Density and viscosity of aqueous solutions of Methyldiethanolamine (MDEA) + Diethanolamine (DEA) at high pressures.

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Abstract

In this paper, high pressure densities (up to 140 MPa) at six different temperatures (from 293.15 K to 393.15 K) are presented and viscosity measurements (up to 100 MPa) at five isotherms in the range of 313.15 K to 393.15 K are also reported, both for two aqueous solutions of MDEA(1) + DEA(2) with mass fractions of $w_1 = 0.1$; $w_2 = 0.3$ and $w_1 = 0.3$; $w_2 = 0.1$. Densities were measured using a vibrating tube densimeter (Anton Paar DMA HPM) with an expanded uncertainty (k = 2) better than 0.7 kg·m⁻³. Viscosity measurements were obtained using a falling body viscometer whose expanded relative uncertainty (k = 2) ranges from 2.5% for the highest viscosity till 3.2% for the lowest.

1. Introduction

Significant increase in atmospheric CO_2 has resulted in climate change. The world greenhouse gas emissions are expected to rise in the following decades, demanding greater efforts from today's society and industrial sectors in developing innovative technology to reduce CO_2 emissions. Carbon dioxide capture and storage contributes in tackling the issue, becoming a key technology for the mitigation of emissions from the use of fossil fuels in the electrical and industrial sectors. Chemical absorption using aqueous mixtures of amines is among the most common methods used to capture CO₂ from industrial post-combustion processes [1, 2].

Aqueous solutions of N-Methyldiethanolamine (MDEA) and Diethanolamine (DEA) are frequently used for the treatment of gases in post-combustion processes. MDEA is used to accomplish selective removal of H₂S present in CO₂ and DEA is widely used for the removal of CO₂ [3, 4]. The study of binary and tertiary amines mixtures, such as MDEA + DEA, for gas treating processes is of great interest today. Mixed amine systems are suitable for the selective capture of gas and the improvement in the energy regeneration requirements [5]. On this matter, the physical properties such as density and viscosity of aqueous solutions of MDEA + DEA are crucial in the design of gas treatment equipment, CO₂ solubility and reaction rate constants [6].

The main objective of this work is to measure density and viscosity of aqueous solution of MDEA(1) + DEA(2), at the mass concentrations of $w_1 = 0.1$; $w_2 = 0.3$ and $w_1 = 0.3$; $w_2 = 0.1$ for the temperature range (293.15 to 393.15) K and pressures up to 140 MPa or up to 100 MPa for density and viscosity respectively. The measured properties are correlated as a function of temperature and pressure using empirical equations. The behavior of the binary systems (MDEA+ water) and (DEA+ water) was previously studied at mass concentrations from $w_{amine} = 0.1$ to $w_{amine} = 0.4$ [7,8].

2. Experimental

2.1. Materials

Methyldiethanolamine and Diethanolamine samples were purchased from Sigma-Aldrich and their purities were double-checked by gas chromatography (GC), resulting in ≥ 0.99 for MDEA and ≥ 0.995 for DEA. Liquid mixtures were prepared using a RADWAG scale model PS750/C/2 with a standard uncertainty (k = 1) in mass fractions of less than $1 \cdot 10^{-4}$. Features are detailed in Table 1.

Compou		C	Mass fraction	Water	Purification
nd	CAS-N°.	Source	purity ^a	content (ppm) ^b	method
DEA	111-42-2	Sigma-Aldrich	≥0.995	20	None

Table 1. Material description.

MDEA	105-59-9	Sigma-Aldrich	≥0.99	10	None
Water	7732-18-5	Sigma-Aldrich	conductivity $\leq 2 \cdot 1$	$0^{-6} \text{ ohm}^{-1} \cdot \text{cm}^{-1}$	None

^a as stated by the supplier by gas chromatography

^b measured using Mitsubishi Chemical - Coulometric Moisture Meter - CA-200, whose sensitivity and working range are 0.1 µg and 10 µg to 100 mg, respectively.

