

Viscosity Measurement and Correlation of Unloaded and CO₂-Loaded Aqueous Blend of Monoethanolamine and Piperazine

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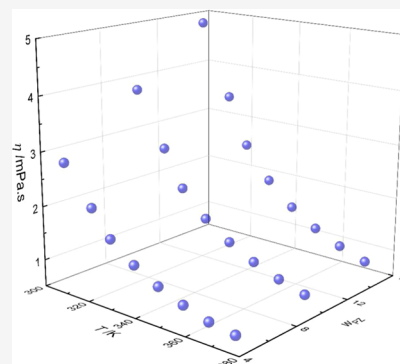


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ABSTRACT: This work provides new viscosity data of unloaded and partially carbonated aqueous monoethanolamine-piperazine (MEA-PZ) solutions at temperatures between 303.15 and 373.15 K and at a N₂ pressure of 400 kPa. In both cases, the viscosity of MEA-PZ solutions was measured at three different concentrations of PZ at 5, 10, and 15 wt % and by keeping the concentration of MEA constant at 30 wt %. For the carbonated solutions, measurements were performed by varying the concentration of CO₂ between 0.13 and 0.55 mol of CO₂/mol of amines. A simple empirical equation and a modified Setschenow equation used to correlate the viscosity of unloaded and CO₂-loaded MEA-PZ solutions, respectively, provide a good representation of the experimental data.



1. INTRODUCTION

Chemical absorption using aqueous alkanolamines is considered the commercial-ready technology to capture flue gas such as carbon dioxide (CO₂) from industrial processes. The flue gas in this case is normally treated after combustion of the fuels, and this method of treating the flue gas is preferred since the process can be retrofitted easily onto existing power plants. The widely used alkanolamine for treating flue gas this way is aqueous monoethanolamine (MEA).^{1–3} In a typical CO₂-capture process,^{2,4} aqueous MEA reacts with flue gas containing CO₂ to produce MEA-carbamate in the absorption column. The MEA-carbamate is then sent to a high-temperature stripper to liberate the CO₂ gas. The treated gas is forwarded for another purpose or sent to storage. The whole CO₂-capture process is energy-intensive: Versteeg and co-workers estimated that a 600 MW coal-fired power plant using MEA as a solvent consumes around 3.8–4.0 GJ/ton CO₂.⁵ This amount of energy constitutes around 10% of the efficiency penalty of such a power plant. As such, efforts are ongoing to reduce the cost associated with CO₂ capture. One way of reducing the cost is by combining two different amines, one of which has high kinetics toward CO₂ and another with a high CO₂ capacity. A number of research articles have shown that this could reduce the energy penalty for CO₂ capture.^{6–9}

One of the amines that has been gaining attention is piperazine (PZ), which is a cyclic diamine.¹⁰ The presence of two available nitrogen atoms on a PZ molecule means that the rate of CO₂ absorption is faster than that of aqueous MEA. Previous studies on PZ also suggested that its thermal degradation to be limited and PZ regeneration required 10–20% less energy than aqueous MEA.^{11–14} Piperazine has been

used with amines such as methyl diethanolamine (MDEA) and 2-amino-2-methyl-1-propanol (AMP) to enhance CO₂ absorption and reduce the energy requirement.^{15–17} A recent HYSYS simulation by Hosseini-Ardali et al. calculated a regeneration energy of around 2.7 GJ/ton CO₂ for MDEA-PZ solution, which is lower than that of the standard 30 wt % aqueous MEA.¹⁸ In this work, aqueous MEA was blended with PZ as another potential amine solution for CO₂ capture.

Data on thermophysical properties of amine blends are necessary for the initial evaluation of amine performance. Liquid phase viscosity data of the amine blend are significant as they inform on the mass transfer, which affects the design of the absorber and the energy requirement of the process. Viscosity data of aqueous MEA are widely reported in the literature;^{19–21} however, viscosity data of the MEA-PZ blend are scarce. We reported in this work new experimental viscosity for aqueous MEA-PZ blend for both unloaded and CO₂-loaded solutions.

