



Density and viscosity measurements of (piperazine + water) and (piperazine + 2-dimethylaminoethanol + water) at high pressures



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ABSTRACT

The purpose of this paper is broadening the understanding of amine solution behaviour through the experimental measurements of density (up to 140 MPa) and viscosity (up to 100 MPa) in a temperature range from (293.15 to 393.15) K. The two selected blends are Piperazine (PZ) + Water (10% amine weight concentration) and Piperazine (PZ) + 2-Dimethylaminoethanol (DMAE) + Water (10% and 30% amine weight concentration, respectively). Densities were measured using a vibrating tube densimeter (Anton Paar DMA HPM) with an expanded uncertainty ($k = 2$) less than $0.7 \text{ kg}\cdot\text{m}^{-3}$. Viscosities were obtained using a falling body viscometer which was calibrated with water and dodecane. The viscosity expanded uncertainty ($k = 2$) ranges from 2.5% for the highest viscosity to 3.2% for the lowest one. Experimental data were fitted to modified Tammann-Tait equation for densities and modified VFT model for viscosities, obtaining good results for both equations.

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1. Introduction

In the next decades, the use of fossil fuels will continue leading the world energy consumption, representing more than 81% of the total energy employed. Energy sector accounts two-thirds of total greenhouse gas emissions (GHG) and 80% of CO_2 . These GHG emissions include carbon dioxide, methane, NO_x , SO_x , etc. Electricity, heat generation and transportation are the main contributors of the worldwide CO_2 emissions which depend heavily on coal, oil and natural gas. Because of that, it is necessary to make an extra effort to reduce these emissions and mitigate climate change and energy sector must be included (IEA, 2017) [1].

Nowadays, there are many technological solutions developed to isolate carbon dioxide from exhaust gases. Post-combustion technology is one of the most suitable and mature technological process to remove CO_2 from the main combustion gas streams, with the advantage that, this technology can be easily installed on the pre-existing power plants [2]. The CO_2 separation from flue gas is based on chemical absorption with amine-based solvents. That process has been extensively studied in terms of cost and energy consumption, regarding as the most effective technology for CO_2 capture [3].

The use of piperazine (PZ) at low concentration shows a high potential as a solvent for CO_2 capture when it is mixed with conventional solvents (MDEA, AMP, etc) due of its CO_2 loading capacity and high reaction rate. Theoretically, PZ is able to absorb two moles of CO_2 for each mole of amine and the rate constant of PZ has been found one order higher than conventional alkanolamines such as MEA [4].

Blends of primary and tertiary amines or secondary and tertiary amines offer enhanced absorption capacity, higher absorption rates and reduced the required regeneration energy. As a result of the use of these mixtures and the lack of literature data, the knowledge of thermodynamic and thermophysical properties, as density, viscosity, heat capacity or surface tension, are essential to design the gas treating units.

The main objective of this work is to report new experimental data of densities and viscosities at wide pressure and temperature ranges. Also, this paper extends the information of thermodynamic properties of amines mixtures previously published concerning aqueous solutions of one amine and water: monoethanolamine (MEA) and n-methyldiethanolamine (MDEA) [5]; diethanolamine (DEA), triethanolamine (TEA) and 2-dimethylaminoethanol (DMAE) [6]. This new research is focused on two mixtures, the aqueous solution of PZ ($w_{\text{PZ}} = 10\%$) and, a ternary aqueous mixture made up of PZ + DMAE ($w_{\text{PZ}} = 10\%$; $w_{\text{DMAE}} = 30\%$) Densities were

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measured up to 140 MPa at temperatures between 293.15 K and 393.15 K (in steps of 20 K), whereas viscosities were obtained up to 100 MPa at the same temperature range as densities. Both properties were fitted as a function of temperature and pressure using empirical equations.

2. Experimental

2.1. Materials

The chemicals were purchased from Sigma-Aldrich with the highest purity available. Their characteristics are summarized in Table 1. Purities were specified by the supplier and no further purification was carried out. Liquid mixtures were prepared by weighting in a high precision balance with a standard uncertainty ($k = 1$) in mass fractions less than $1 \cdot 10^{-4}$ (neglecting water content of amines).

2.2. Apparatus and procedure

A vibrating tube densimeter (Anton Paar DMAHPM), that is able to measure density in the range of (0–3000) $\text{kg} \cdot \text{m}^{-3}$ with a resolution of $10^{-2} \text{ kg} \cdot \text{m}^{-3}$, was used for the density measurements. The apparatus is fully automated using the Agilent VEE Pro software for controlling the system and acquiring data. The technique was calibrated with water and vacuum from 283.15 K to 353.15 K. In the case of the 373.15 K and 393.15 K isotherms, the apparatus was calibrated using as reference decane and vacuum, being the procedure previously described in [7]. The uncertainty calculations were performed following “The guide to the expression of uncertainty in measurement JCGM100: 2008” [8] whose procedure was deeply explained in [7], obtaining an expanded uncertainty ($k = 2$) less than $0.7 \text{ kg} \cdot \text{m}^{-3}$.