2.2. Apparatus and procedure

Density measurements were performed with a vibrating tube densimeter (Anton Paar DMA-HPM) using water and vacuum for its calibration as described in previous works [7, 8, 9]. The employed technique measures densities, ranging from (0 a 3000) kg·m⁻³, with a resolution of 10^{-2} kg·m⁻³. Uncertainty calculations were carried out following the procedure described in JCGM100: 2008 [10] as explained in [11], obtaining an expanded uncertainty (k = 2) of 0.7 kg·m⁻³.

Viscosities were determined using a falling body viscometer which is based on the falling time measurement of a body when it falls through a vertical pipe containing the fluid under examination. The viscometer is capable of measuring viscosity in the pressure and temperature ranges of (0.1 to 140) MPa and (253.15 to 523.15) K, respectively. It was designed by TERMOCAL and the Groupe de Haute Pression, Laboratoire des Fluides Complexes of the University of Pau and explained in [12, 13]. The technique is based on Stoke's Law and Newton's Law of motion, previously described in [7, 8, 9], and the viscosity is evaluated taking into account the difference between the density of the body and the liquid density and the falling time.

The measurement procedure and the calibration of the viscometer are described deeply on [7, 8]. Uncertainty calculations were carried out using the procedure described in JCGM100: 2008 [10]. It has been considered a normal distribution with a coverage factor of k=2 (confidence level of 95.45%), obtaining a relative expanded uncertainty varying from 2.5% to 3.2% for the highest and lowest viscosities, respectively. Stabinger SVM3000 viscometer was used in order to check the viscosities obtained from

the falling body viscometer at atmospheric pressure. This commercial apparatus was previously described in [9] being the relative expanded uncertainty (k=2) 2%.

3. Results and discussion

Density measurements of aqueous solutions of Methyldiethanolamine (MDEA) and Diethanolamine (DEA) were performed at pressures from 0.1 MPa to 140 MPa and six temperatures ranging from (293.15 and 393.15) K for amine mass fractions of w = 0.3/0.1and w = 0.1/0.3 for MDEA/DEA mixtures. The experimental results are detailed in Tables 2 and 3, respectively.

Table 2. Experimental densities, ρ , for MDEA(1) + DEA (2) + H ₂ O (3) mixture w_1 =
0.2999 and $w_2 = 0.1001$ at different conditions of temperature, <i>T</i> , and pressure, <i>p</i> . ^{a,b}

			$\rho/\mathrm{kg}\cdot\mathrm{m}^{-3}$			
			<i>T</i> /1	K		
p/MPa	293.15	313.15	333.15	353.15	373.15	393.15
0.1	1039.5	1028.8	1016.3	1002.2	986.4	969.3
0.5	1039.6	1028.9	1016.4	1002.4	986.7	969.6
1	1039.8	1029.1	1016.6	1002.6	986.9	969.9
2	1040.1	1029.5	1017.0	1003.0	987.4	970.4
5	1041.2	1030.6	1018.2	1004.3	988.7	971.9
10	1043.0	1032.5	1020.2	1006.4	991.0	974.4
15	1044.8	1034.3	1022.1	1008.5	993.3	976.8
20	1046.5	1036.0	1024.0	1010.5	995.5	979.3
30	1050.0	1039.7	1027.8	1014.5	999.9	984.0
40	1053.4	1043.2	1031.5	1018.5	1004.2	988.7
50	1056.7	1046.6	1035.0	1022.2	1008.0	992.8
60	1060.1	1050.1	1038.6	1026.1	1012.0	997.2
70	1063.3	1053.4	1042.0	1029.5	1015.8	1001.3
80	1066.4	1056.6	1045.3	1033.1	1019.7	1005.4
90	1069.4	1059.6	1048.6	1036.5	1023.3	1009.2
100	1072.6	1062.9	1051.9	1040.0	1026.9	1013.0
110	1075.5	1065.9	1055.0	1043.3	1030.4	1016.8
120	1078.5	1068.9	1058.2	1046.6	1033.9	1020.4
130	1081.5	1071.9	1061.3	1049.8	1037.3	1024.0

140 1084.4 1075.0 1064.4 1052.9 1040.6 10	27.5
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^a w_i : mass fraction of component i.

