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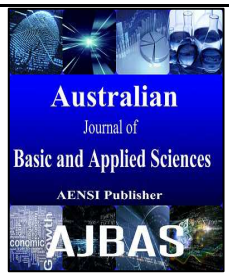

Physicochemical Properties of Aqueous Potassium Salt of α-Methylalanine (K-AMALA) as an absorbent for CO_2 removal

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ABSTRACT

Background: α-methylalanine (AMALA) is one of the sterically hindered amino acids which have potential for CO_2 removal. They have similar amine functionality as alkanolamine solvents and behave in a similar way during CO_2 capture. It is found that sterically hindered amino acids (SHAA) shows better performance in terms of absorption and desorption. However there are few studies to be carried out on this solvent for their commercial applicability. **Objective:** This work focuses on the physicochemical properties such as density, viscosity, and refractive index of aqueous potassium salt of α-methylalanine (K-AMALA) at concentrations ranging from 0.05 to 0.30 mass fractions and temperatures from 298.15 to 333.15 K. Additional objectives of this work is the determination of thermal expansion coefficients by using the density data and the correlation with temperature of each physical property by least square method. **Results:** It was found that the densities, viscosities, and refractive indices of the aqueous solutions of K-AMALA decrease significantly with increasing temperature at all concentrations and whereas with increasing the concentration, all three properties increase. The coefficient of thermal expansion increased at high temperatures and concentrations. **Conclusion:** The correlations for all measured properties were satisfactory over the whole range of temperatures and concentrations. The data presented in this work will enhance the body of the literature and will be helpful for the design of the CO_2 removal process.

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INTRODUCTION

Carbon dioxide (CO_2) is a well-known greenhouse gas which contributes toward global warming. Estimates have indicated that, the combustion of fossil fuels such as coal, oil and natural gas have contributed to about 80 % of world energy (Abu-Zahra *et al*. 2007, Kang *et al*. 2013). Due to this reason, the large amounts of CO_2 released into the atmosphere which adversely affect the climate on earth. National Oceanographic and Atmospheric Administration (NOAA) reported that the CO_2 concentrations have increased 2.25 ppm per year from 2012 to 2014 and the present results of the CO_2 content show that the monthly global average concentration is already exceed 400 ppm in March 2015 (NOAA 2015). Therefore, many studies have been done on capturing and storing CO_2 from the combustion of power plant in order to alleviate the CO_2 emissions from the atmosphere. As a result, various technologies have been introduced by researchers such as absorption, adsorption, membrane, and cryogenic processes. One of the method is absorption by chemical solvents, since that has proven experimentally to be effective in power plant applications and extensively used for decades (Kang *et al*. 2013). In the past, amine based solvents such as monoethanolamine (MEA), diethanolamine (DEA), triethanolamine (TEA), methyldiethanolamine (MDEA) have been used for this application. Over the years, a lot of investigations have been carried out that pointed out several shortcomings by these solvents. It includes the short life cycle of solvents since they have lower resistances to oxidative and thermal degradation, high volatility which result in loss of solvent, causing the corrosion of equipment, higher regeneration energy and toxicity (Meisen and Shuai 1997, Shuaib Shaikh Muhammad 2014, Aronu, Hartono and Svendsen 2011, Portugal *et al*. 2009). These shortfalls have introduced new challenges to overcome, and it became significant for researchers to search for the potential solvents which offer better operating efficiency than amine solvents.

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Recently, amino acid solvents have been identified as a good candidate for CO_2 absorbents. Hook *et al*. (1997) and Kumar *et al*. (2003) have reported that amino acids have similar amine functionality as amines and behave in a similar way during CO_2 capture. They also found that amino acid based solvent systems have higher resistance to oxidative, thermal degradation and less metal-corrosion (Hook 1997, Kumar *et al*. 2003). According to Brouwer *et al*. (2005), Majchrowicz *et al* (2009), and Park *et al* (2014), the amino acid salts precipitate when the absorbent reaches at a certain concentration. Moreover, due to the ionic nature of amino acid salts, these have negligible volatility (J. P. Brouwer 2005, Majchrowicz, Brilman and Groeneveld 2009, Park, Song and Park 2014). In another work, Eide-Haugmo *et al*. (2009) and Aronu *et al*. (2011) have found that amino acid based solvent systems are less toxic and environmental friendly since they produce less toxic degradation products (Eide-Haugmo *et al*. 2009, Aronu *et al*. 2011). These potential advantages of amino acids make it essential to carry out a detail investigation on these solvent systems. Various studies have been carried out on amino acid salts as a solvent for CO_2 capture such as glycine, taurine, alanine and proline (Portugal *et al*. 2007, Holst, Kersten and Hogendoorn 2008, Kim *et al*. 2012, Wei, Puxty and Feron 2014, Tirona *et al*. 2014, Shaikh *et al*. 2014, NOAA 2015, Shaikh *et al*. 2015).