Viscosities were measured using a falling body viscometer whose operation is based on the fall time measurement of a body through a vertical tube containing the fluid to be measured. Although the cell was developed by Groupe de Haute Pression, Laboratoire des Fluides Complexes of the University of Pau [9], it was implemented in our laboratory and the experimental setup was entirely developed by our research group as it is described in [5,6,10]. This equipment works in wide pressure (0.1–140) MPa and temperature (253.15–523.15) K ranges.

This technique also requires a calibration procedure, described in [11,12], which is based on the use of known viscosity reference fluids under (p, T) conditions in which the viscosity is sought. For this work, the calibration was performed at $p = (0.1 \text{ to } 100) \text{ MPa}$ and $T = (293.15 \text{ to } 393.15) \text{ K}$ using fluids which were extensively studied in the pressure and temperature ranges such as water [13] and dodecane [10,14].

Uncertainty budget was also calculated according to the procedure JCGM 100:2008 [8], and all the details can be found in previous works [6,10]. Uncertainty was evaluated at the limits of the viscosity calibration range for all the studied mixtures: the lowest viscosity was $0.260 \text{ mPa} \cdot \text{s}$ for water at $T = 393.15 \text{ K}$ and $p = 5 \text{ MPa}$, and the highest viscosity was $7.591 \text{ mPa} \cdot \text{s}$ for aqueous DMAE solu-

tion ($w = 0.4$) at $T = 293.15 \text{ K}$ and $p = 60 \text{ MPa}$ [6]. A normal distribution was considered with a coverage factor $k = 2$ (confidence level of 95.45%), obtaining a relative expanded uncertainty which varies from 2.5% to 3.2% for the highest and lowest viscosities, respectively.

Stabinger SVM3000 viscometer was used in order to double-check the viscosities obtained from the falling body viscometer at atmospheric pressure. The principle of measurement is based on the different velocity from a high speed rotating outer tube and the free buoyant inner rotor with a built-in magnet which is immersed in the fluid sample. When the system reaches equilibrium, which means constant rotating velocity at the tube and the floating rotor, then viscosity is calculated from the floating rotor speed. Uncertainty was calculated according to the procedure JCGM 100:2008 [8], obtaining a relative expanded uncertainty ($k = 2$) better than 2%.

3. Results and discussion

Density measurements of one binary system {PZ (1) + H₂O (2)} (with $w_1 = 0.1$ due to low solubility of PZ in water [15]) and, one ternary system {PZ (1) + DMAE (2) + H₂O (3)}, with $w_1 = 0.1$ and $w_2 = 0.3$, were carried out at pressures from 0.1 MPa to 140 MPa and at six temperatures between 293.15 K and 393.15 K. The experimental values are reported in Tables 2 and 3, respectively.

As expected, density increases with pressure and decreases with temperature for both systems. The increase of density due to an increase of pressure (from 0.1 MPa to 140 MPa), is similar in both systems. In the case of the aqueous solution of PZ, this effect varies from 5.1% to 6.3%, increasing with temperature, and quite similar behaviour is observed in the ternary system ranging from 4.4% to 6.8%, at 293.15 K and 393.15 K, respectively.

In addition, density decreases when temperature is raised from 293.15 K to 393.15 K. For the aqueous solution of PZ, that reduction in density ranges from 5.6% to 4.6% at 0.1 MPa and 140 MPa respectively, in the aforementioned temperature range. This behaviour is greater for the ternary system, decreasing density between 8.2% and 6.0% in the same ranges of temperature and pressure and, being the effect higher at lower pressures.

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

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

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

Moreover, the experimental and calculated densities of both systems are plotted as function of pressure for the six measured isotherms in Fig. 1.

Experimental densities were also compared with literature data available at the same conditions (temperature, pressure and composition) but unfortunately, only data at atmospheric pressure are in existence.

Table 1
Material description.

Compound	CAS-NO.	Source	Mass fraction purity ^a	Mass water Content/(%)	Purification method
Piperazine (PZ)	110-85-0	Sigma-Aldrich	≥0.99	<0.1 ^b	None
DMAE	108-01-0	Sigma-Aldrich	≥0.995	<0.1 ^b	None
Water	7732-18-5	Sigma-Aldrich	conductivity ≤ $2 \times 10^{-6} \text{ ohm}^{-1} \cdot \text{cm}^{-1}$		None
Dodecane	112-40-3	Sigma-Aldrich	≥0.99	≤0.01 ^a	None

^a Stated by the supplier by gas chromatography.

^b Measured by Karl Fisher titration (Mitsubishi CA-200).

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