^b Standard uncertainties (*k*=1): u(T) = 0.01 K; $u_r(p) = 0.0001$; u(w) = 0.0001 and $u(\rho) = 0.0001$

 $0.35 \text{ kg} \cdot \text{m}^{-3}$

Table 3. Experimental densities, ρ , for MDEA(1) + DEA (2) + H₂O (3) mixture w_1 =

0.1005/ $w_2 = 0.3001$ at different conditions of temperature, *T*, and pressure, *p*.^{a,b}

			$\rho/\mathrm{kg}\cdot\mathrm{m}^{-3}$			
			<i>T</i> /K			
p/MPa	293.15	313.15	333.15	353.15	373.15	393.15
0.1	1045.9	1035.7	1023.9	1010.8	995.9	979.4
0.5	1045.8	1035.9	1024.1	1010.8	996.1	979.7
1	1046.0	1036.0	1024.3	1011.0	996.3	980.1
2	1046.4	1036.4	1024.7	1011.4	996.8	980.5
5	1047.4	1037.5	1025.9	1012.7	998.1	982.0
10	1049.2	1039.3	1027.8	1014.8	1000.3	984.4
15	1050.9	1041.1	1029.7	1016.8	1002.5	986.7
20	1052.7	1042.9	1031.5	1018.8	1004.6	989.0
30	1056.2	1046.4	1035.3	1022.7	1008.7	993.4
40	1059.6	1050.0	1039.1	1026.4	1013.0	997.8
50	1062.9	1053.3	1042.4	1030.2	1016.7	1001.9
60	1066.0	1056.7	1045.8	1033.9	1020.6	1006.1
70	1069.4	1059.9	1049.3	1037.3	1024.3	1010.3
80	1072.4	1063.1	1052.5	1040.8	1028.0	1014.0
90	1075.5	1066.2	1055.7	1044.3	1031.5	1017.9
100	1078.6	1069.4	1059.0	1047.6	1035.0	1021.5
110	1081.6	1072.4	1062.1	1051.0	1038.5	1025.2
120	1084.5	1075.4	1065.3	1054.2	1041.8	1028.7
130	1087.5	1078.4	1068.3	1057.2	1045.1	1032.1
140	1090.2	1081.4	1071.3	1060.3	1048.3	1035.6

^a w_i : mass fraction of component i.

^b Standard uncertainties (*k*=1): u(T) = 0.01 K; $u_r(p) = 0.0001$; u(w) = 0.0001 and $u(\rho) = 0.35$ kg·m⁻³

Despite of the estimated vapor pressure of the mixture is above 0.1 MPa, we found that the boiling point of the mixtures is higher than 393.15 K. As the densimeter is automated, several security loops are implemented. Related with that, pressure stability is achieved minimizing the standard deviation of the last 10 pressure measurements. Then, density measurement starts checking simultaneously pressure and the vibration period of the U tube. Also, the vibrating period is only recorded when the standard deviation of the last 10 measurements are less than $1 \cdot 10^{-3}$ µs from the mean value. That means, if there are bubbles (two phases) inside the U tube, the stability criteria will never be accomplish (pressure or vibration period stability).

Experimental density data show that densities of aqueous mixture MDEA (0.3) + DEA(0.1)in mass fraction are lower than mixture MDEA (0.1)+DEA(0.3) at the same conditions of pressure and temperature, in agreement with the fact that densities of MDEA aqueous solutions ($w_{MDEA} = 0.4$) were lower than DEA aqueous solutions ($w_{DEA} = 0.4$) being the mixtures of both amines in between.

Additionally, density increases with pressure and decreases with temperature in like manner for both mixtures. Density increases from 4.3% to 6.0% when pressure is increased from 0.1 MPa to 140 MPa for mass fraction mixture $w_1 = 0.3/w_2 = 0.1$ and from 4.2% to 5.7% for mixture $w_1 = 0.1/w_2 = 0.3$, being the lowest density increase at 293.15 K and the highest at 393.15 K. On the other hand, MDEA(1)/DEA(2)/H₂O(3) ($w_1 = 0.3/w_2 = 0.1$) mixture shows a decrease in density between 6.7% and 5.2% (at 0.1 MPa and 140 MPa respectively) when temperature is changed from 293.15 K to 393.15 K. In the case of the system MDEA(1)/DEA(2)/H₂O(3) ($w_1 = 0.1/w_2 = 0.3$), densities decrease between 6.4% and 5.0% at the same conditions. In figure 1, the experimental density data as function of pressure at different temperatures are plotted.