In 2012, Song *et al*. have evaluated the cyclic CO_2 absorption performances of several, linear, sterically hindered, cyclic, and poly amino acids. It was observed that sterically hindered amino acid such as α-methylalanine (AMALA) show higher net cyclic capacity compared to other linear, cyclic, and poly amino acids (Song *et al*. 2012). Very few studies are available in open literature on this solvent system. This encourages us to select AMALA as an absorbent for CO_2 capture in this work. Thus, in the present study physicochemical properties such as density, viscosity, and refractive index of aqueous potassium salt of α-methylalanine (K-AMALA) are presented. All measurements have been done at temperature range of 298.15 to 333.15 K and at concentrations from 0.05 to 0.30 mass fractions.

1 Experimental Section:

α-methylalanine (≥ 98 % pure), and potassium hydroxide (≥ 99 % pure) were purchased from Merck Sdn. Bhd, Malaysia. They were used without further purification. For the preparation of K-AMALA salt solution, amino acid, AMALA was neutralized with an equimolar amount of potassium hydroxide, following literature procedure (Shaikh *et al*. 2013, Shaikh *et al*. 2015). All weight measurements were performed in an electronic analytical balance (Sartorius, model BSA-224S-CW) with $\pm 1.10^{-4}$ g accuracy. The purpose of neutralization reaction is to activate the amino group of AMALA. Samples with different mass fractions (0.05, 0.10, 0.20 and 0.30) were used at different temperatures from 298.15 K to 333.15 K.

Measurement of density:

Digital Anton Par density meter (DMA-4500 M) with $\pm 5.10^{-5}$ g.cm^{-3} accuracy was used to measure the densities of K-AMALA solutions. Before filling the samples into the measuring cell, the calibration steps are required in order to validate the quality of the measurements. The equipment was calibrated using pure water at 293.15 K. Each sample was measured for three times and the average values of the density were reported. The uncertainty of density and temperature were $\pm 6.10^{-5}$ g.cm^{-3} and ± 0.01 K respectively.

Measurement of viscosity:

For the measurement of viscosities of the aqueous solutions of K-AMALA, a digital rolling ball micro viscometer (Anton Par, model; Lovis-2000 M/ME) with $\pm 5.10^{-3}$ mPa.s accuracy was used in this work. After selecting a suitable capillary and ball, the capillary tube must be cleaned with acetone and air-dried before filing the samples in order to obtain accurate results and to avoid the sample contamination. Before carrying out the experiment, the equipment was calibrated with pure water to ensure proper measurement. The capillary filled with sample should be free from the air bubble. The reason is air bubbles will slow down the movement of the ball and give faulty results. The measuring procedure of viscosity is based on the rate of the rolling ball in the samples from certain angle. With triplicate runs, the viscosities in mPa.s of each concentration of K-AMALA solutions were obtained by the average of three measurements. The uncertainties for viscosity and temperature were $\pm 7.10^{-3}$mPa.s and ± 0.02 K, respectively.

Measurement of refractive index:

Refractive index of each sample was measured using a digital automatic refractometer (Antor Par, model; Abbemet) with $\pm 4.10^{-5}$ n_D accuracy, at 5 K intervals from 298.15 K to 333.15 K. The wavelength of the illuminating ray was set at 589.3 nm. Before starting the measurement, the measuring prism was cleaned thoroughly with acetone and pure water to eliminate any dirt or stain. To achieve accurate results, the equipment was calibrated with pure water before and after experiment. The measurements were performed in triplicate, and the average of the three readings was reported. The experimental uncertainty of refractive index measurement and temperature was estimated to be $\pm 5.10^{-5}$ n_D and ± 0.03 K, respectively.