2. MATERIALS AND METHODS

The chemicals used in this work are shown in Table 1. Apart from MEA, which was degassed, all other chemicals were used as received. Aqueous solutions were prepared using Type-1 water. For preparation of unloaded solutions, the calculated

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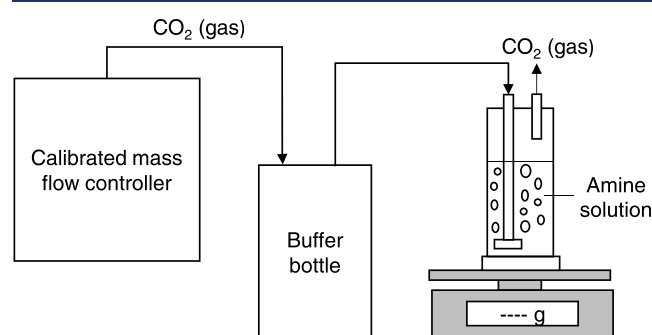


Table 1. Chemicals Used in This Work

chemical name	CAS number	mole fraction purity ^a	supplier	purification
ethanolamine (MEA)	141-43-5	≥0.99	Sigma-Aldrich	degassing
piperazine (PZ)	110-85-0	≥0.99	Sigma-Aldrich	no
carbon dioxide (CO ₂)	124-38-9	0.99999	AGA Norge AS	no

^aAs stated by the supplier.

amount of MEA, PZ, and Type-1 water were weighed using an XS-403S analytical balance from Mettler Toledo (accuracy: ±0.001 g). As an example, a 30–5 wt % MEA-PZ solution was prepared by weighing 30 g of MEA, 5 g of PZ, and 65 g of Type-1 water. The setup used to load MEA-PZ blends with CO₂ is shown in Figure 1. High-purity CO₂ gas (from AGA

Figure 1. Schematic diagram of the CO₂-loading procedure.

Norway, purity 0.9999) was first wetted with Type-1 water before being supplied to the gas washing bottle containing a known amount of unloaded MEA-PZ blend solution. A mass flow controller (Smart-Trak 100 from Sierra Instruments) was used to control the flow of CO₂ gas. Usually, the flow rate was kept at 0.15 L/min. To ensure homogeneity, a stirrer was placed at the bottom of the gas washing bottle. The loading process was stopped when the required amount of CO₂ was reached. To confirm the amount of CO₂ present in the MEA-PZ solution, the CO₂-loaded solutions were also subjected to an acid–base titration, as previously reported.²² In this work, the concentration of CO₂ in the amine blend is represented by CO₂-loading (alpha, α), which is defined as the mole of CO₂ divided by the moles of MEA plus PZ in the solution. It is worth noting here that the CO₂-loaded samples were prepared at nonequilibrium conditions. After preparation, these samples were stored in a refrigerator at a temperature of ~277.15 K until needed.

Viscosity measurements were performed using an Anton Paar Physica MCR 101 rheometer employing a double-gap XL DG pressure cell measuring system. To calibrate the rheometer, we have used a standard oil reference S3 from Paragon Scientifics. Experiments in this work were performed using ~6 mL solution. Dynamic viscosity values were determined using the rheometer automatically from the slope between shear rate and shear stress. Throughout the experiments, the pressure of the measuring system was kept at 400 kPa using high-purity nitrogen gas. Details of the viscosity measurement can be retrieved from our earlier publication.¹⁹ The reported experimental data in this work are the average values from three replicates. As in our previous

publication, the combined standard uncertainties for the unloaded and CO₂-loaded MEA-PZ solutions are calculated to be 0.10 and 0.15 mPa·s, respectively.¹⁹

3. RESULTS AND DISCUSSION

This section discusses viscosities of aqueous amines with and without CO₂-loading. To evaluate our measurement system, we performed viscosity checks using a standard oil reference S3 from Paragon Scientifics at temperatures relevant to this study. Figure 2 displays the measured viscosity in this work plotted