Figure 1. Experimental density of the system MDEA (1) + DEA (2) + H₂O (3) at the concentration: (a) $w_1 = 0.1/w_2 = 0.3$; (b) $w_1 = 0.3/w_2 = 0.1$ as function of pressure and the isotherms: (**x**) 293.15 K; (**a**) 313.15 K; (**b**) 333.15 K; (**c**) 353.15 K; (**x**) 373.15 K; (**o**) 393.15 K. Lines represent the calculated values using modified Tammann-Tait equation with the parameters given in Table 4.

The experimental values were correlated using a modified Tammann–Tait equation (Eq. (1)) for each composition:

$$\rho(T, p) = \frac{A_0 + A_1 T + A_2 T^2}{1 - C \ln\left(\frac{B_0 + B_1 T + B_2 T^2 + p}{B_0 + B_1 T + B_2 T^2 + 0.1MPa}\right)}$$
(1)

The fitting results are shown in Table 4, which contains the adjustable parameters and the standard deviation of the adjustment (σ).

MDFA (1) + DFA(2)+ H ₂ O(3)	$w_1 = 0.2999,$	$w_1 = 0.1005,$
$(1) + DDA(2) + H_2O(3)$	$w_2 = 0.1001^a$	$w_2 = 0.3001^a$
$A_0/kg \cdot m^{-3}$	1015.23	1014.9
$A_1/kg \cdot m^{-3} \cdot K^{-1}$	0.666	0.676
$A_2/kg \cdot m^{-3} \cdot K^{-2}$	-1.99·10 ⁻³	-1.95·10 ⁻³
B ₀ /MPa	258.29	272.8
$B_1/MPa \cdot K^{-1}$	1.15	-1.26
$B_2/MPa \cdot K^{-2}$	-3.20·10 ⁻³	-3.25.10-4
С	0.1143	0.116

Table 4. Fitting parameters of Eq. (1) and standard deviations σ for the density correlations

^a w_i : mass fraction of component i.

Experimental densities were compared to literature data which were reported at similar composition, and only data measured by Águila-Hernández et al. [14], MDEA(1)+ DEA (2) +water (3) at T = 313.15 K for mass fractions ($w_1 = 0.3046/w_2 = 0.0999$) and ($w_1 = 0.1004/w_2 = 0.3017$) and, at T = 333.15 K for mass fractions ($w_1 = 0.2998/w_2 = 0.1015$) and ($w_1 = 0.1017/w_2 = 0.2989$) were found at atmospheric pressure. The relative deviations of our measurements and literature values are plotted in Figure 2.



Figure 2. Relative deviations of density measurements (ρ_{exp}) in comparison with literature values (ρ_{lit}): (\Box) Águila-Hernández et al. al.[14]. Dotted lines represent the relative expanded uncertainty of our measurements.

Average absolute relative deviations for the ternary mixtures are 0.05% in agreement with the uncertainty. It is relevant to highlight that no experimental data are available at pressures different to atmospheric pressure. Other authors have measured this ternary system at atmospheric pressure but at different compositions: Rinker et al. [3] measured at $w_{amines} = 0.5$ and T = (293.15 - 373.15) K, Teng et al. [15] measured at $w_{amines} = 0.25 - 0.32$ and T = (298.15 - 353.15) K, Hsu et al. [16] measured $w_{amines} = 0.20 - 0.30$ and T = (303.15 - 353.15) K, Águila-Hernández et al. [14] measured at $w_{amines} = 0.3 - 0.5$ and T = (313.15, 323.15, 333.15) K, Rebolledo-Libreros et al. measured at $w_{amines} = 0.45$ and T = (303.15 - 343.15) K [17] therefore, the comparison is not possible.

Viscosity measurements of MDEA-DEA-water mixtures were carried out at amine weight fractions $w_1=0.1/w_2=0.3$ and $w_1=0.3/w_2=0.1$, p = (0.1 to 100) MPa and five temperatures

T = (313.15, 333.15, 353.15, 373.15, 393.15) K, using the falling body viscometer. The experimental measurements are listed in Tables 5 and 6.