RESULTS AND DISCUSSION

Data validation is significant in order to ensure the experimental data is reliable. In this work, the results obtained were compared with literature data on density, viscosity and refractive index of pure water at temperature, T= (298.15, 303.15 and 308.15) K. The comparison results for all properties of water are presented in Table 1. The validity was evaluated by the percent average absolute deviation (% AAD)

between experimental and literature values with the corresponding equation as below.

$$\%\ AAD = \frac{1}{n}\sum \left| \frac{X_{exp} - X_{lit}}{X_{lit}} \right| 100 \quad (1)$$

where n is the number of experimental data points, X_{exp} and X_{lit} are experimental and literature values, respectively.

Table 1: Comparison of experimental data of density (ρ), viscosity (η), and refractive index (n_D) of pure water with literature values.

T/K	*ρ (g.cm^{-3})*		
	This work	*Literature*	*AAD %*
298.15	0.99739	0.997081[a]	0.0269
303.15	0.99599	0.995682[a]	
308.15	0.99438	0.994194[b]	
	η (mPa.s)		
298.15	0.8970	0.8937[c]	0.6020
303.15	0.8050	0.8007[c]	
308.15	0.7290	0.7225[c]	
	n_D		
298.15	1.33285	1.3328[d]	0.0053
303.15	1.33230	1.3322[d]	
308.15	1.33166	1.3316[d]	

a Handbook of Chemistry and Physic, 2008
b Lange's Handbook of Chemistry
c CRC Handbook of Chemistry and Physic, 2004
d American Institute of Physics Handbook, 2005

The results from the experimental work are presented in tables, for densities in Table 2, for viscosity in Table 3 and for refractive index in Table 4. The graphical representation of density, viscosity and refractive index at each temperature are shown in Figs. 1, 2 and 3 respectively. In Table 2, the density of a solution of K-AMALA shows a maximum density at mass fraction w = 0.30 and temperature 298.15 K. This indicates the density of K-AMALA solution is increased as the concentration increases and the temperature decreases (Aronu *et al*. 2011, Aboudheir and ElMoudir 2009, Kadiwala, Rayer and Henni 2010, Chakraborty, Astarita and Bischoff 1986). This is because in high concentration solutions, more molecules present compared to lower concentration solutions. This condition makes the molecules closer to each other and there is limited space for them to move around (Chakraborty *et al*. 1986). So that, when its volume is decreased, the density is increased.

Table 2: Densities (in g.cm^{-3}) of different mass fractions of aqueous potassium salt of AMALA.

Density (ρ), g.cm^{-3}				
T/K	*w = 0.05*	*w = 0.10*	*w.= 0.20*	*w =0.30*
298.15	1.01405	1.03037	1.06494	1.10031
303.15	1.01253	1.02872	1.06301	1.09808
308.15	1.01080	1.02689	1.06093	1.09572
313.15	1.00889	1.02488	1.05870	1.09325
318.15	1.00681	1.02272	1.05637	1.09068
323.15	1.00457	1.02041	1.05387	1.08801
328.15	1.00219	1.01795	1.05127	1.08523
333.15	0.99965	1.01538	1.04856	1.08236

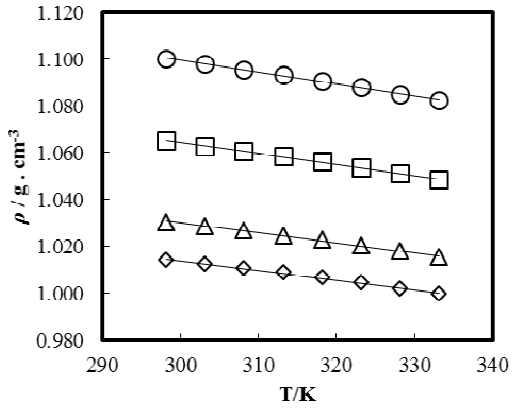

Fig. 1: Densities of different mass fractions of aqueous potassium salt of AMALA as a function of temperature: (◇) 0.05; (△) 0.10; (□) 0.20; (o) 0.30.

