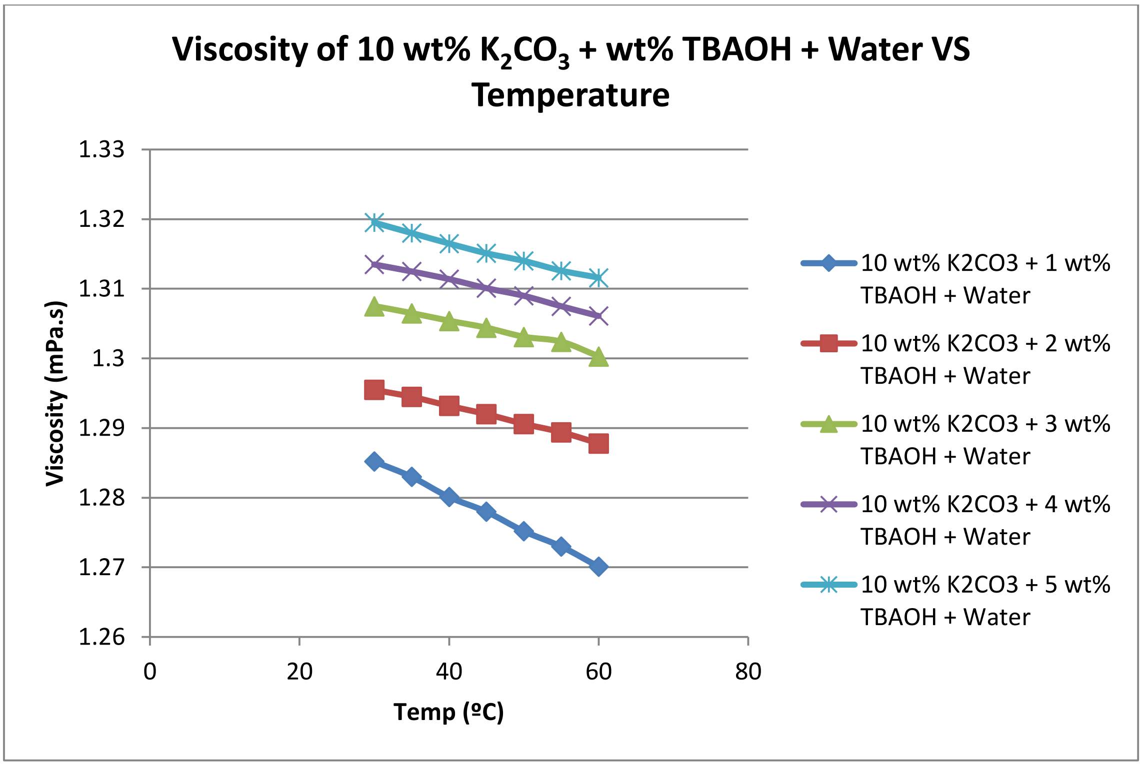


Figure 3: Plot of Viscosity of 10wt% K_2CO_3 + wt% TBAOH + Water against Temperature (30-60˚C)

Table 6: Viscosity of 20wt% K_2CO_3 + wt% TBAOH

		Viscosity (mPa.s)				
K_2CO_3 (wt%)		20				
TBAOH (wt%)		1	2	3	4	5
Temp (°C)	Temp (K)					
30	303.15	1.3325	1.341	1.3490	1.3545	1.3596
35	308.15	1.3302	1.3395	1.348	1.3525	1.3582
40	313.15	1.3285	1.3385	1.3471	1.3515	1.3570
45	318.15	1.3265	1.3375	1.346	1.3503	1.355
50	323.15	1.3235	1.3365	1.3449	1.3491	1.3525
55	328.15	1.3215	1.3355	1.3438	1.3478	1.3501
60	333.15	1.3201	1.3345	1.3427	1.3466	1.3485