Table 5. Experimental viscosities, η , for MDEA(1) + DEA (2) + H₂O (3) mixture ($w_1 = 0.2999, w_2 = 0.1001$) at different conditions of temperature, *T*, and pressure, *p*. ^{a,b}

η/mPa⋅s					
			<i>T</i> /K		
p/MPa	313.15	333.15	353.15	373.15	393.15
0.1	3.076	1.779	1.172	0.799	0.599
5	3.114	1.798	1.173	0.814	0.607
10	3.136	1.819	1.188	0.826	0.618
15	3.169	1.827	1.200	0.837	0.624
20	3.198	1.851	1.210	0.847	0.632
25	3.224	1.862	1.223	0.855	0.639
30	3.249	1.878	1.234	0.865	0.650
40	3.291	1.910	1.258	0.883	0.679
60	3.395	1.972	1.302	0.916	0.702
80	3.526	2.038	1.341	0.951	0.729
100	3.650	2.107	1.386	0.984	

^a w_i : mass fraction of component i.

^b Standard uncertainties (k=1): u(T) = 0.01 K; $u_r(p) = 0.0001$; u(w) = 0.0001 and $u_r(\eta) = 0.0001$

0.016

Table 6. Experimental viscosities, η , for MDEA(1) + DEA (2) + H₂O (3) mixture ($w_1 = 0.1005, w_2 = 0.3001$) at different conditions of temperature, *T*, and pressure, *p*.^{a,b}

		η/mPa	ı•s		
			<i>T</i> /K		
p/MPa	313.15	333.15	353.15	373.15	393.15
0.1	2.822	1.713	1.138		
5	2.858	1.732	1.155	0.817	0.605
10	2.870	1.741	1.166	0.828	0.613
15	2.894	1.754	1.174	0.830	0.620
20	2.913	1.771	1.182	0.841	0.624

25	2.935	1.785	1.193	0.847	0.630
30	2.960	1.798	1.205	0.860	0.636
40	2.995	1.830	1.222	0.875	0.649
60	3.103	1.881	1.263	0.906	0.673
80	3.187	1.949	1.316	0.935	
100	3.349	2.004	1.361	0.965	

^a w_i : mass fraction of component i.

^b Standard uncertainties (k=1): u(T) = 0.01 K; $u_r(p) = 0.0001$; u(w) = 0.0001 and $u_r(\eta) = 0.0001$

0.016

Experimental results for viscosity measurements of both MDEA+DEA aqueous solutions show similar behavior, with a significant decrease when temperature rises and a slight increase with increasing pressure as can be seen in Figure 3. When temperature is increased from 313.15 K to 393.15 K, the viscosity decreases by 81% for MDEA(0.3)/DEA(0.1) aqueous mixture and by 79% for MDEA(0.1)/DEA(0.3) aqueous solution. On the contrary, the increase of the viscosity when pressure is modified from 0.1 MPa to 100 MPa ranges from 19% to 21% for MDEA(0.3)/DEA(0.1) and between 11% and 19% for MDEA(0.1)/DEA(0.3). On the other hand, viscosities of {MDEA (0.3) + DEA (0.1) + water (0.6)} mixture are higher than {MDEA (0.1) + DEA (0.3) + water (0.6)} mixture at the same conditions and both are lower than the binary mixtures {MDEA (0.4) + water (0.6)} [8] or {DEA (0.4) + water (0.6)} [7].



Figure 3. Experimental viscosity of the system MDEA (1) + DEA (2) + H₂O (3) at the concentration: (a) $w_1 = 0.1/w_2 = 0.3$; (b) $w_1 = 0.3/w_2 = 0.1$ as function of pressure and the isotherms: (**a**) 313.15 K; (**A**) 333.15 K; (**()** 353.15 K; (**x**) 373.15 K; (**()** 393.15 K. Lines represent the calculated values using modified VFT model with the parameters given in Table 7.

Experimental viscosities were correlated using a modified VFT model, Eq (2) successfully used by Harris et al. [18] and in previous work [7, 8, 9].

$$\eta(T,p) = \exp[a + b.p + (c + d.p + e.p^2)/(T - f)]$$
(2)

Data fitting was performed using the method of least squares of the MATLAB software

[19]. Table 7 contains the results of the fitting parameters and the standard deviations.