Table 3 shows the measured viscosity results for aqueous solutions of K-AMALA at different concentrations and temperature. By increasing the concentration from 0.05 to 0.30 mass fractions and at low temperature of 298.15 K, it can be found that the viscosity increases. The reason is, as the concentration of the solution increased, more numbers of molecules are presented in the solution system and increase the interaction between the molecules. Hence, it is harder for them to move past each other and limit the solution molecules to flow. However, as the temperature increased, the force of attraction between molecules become weaker and reduce the resistance for the solution to flow. As a result, the viscosity of the solution is decreased (Shaikh *et al.* 2014).

Table 3: Viscosities (in mPa.s) of different mass fractions of aqueous potassium salt of AMALA.

Viscosity (η)/ mPa.s				
T/K	*w = 0.05*	*w = 0.10*	*w = 0.20*	*w =0.30*
298.15	0.9156	1.0883	1.4472	2.1076
303.15	0.8186	0.9698	1.2799	1.8465
308.15	0.7375	0.8702	1.1413	1.6306
313.15	0.6689	0.7871	1.0243	1.4522
318.15	0.6106	0.7159	0.9258	1.3028
323.15	0.5610	0.6552	0.8415	1.1758
328.15	0.5178	0.6025	0.7694	1.0674
333.15	0.4805	0.5572	0.7067	0.9753

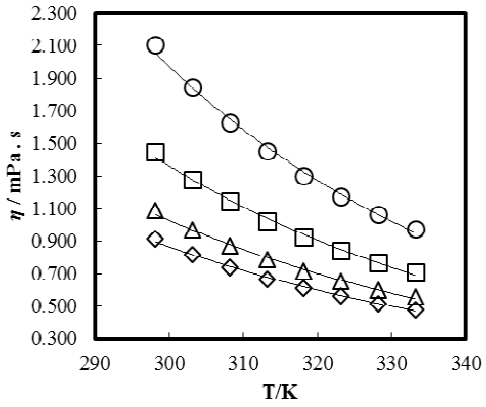

Fig. 2: Viscosities of different mass fractions of aqueous potassium salt of AMALA as a function of temperature: (◇) 0.05; (△) 0.10; (□) 0.20; (o) 0.30.

Based on results for refractive index of K-AMALA in Table 4, it can be observed that the maximum measured values were at high concentration of 0.30 mass fractions and at low temperature of 298.15 K. At this state, more particles strike by the light and lead the refractive index values to increase (Shaikh *et al*. 2014, Murshid *et al*. 2011).

Table 4: Refractive indices (n_D) of different mass fractions of aqueous potassium salt of AMALA.

Refractive indices (n_D)				
T/K	*w =0.05*	*w =0.10*	*w = 0.20*	*w= 0.30*
298.15	1.33904	1.34538	1.35907	1.37305
303.15	1.33837	1.34469	1.35829	1.37217
308.15	1.33769	1.34403	1.35759	1.37137
313.15	1.33697	1.34327	1.35690	1.37058
318.15	1.33621	1.34256	1.35621	1.36996
323.15	1.33545	1.34186	1.35565	1.36946
328.15	1.33466	1.34118	1.35523	1.36916
333.15	1.33388	1.34054	1.35485	1.36912

For the prediction of the results, the measured values for densities, viscosities and refractive indices were correlated as a function of temperature and mass fraction, according to standard equation (2), (3), and (4), respectively.

The best fit of experimental data for density was obtained by solving a linear system of equation, exponential-type equation was used for viscosity data and refractive index data was approximated by the polynomial function.

$$\rho = A_0 + A_1 T \quad (2)$$

where ρ is the density (g.cm^{-3}), A_0 and A_1 are the correlation coefficients, and T is the temperature.

$$\eta = B_0 \exp(-B_1 . T) \quad (3)$$

where η is the viscosity (mPa.s), B_0 and B_1 refers to correlation coefficients.

$$n_D = C_0 + C_1T + C_2T^2 \quad (4)$$

where Z is refractive index, C_0, C_1, and C_2 are the fitting parameters, and T is temperature.