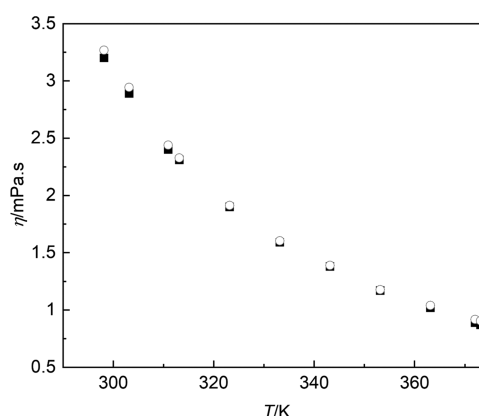


Figure 2. Comparison of viscosity values obtained in this work (■) with values reported for standard oil reference S3 from Paragon Scientifics (○) at different temperatures.

against the reference values from Paragon Scientifics. The values are also tabulated in Table S1. It can be seen from the figure that viscosities measured using our MCR 101 rheometer show a good agreement with the reference values from the manufacturer. In addition, the viscosity of pure MEA was also measured at different temperatures and compared with selected literature data (Table S2).

Values of average absolute deviation (AAD) were calculated using eq 1 to evaluate measured viscosities with values from the standard oil reference, pure MEA, or regression models. In eq 1, η_{exp} , η_{calc} , and N represent the measured viscosity, standard or correlated viscosity, and the number of data, respectively. In the case of the standard oil reference, an AAD value of 0.042 mPa·s was calculated. For pure MEA, a comparison with the literature from Mandal et al.,²³ Song et al.,²⁴ Amundsen et al.,²⁰ Li et al.,²⁵ Li and Lie,²⁶ Xu et al.,²⁷ Geng et al.,²⁸ and Arachchige et al.²⁹ provides AAD values of 0.12, 0.15, 0.12, 0.04, 0.12, 0.09, 0.12, and 0.12 mPa·s, respectively. The low average deviation value suggests that our measurement system works properly and would yield reliable results.

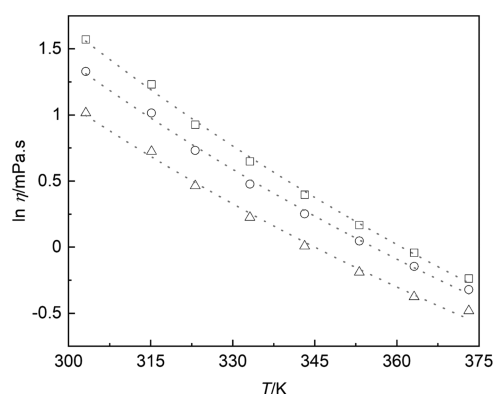
$$\text{AAD} = \frac{\sum |\eta_{\text{calc}} - \eta_{\text{exp}}|}{N} \quad (1)$$

3.1. Viscosity of MEA-PZ Mixtures. Solutions of MEA-PZ were prepared by varying concentrations of PZ at 5, 10, and 15 wt % while keeping the MEA concentration constant at 30 wt %. Measurements were performed at temperatures between 303.15 and 373.15 K and at a N₂ pressure of 400 kPa. Viscosities of MEA-PZ mixtures are tabulated in Table 2, and a graphical representation of changes of viscosity against temperature is shown in Figure 3. It can be seen that the viscosity of the mixtures increases with an increase in PZ

Table 2. Viscosity, η , of Aqueous 30 wt % MEA in Different PZ Concentrations^{a,b}

w_2	5	10	15
T (K)	η (mPa·s)		
303.15	2.76	3.78	4.81
313.15	2.06	2.76	3.43
323.15	1.59	2.08	2.52
333.15	1.25	1.61	1.91
343.15	1.01	1.29	1.49
353.15	0.83	1.05	1.18
363.15	0.69	0.86	0.96
373.15	0.62	0.73	0.79

^aExperiments were performed at a constant MEA mass fraction $w_1 = 0.30$, different PZ mass fractions w_2 , and temperatures T . The operating pressure was maintained using N_2 gas throughout the temperature range ($p = 400$ kPa). ^bStandard uncertainties u are $u(w_1) = 0.01$, $u(w_2) = 0.01$, $u(T) = 0.03$ K, and $u(P) = 0.2$ kPa. The combined standard uncertainty for viscosity measurement $u_c(\eta)$ is 0.10 mPa·s.