Table 7. Fitting parameters of Eq. (2) and standard deviations σ for the viscosity correlations

MDEA (1) + DEA (2) + H ₂ O (3)	$w_1 = 0.2999,$	$w_1 = 0.1005,$
	$w_2 = 0.1001^a$	$w_2 = 0.3001^a$
a	-3.567	-4.130
b /MPa ⁻¹	3.36.10-3	2.5063 • 10-3
c /K	706.17	972.01
d /K·MPa ⁻¹	-0.2178	-0.1880
e /K·MPa ⁻²	5.976 • 10-4	-1.132.10-4
f/K	162.64	125.08
σ/mPa·s	0.012	0.0090

^a w_i : mass fraction of component i.

Results of the fitting give a standard deviation of $0.012 \text{ mPa} \cdot \text{s}$ for the aqueous mixture of mass fractions MDEA (0.3)/DEA (0.1) and 0.0090 mPa \cdot \text{s} for MDEA (0.1)/DEA (0.3). These values are lower than the uncertainties of the experimental measurements proving the validity of the fitting equation.

Moreover, in order to verify the reliability of the viscosity data and the technique, a comparison was carried out using a Stabinger SVM 3000 viscometer available in our laboratory which is able to measure at atmospheric pressure. The results of the comparison are given in Table 8 and depicted in Figure 4. As can be seen, the viscosities obtained with both techniques are in a good agreement with the uncertainties.

Table 8. Viscosity comparison between falling body viscometer $(\eta_{FB})^a$ and Stabinger SVM 3000 viscometer $(\eta_{SV})^b$ at p = 0.1 MPa.

w_1 / w_2^{c}	T/K	η _{FB} /mPa∙s	η _{sv} /mPa∙s	$\Delta \eta / \eta_{ m FB}$
		-	-	

MDEA (1) + DEA (2)				
	313.15	3.076	3.172	-3.1
0.2999/0.1001	333.15	1.779	1.822	-2.4
	353.15	1.172	1.163	0.8
MDEA(1) + DAE(2)				
0.1005/0.3001	313.15	2.822	2.798	0.8
	333.15	1.713	1.702	0.6
	353.15	1.138	1.125	1.1

^a Standard uncertainties (k = 1): u(T) = 0.01 K; $u_r(p) = 0.0001$; u(w) = 0.0001; $u_r(\eta) = 0.016$ ^b Standard uncertainties (k = 1): u(T) = 0.02 K; $u_r(p) = 0.005$; u(w) = 0.0001; $u_r(\eta) = 0.01$ ^c w_i : mass fraction of component i.



Figure 4. Relative viscosity deviation between falling body viscometer (η_{FB}) and Stabinger SVM 3000 viscometer (η_{SV}) for the mixtures: (\circ) MDEA/DEA ($w_1 = 0.3 / w_2 = 0.1$); (×) MDEA/DEA ($w_1 = 0.1 / w_2 = 0.3$). Lines represent the relative expanded uncertainty of our measurements.

Finally, we have tried to compare our viscosity measurements with the data available in the literature but there are few measurements at atmospheric pressure for the ternary mixture but at different compositions: Rinker et al. [3] also measured viscosities at $w_{\text{amines}} = 0.5$ and T = (293.15 - 373.15) K, and Rebolledo-Libreros et al. [17] at $w_{\text{amines}} = 0.45$ and T = (303.15 - 343.15) K, therefore, the comparison is not possible.

4. Conclusions

New density and viscosity measurements were performed with mass fractions ($w_1 = 0.30/w_2 = 0.1$) and ($w_1 = 0.1/w_2 = 0.3$) for mixtures of MDEA(1) +DEA(2) + H₂O(3) at five temperatures and pressures up to 140 MPa. Both admixtures show similar behavior in terms of the effect of temperature and pressure in the variation of density and viscosity. However, densities of aqueous solutions rank MDEA(0.4) [8] < MDEA(0.3)+DEA(0.1) < MDEA(0.1)+DEA(0.3) < DEA(0.4) [7], in contrast with viscosities which are lower for the ternary mixtures than the binary mixture amine (0.4). Furthermore, the Tamman-Tait equation and modified VFT model are adequate to correlate the experimental data of densities and viscosities, respectively.

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