The standard deviations for all properties were calculated by Eq. (5).

$$SD = \left[\frac{\sum_i^n (X_{exp} - X_{calc})^2}{n}\right]^{0.5} \quad (5)$$

where SD is standard deviation, Xexp is the experimental value of the density, Xcalc is the calculated.

The correlation parameters of equation (2), (3) and, (4) were tabulated in Table 5 to 7 while the graphical representations between experimental and predicted values are presented in Figure 4 to 6. As shown, the predicted values are in good agreement with the experimental values with the low values of the standard deviation (SD) between the two set of data. A high R^2 value ($R^2 \geq 0.99$ %) indicates that the data is reliable and these results suggest that these correlations can be used to predict the values of density,viscosity and refractive index in the present aqueous solution.

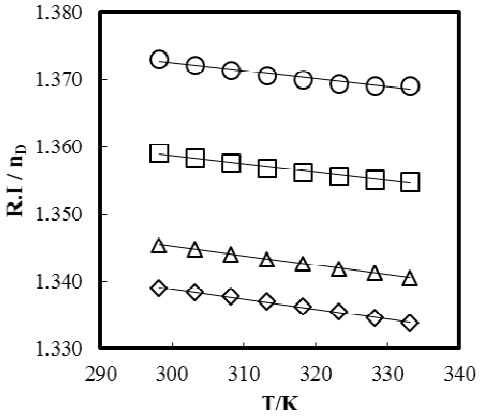

Fig. 3: Refractive indices of different mass fractions of aqueous potassium salt of AMALA as a function of temperature: (◇) 0.05; (△) 0.10; (□) 0.20; (o) 0.30.

Table 5: Correlation parameters and SD for densities of different mass fractions of aqueous potassium salt of AMALA.

Mass fraction, *w*	A_0	A_1	R^2	10^4 SD
0.05	1.13764	-0.000412	0.9935	4.12
0.10	1.15896	-0.000429	0.9948	3.79
0.20	1.20517	-0.000469	0.9969	3.09
0.30	1.25373	-0.000513	0.9983	2.60

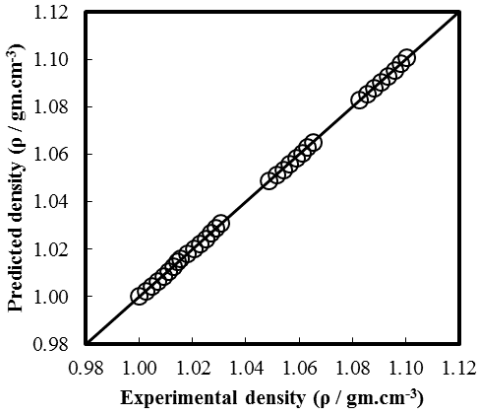

Fig. 4: Comparison between experimental values versus predicted values of density of aqueous K-AMALA.

Table 6: Correlation parameters and SD for viscosities of different mass fractions of potassium salt of AMALA solutions.

Mass fraction, *w*	B_0	B_1	R^2	SD
0.05	214.060	0.018	0.9955	0.0100
0.10	314.130	0.019	0.9959	0.0120
0.20	623.651	0.020	0.9962	0.0160
0.30	1433.518	0.022	0.9960	0.0250

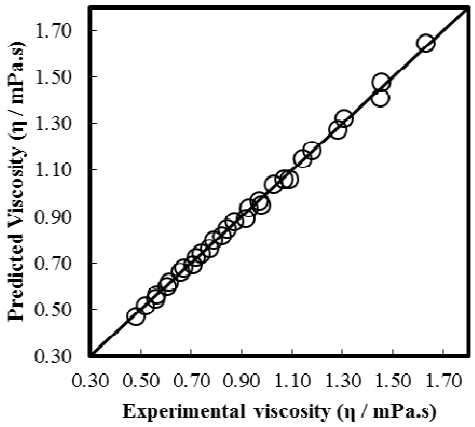

Fig. 5: Comparison between experimental values versus predicted values of viscosity of aqueous K-AMALA.

Table 7: Correlation parameters and SD for refractive index of different mass fractions of aqueous potassium salt of AMALA solutions.