**Figure 3.** Viscosity of unloaded MEA-PZ solutions as a function of temperature. Δ , \circ , and \square refer to different MEA-PZ concentrations of 30–5, 30–10, and 30–15 wt %, respectively. The dotted lines refer to regressed values from eq 2.

concentration at all temperatures studied in this work. For example, at a temperature of 303.15 K, the viscosity increases by $\sim 74\%$ from 2.76 to 4.81 mPa·s when the concentration of PZ is varied from 5 to 15 wt %. The effect of varying the PZ concentration on the viscosity of the mixtures is also evident at 343.15 K; the viscosity changes by around 28% from 0.62 to 0.79 mPa·s at this temperature. The increase in viscosity as the concentration of PZ increases can be associated with the increase in hydrogen bonds between PZ, MEA, and water molecules. At constant MEA and PZ concentrations, viscosity decreases as temperature increases. The kinetic energy increases as movements of MEA, PZ, and water molecules increase at higher temperatures. As a result, these molecules can then surpass the intermolecular forces, thus lowering the viscosity of the solution at higher temperatures.

The experimental data were then regressed according to a simple empirical mathematical equation³⁰ as a function of temperature T

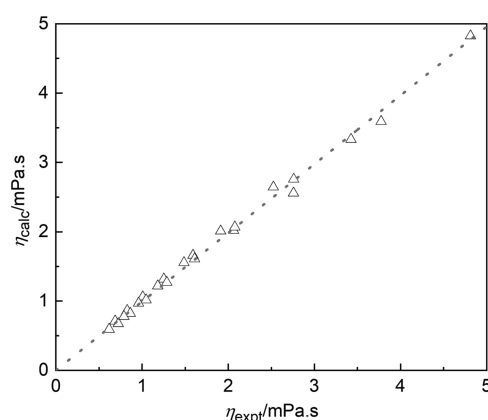
$$\eta = Ae^{B/T} \quad (2)$$

where η and A and B represent the viscosity of the mixture and the regressed parameters respectively. Values of A and B for each set of MEA-PZ solutions studied in this work are tabulated in Table 3. This simple empirical equation (eq 2)

Table 3. Values of Regressed A and B Parameters Based on eq 2

MEA/PZ	A	B
30/5	0.00057	2575.4
30/10	0.00041	2771.2
30/15	0.00022	3026.7

was used by Andrade to represent the viscosity of butyl alcohol and octane over a wide temperature range.³⁰ A slightly modified version of eq 2 was also used by other researchers including in our earlier publication to represent the viscosity of unloaded amine solutions.^{31–33} In this work, we have opted to use the original equation to correlate the viscosity of unloaded MEA-PZ solutions. The viscosity of unloaded MEA-PZ solutions is well represented using eq 2, as evident from the dotted lines in Figure 3 and the parity plot in Figure 4. An

**Figure 4.** Linear relationship between calculated and experimental viscosity values for unloaded MEA-PZ solutions. The dotted line refers to $\eta_{\text{calculated}}$, which is equal to $\eta_{\text{experiment}}$.

AAD value of 0.039 mPa·s was calculated when comparing the experimental and calculated viscosities for unloaded MEA-PZ solutions. This value is smaller than our calculated experimental uncertainty value suggesting that eq 2 can be employed satisfactorily to correlate viscosity data for unloaded MEA-PZ solutions.