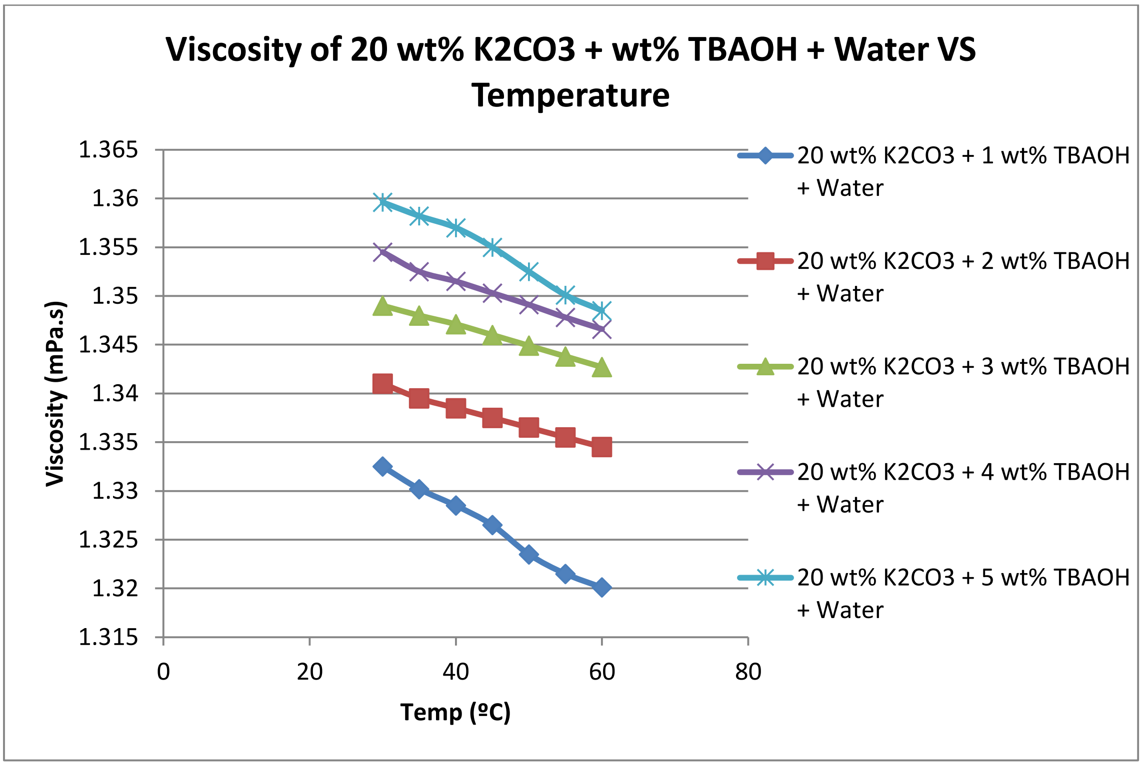


Figure 4: Plot of Viscosity of 20wt% K_2CO_3 + wt% TBAOH + Water against Temperature (30-60˚C)

Table 7: Viscosity of 30wt% K_2CO_3 + wt% TBAOH

		Viscosity (mPa.s)				
K_2CO_3 (wt%)		30				
TBAOH (wt%)		1	2	3	4	5
Temp (°C)	Temp (K)					
30	303.15	1.3596	1.3635	1.3705	1.3755	1.3795
35	308.15	1.3585	1.3625	1.369	1.3735	1.3785
40	313.15	1.3575	1.3615	1.3675	1.3715	1.3775
45	318.15	1.3564	1.3602	1.366	1.3705	1.376
50	323.15	1.3534	1.3589	1.3645	1.367	1.3743
55	328.15	1.3523	1.3573	1.363	1.3655	1.3721
60	333.15	1.3512	1.356	1.3615	1.363	1.371

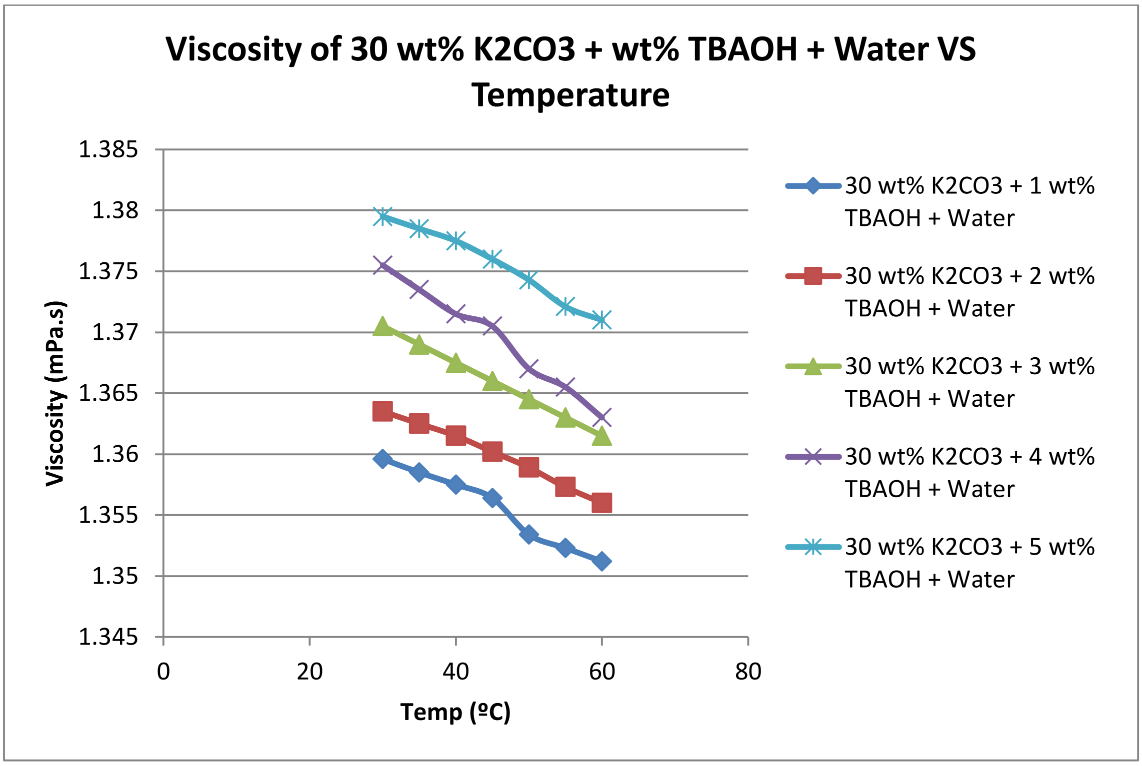


Figure 5: Plot of Viscosity of 30wt% K_2CO_3 + wt% TBAOH + Water against Temperature (30-60˚C)