Mass fraction, *w*	C_0	C_1	C_2	R^2	10^{-4} SD
0.05	1.33877	0.0001	-0.0000005	0.9999	3.21
0.10	1.39724	-0.0002	0.0000001	0.9998	2.88
0.20	1.53071	-0.0010	0.0000014	0.9994	0.36
0.30	1.67604	-0.0018	0.0000027	0.9981	0.64

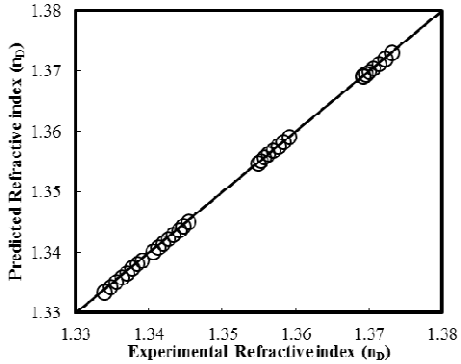

Fig. 6: Comparison between experimental values versus predicted values of refractive index of aqueous K-AMALA.

An Additional goal of this work is the determination of thermal expansion coefficient (α) of K-AMALA solutions. This property is significant to this work in order to measure changes of solution volume in a given temperature. The density data was used to estimate this property and the values of thermal expansion coefficient was determined using the relationship given by Eq. (6).

$$\alpha = -\frac{A_1}{A_0 + A_1 T} \quad (6)$$

where α is thermal expansion coefficient, A_1 and A_0 are the fitting parameters, and T is temperature.

The data of thermal expansion coefficient of K-AMALA solutions for various temperature is shown in Table 8. The obtained results of thermal expansion coefficient (α) indicate that the value of this property increased with increase in temperature and concentration and their trends are shown in Fig. 7.

Table 8: Coefficient of thermal expansion of different mass fractions of aqueous potassium salt of AMALA at different temperatures.

Coeffcient of thermal expansion, α. 10^{-4} / (K^{-1})				
T/K	*w = 0.05*	*w = 0.10*	*w = 0.20*	*w = 0.30*
298.15	4.06	4.16	4.40	4.66
303.15	4.07	4.17	4.41	4.67
308.15	4.08	4.18	4.42	4.68
313.15	4.08	4.19	4.43	4.69
318.15	4.09	4.20	4.44	4.70
323.15	4.10	4.20	4.45	4.72
328.15	4.11	4.21	4.46	4.73
333.15	4.12	4.22	4.47	4.74

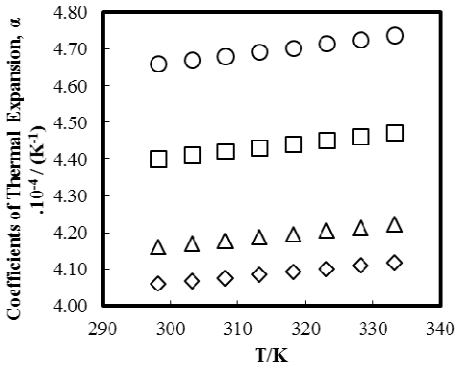

Fig. 7: Coefficient of thermal expansion (α) of different mass fractions of aqueous potassium salt of AMALA as a function of temperature: (◇) 0.05; (△) 0.10; (□) 0.20; (o) 0.30.

In general, the molecules within the solution will expand in volume with an increase in temperature. It was noted that the change in coefficient of thermal expansion of K-AMALA is quite small with respective to maximum and minimum values in the range of 4.06 to 4.72 ($X10^{-4}$) K^{-1}. The similar trends have been reported in the literature for various aqueous systems (Shaikh *et al*. 2015, Shaikh *et al*. 2014).

2 Conclusion:

The experimental data of density, viscosity and refractive index of K-AMALA solutions were measured, for temperature ranging from 298.15 to 333.15 K and 0.05 to 0.30 mass fractions. Based on the experimental results, the measured values of density, viscosity and refractive index decreased with the increase in the temperature at fixed concentration. However, the measured values increased as the concentrations increased. The measurement of the three physicochemical properties in this work were correlated well with the experimental values. For thermal expansion coefficient results, the values showed linear increase with respect to increase in temperature and concentration.

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