3.2. Viscosity of CO_2 -Loaded MEA-PZ Mixtures. In this work, the viscosity of CO_2 -loaded MEA-PZ solutions was measured at three PZ concentrations of 5, 10, and 15 wt % at a constant MEA concentration of 30 wt %. The pressure of the measurement cell was kept at 400 kPa using N_2 gas during the duration of the experiment. Similar to the unloaded experiments, viscosities were measured at temperatures between 303.15 and 373.15 K. Viscosities of CO_2 -loaded 30–5, 30–10, and 30–15 wt % MEA-PZ solutions are presented in Tables 4, 5, and 6, respectively. It is evident that the viscosity of CO_2 -loaded solutions is higher than that of unloaded solutions. For example, the viscosity for the 30–5 MEA-PZ solution increases from 2.76 to 4.37 mPa·s as the CO_2 -loading increases from 0 to 0.55 mol of CO_2 /mol of amines. The increasing concentration of PZ also increases the viscosity of the solution. Higher viscosity for the carbonated MEA-PZ solutions can be explained by the increase in intermolecular forces between molecules of MEA, PZ, water, CO_2 , and associated products from the chemical reactions. Viscosities for the CO_2 -loaded MEA-PZ solutions as a function of temperature are illustrated

Table 4. Viscosity, η , of Aqueous 30–5 wt % MEA-PZ Solutions Loaded with CO₂^{a,b}

α	0.15	0.30	0.40	0.55
T (K)	η (mPa·s)			
303.15	3.14	3.51	3.94	4.37
313.15	2.38	2.67	3.00	3.33
323.15	1.87	2.09	2.37	2.61
333.15	1.50	1.68	1.90	2.10
343.15	1.23	1.38	1.56	1.73
353.15	1.04	1.15	1.31	1.45
363.15	0.89	0.98	1.12	1.23
373.15	0.78	0.85	0.98	1.07

^aExperiments were performed at MEA mass fraction $w_1 = 0.30$, PZ mass fraction $w_2 = 0.05$, different CO₂-loadings α , and temperatures T . Pressure throughout experiment was kept at $p = 400$ kPa using N₂ gas. ^bStandard uncertainties u are $u(w_1) = 0.01$, $u(w_2) = 0.01$, $u(T) = 0.03$ K, and $u(P) = 0.2$ kPa. The combined standard uncertainty for viscosity measurement $u_c(\eta)$ is 0.10 mPa·s.

Table 5. Viscosity, η , of Aqueous 30–10 wt % MEA-PZ Solutions Loaded with CO₂^{a,b}

α	0.15	0.30	0.40	0.55
T (K)	η (mPa·s)			
303.15	4.39	4.93	5.74	6.62
313.15	3.23	3.66	4.25	4.91
323.15	2.46	2.82	3.27	3.78
333.15	1.94	2.24	2.61	3.01
343.15	1.58	1.83	2.12	2.45
353.15	1.31	1.51	1.77	2.04
363.15	1.10	1.27	1.48	1.74
373.15	0.95	1.15	1.30	1.51

^aExperiments were performed at MEA mass fraction $w_1 = 0.30$, PZ mass fraction $w_2 = 0.10$, different CO₂-loadings α , and temperatures T . Pressure throughout the experiment was kept at $p = 400$ kPa using N₂ gas. ^bStandard uncertainties u are $u(w_1) = 0.01$, $u(w_2) = 0.01$, $u(T) = 0.03$ K, and $u(P) = 0.2$ kPa. The combined standard uncertainty for viscosity measurement $u_c(\eta)$ is 0.10 mPa·s.

Table 6. Viscosity, η , of Aqueous 30–15 wt % MEA-PZ Solutions Loaded with CO₂^{a,b}

α	0.13	0.25	0.55
T (K)	η (mPa·s)		
303.15	6.00	7.12	10.88
313.15	4.29	5.10	7.81
323.15	3.19	3.79	5.85
333.15	2.45	2.93	4.53
343.15	1.94	2.32	3.61
353.15	1.58	1.89	2.94
363.15	1.31	1.57	2.46
373.15	1.10	1.33	2.09

^aExperiments were performed at MEA mass fraction $w_1 = 0.30$, PZ mass fraction $w_2 = 0.15$, different CO₂-loadings α , and temperatures T . Pressure throughout the experiment was kept at $p = 400$ kPa using N₂ gas. ^bStandard uncertainties u are $u(w_1) = 0.01$, $u(w_2) = 0.01$, $u(T) = 0.03$ K, and $u(P) = 0.2$ kPa. The combined standard uncertainty for viscosity measurement $u_c(\eta)$ is 0.10 mPa·s.

in Figure 5. Based on the figures, viscosity for the carbonated solutions decreases as temperature increases. This effect on temperature is as expected, as shown for the unloaded MEA-PZ solution earlier.