From Figure 3, Figure 4 and Figure 5, it is apparent that the viscosity decreases with increasing temperature and with increasing concentration of potassium carbonate and tetrabutylammonium hydroxide in the aqueous solutions, the viscosity tend to increases.

4.3 Viscosity Fitting and Prediction

In order to obtain the fitting result, the experimental data were characterized as a form viscosity, η versus temperature T. Correlations were developed to allow the prediction of the viscosity of the aqueous solutions. Correlations of the viscosity as a function of temperature are found using least-square method and regression line by means of the experimental data. The following expressions are used for the regression equations of the experimental data

$$\eta = m.T + q \qquad (1)$$

where η is the viscosity in mPa.s, T is the temperature in ℃, m, and q are fitting parameters. To evaluate accuracy of prediction, average absolute deviation is calculated by using this equation:

$$AAD = \frac{1}{N}\sum_{i=1}^{N}\frac{|\eta_{exp,i}-\eta_{cal,i}|}{\eta_{exp,i}} \qquad (2)$$

Where N is total number of samples, subscript exp and cal denotes experimental and calculated values from regression equation respectively. The values of the parameters and average absolute deviations (AAD) are listed in Table 8. Eq. (2) correlates the viscosity values of the temperature with an AAD of 0.079% and the results are completely acceptable. It illustrates that the prediction from Eq. (1) is in good agreement with the experimental values and can be used as theory calculation and engineering designs.

Table 8: Fitting parameters and average absolute deviation

K_2CO_3 (wt%)	TBAOH (wt%)	m	q	R^2	AAD
10	1	-0.0005	1.3004	0.9988	0.0001453
	2	-0.0003	1.3034	0.9972	0.0015156
	3	-0.0002	1.3145	0.9862	0.0009753
	4	-0.0002	1.3211	0.9960	0.0015928
	5	-0.0003	1.3272	0.9963	0.0012387
20	1	-0.0004	1.3453	0.9942	0.0008948
	2	-0.0002	1.3471	0.9957	0.0003953
	3	-0.0002	1.3554	0.9989	0.0003503
	4	-0.0003	1.3617	0.9937	0.001577
	5	-0.0004	1.3718	0.9871	0.0004958
30	1	-0.0003	1.3690	0.9744	0.000295
	2	-0.0003	1.3714	0.9938	0.0015343
	3	-0.0003	1.3795	1.0000	0.000000
	4	-0.0004	1.3881	0.9845	0.0005219
	5	-0.0003	1.3889	0.9988	0.0002595

4.3 Discussion

After analysing results from Figure 3, Figure 4 and Figure 5, it is apparent that the viscosity decreases with increasing temperature. This is possibly due to the decrease in the internal resistance of the molecules with increasing temperature, which allows the solution molecules to flow easily, thereby decreasing the viscosity. However, increase in concentration of potassium carbonate and tetrabutylammonium hydroxide in the aqueous solutions causes the viscosity of aqueous blend to increase. The higher concentration solutions have higher viscosity than the lower ones, due to the fact that molecular resistance increases in the higher concentration solutions. This explanation is consistent with Mannar's [38] result.

Kar and Arslan [39] also described dependency of viscosity from concentration and temperature form molecular point of view. As solution is heated, thermal expansion causes intermolecular distance to increase which in turn reduces resistance among molecules. Increase in viscosity due to concentration is described due to increase of number of hydroxide and hydrogen bonding which in turn increases fluid resistance and increases viscosity.

By comparing the results and explanations from both literatures, it is consistent to the results of this experiment which shows a predictable behaviour since all fluid studied here are Newtonian fluids. The correlation made by least square method and AAD as low as 0.079% shows that this findings may be used for theoretical calculation and engineering designs.

CHAPTER 5: CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Viscosity of aqueous solution of potassium carbonate activated by tetrabutylammonium hydroxide were measured at a wide range of temperature from 30 to 60 ˚C. With increasing temperature, viscosity tends to decrease while increasing solution concentration increases the viscosity. By using least-square method, correlation between temperatures and viscosity for all blends can be made and shows positive outcome for application in engineering designs

5.2 Recommendation

1. Perform further study on thermophysical properties in other ranges of temperature and concentrations. Consequently, perform physical correlation with least-square regression to observe consistency in behaviour.
2. To study the feasibility of having the blend K_2CO_3/TBAOH as the CO_2 removal agent in the gas processing plant.

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