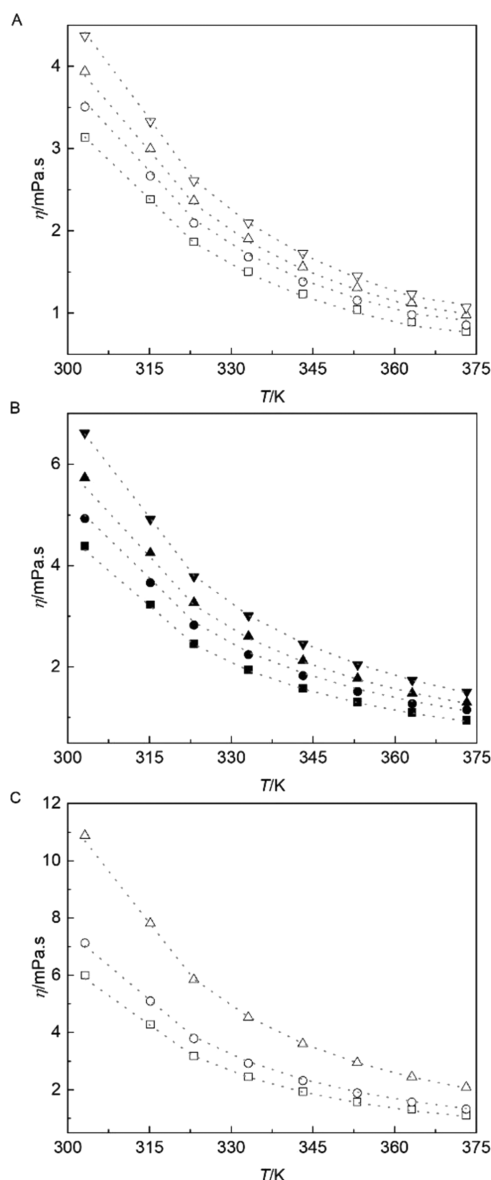


Figure 5. Viscosity against temperature plots of CO₂-loaded solutions at different MEA-PZ concentrations of 30–5 (A), 30–10 (B), and 30–15 (C). Symbols referring to different CO₂-loadings in mol of CO₂/mol of amines: In (A), $\square = 0.13$, $\circ = 0.30$, $\triangle = 0.40$, and $\nabla = 0.55$; in (B), $\blacksquare = 0.15$, $\bullet = 0.30$, $\blacktriangle = 0.40$, and $\blacktriangledown = 0.55$, and in (C), $\square = 0.13$, $\circ = 0.25$, and $\triangle = 0.55$. Values regressed using the modified Setschenow equation are shown as dotted lines.

A method based on the modified Setschenow equation was used to correlate the viscosity of carbonated MEA-PZ solutions. This method was used to originally represent the effect of salting-out in solutions.³⁴ The modified version of the equation was shown to be able to satisfactorily represent several physical data such as density, viscosity, and thermal conductivity with minimal deviations.^{35–37} The modified Setschenow equation is shown in eq 3

$$\ln\left(\frac{\eta}{\eta_r}\right) = (k_{1,0} + k_{1,1} \cdot T) \cdot x_{\text{CO}_2} + (k_{2,0} + k_{2,1} \cdot T) \cdot (x_{\text{CO}_2})^2 \quad (3)$$

where the logarithmic ratio between the viscosity of CO₂-loaded and unloaded MEA-PZ solutions is represented by

$\ln(\eta/\eta_r)$. Parameters α and k_j represent the mole fraction of CO_2 in the solution and the regressed temperature-dependent Setschenow coefficients, respectively. For the MEA-PZ solutions studied in this work, regressed coefficients are presented in Table 7 together with the calculated average

Table 7. Regressed Parameters (eq 3) and Average Absolute Deviation (AAD) Values Based on Setschenow's Equation for CO_2 -Loaded MEA-PZ Solutions

30–5 wt % MEA-PZ		
$k_{1,0} = 0.084$	$k_{1,1} = -18.881$	AAD = 0.03 mPa·s
$k_{2,0} = -0.906$	$k_{2,1} = 280.97$	
30–10 wt % MEA-PZ		
$k_{1,0} = 0.087$	$k_{1,1} = -20.983$	AAD = 0.04 mPa·s
$k_{2,0} = -0.726$	$k_{2,1} = 243.85$	
30–15 wt % MEA-PZ		
$k_{1,0} = 0.072$	$k_{1,1} = -13.076$	AAD = 0.04 mPa·s
$k_{2,0} = -0.522$	$k_{2,1} = 159.39$	

absolute deviations. The calculated AAD values for 30–5, 30–10, and 30–15 wt % MEA-PZ solutions are 0.03, 0.04, and 0.04 mPa·s, respectively. These values are smaller than our calculated experimental uncertainty. A parity plot of the experimental against calculated viscosity data for the CO_2 -loaded solutions is illustrated in Figure 6. The linearity of the plot also suggests that the modified Setschenow equation can satisfactorily be employed to represent the viscosity of CO_2 -loaded MEA-PZ solutions.

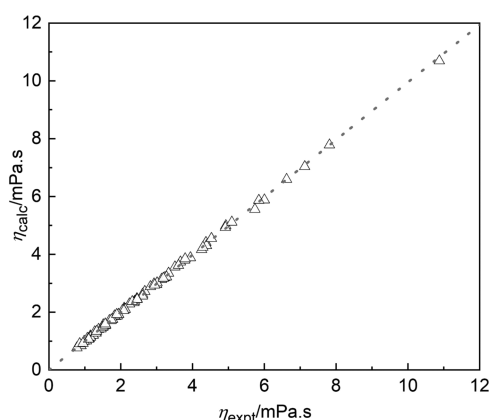


Figure 6. Parity plot showing the linear relationship between calculated and experimental viscosities for the CO_2 -loaded MEA-PZ solutions.

4. CONCLUSIONS

This work presents new viscosity data for unloaded and CO_2 -loaded MEA-PZ solution temperatures between 303.15 and 373.15 K. The concentration of MEA was kept constant at 30 wt %, while the concentration of PZ was varied at 5, 10, and 15 wt %. Experiments were conducted on an Anton Paar Physica MCR 101 rheometer with a double-gap pressure cell XL measuring system under a N_2 atmosphere of 4 bar. The measurement system was calibrated using a viscosity standard oil solution at temperatures relevant for this work. Evaluation of the experimental and reference data suggests that our measurement system works well and would give credible results. For both unloaded and carbonated MEA-PZ solutions,

viscosity is temperature-dependent. The decrease in viscosity at high temperatures could be attributed to the increase in the kinetic energy of the molecules. Increasing the concentration of PZ also increases the viscosity of the solution. The viscosities of unloaded MEA-PZ solutions were correlated using the simple empirical equation used by Andrade. An average absolute deviation of 0.039 mPa·s was calculated, and this value is smaller than our expected experimental uncertainty of 0.1 mPa·s, suggesting that the equation can be used to represent the viscosity of unloaded MEA-PZ solutions. Viscosity for the CO_2 -loaded MEA-PZ solutions was measured at different CO_2 concentrations, and, as expected, increasing the CO_2 concentration increased the viscosity of the solution. The experimental data were then regressed using a modified version of the Setschenow equation. The calculated AAD values between experimental and regressed viscosities are 0.03, 0.04, and 0.04 mPa·s for the 30–5, 30–10, and 30–15 wt % MEA-PZ solutions, respectively. These values are lower than our calculated experimental uncertainty of 0.15 mPa·s. The experimental data collected in this work add to the database of available physiochemical data for amine-based CO_2 -capture solvents and can be used for the development of process and plant design.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.jced.1c00510>.

Viscosity of the standard oil S3 obtained in this work versus values from the supplier (PDF)

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Author Contributions

The manuscript was written through the contributions of all authors. All authors have given approval to the final version of the manuscript.

Notes

The authors declare no competing financial interest.

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