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Thermophysical Properties of Aqueous Piperazine and Aqueous (*N*-Methyldiethanolamine + Piperazine) Solutions at Temperatures (298.15 to 338.15) K

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In the present work, thermophysical properties such as density (ρ), dynamic viscosity (η), refractive index (n_D), and surface tension (σ) of aqueous piperazine (PZ) solutions with mass fractions ($100w = 1.74, 5.16, 10.35$) have been measured from temperatures (298.15 to 338.15) K. All of these properties have also been measured for the aqueous blends of *N*-methyldiethanolamine (MDEA) and piperazine (PZ) with mass fractions $\{100(w_1/w_2) = 32.28/1.74, 32.28/5.16, 32.28/10.35, 48.80/1.74, 48.80/5.16, 48.80/10.35)\}$ for the entire temperature range. The limit of PZ solubility in water was measured by the addition of a known quantity of anhydrous PZ (solid crystals) in 20 mL of distilled water at temperatures (278 to 343) K with a regular interval of 5 K. The experimental values of the studied physical properties have been compared with the literature and also correlated as a function of temperature. The coefficients of thermal expansion (α_p) values have been calculated from their experimental density data using an empirical correlation. A thermogravimetric analyzer (TGA) has been used to explore the thermal stability of the studied solvent systems at a heating rate of 10 °C·min^{-1}.

Introduction

Alkanolamines such as monoethanolamine (MEA), diethanolamine (DEA), 2-amino-2-methyl-1-propanol (AMP), and *N*-methyldiethanolamine (MDEA) are commonly used in the process industries to capture carbon dioxide (CO_2) from natural gas by absorption with chemical reaction in the liquid phase. The use of MDEA for sour gas treatment is preferred because of its high equilibrium loading capacity and low heat of reaction with CO_2, which is associated with low energy requirement for regeneration.[1] Recently, piperazine (PZ) has been used as an effective promoter because of its rapid formation of carbamates with CO_2 when mixed with MDEA, and the rate constant value of PZ has also been found to be one order higher as compared with MEA.[2,3] The successful application of the (MDEA + PZ) system could be due to the relatively high reaction rate of PZ and the low enthalpy of MDEA reaction with CO_2, which leads to high gas absorption rates and low heat of regeneration requirements during the CO_2 removal processes.[4] Therefore, PZ-activated aqueous MDEA solutions appear to be an attractive alternative solvent for the bulk removal of CO_2. The accurate determination of the physical properties of solvents over a wide temperature range is essential for the rational design and optimization of gas treating units. Aqueous solutions of PZ could potentially be high-efficiency solvents for CO_2 removal; however, these systems have not been extensively investigated so far.[5] Moreover, PZ is also used as an activator in BASF-activated MDEA technology.[6] Despite, the widespread applications of aqueous PZ and aqueous (MDEA + PZ) systems, little effort has been made for determining their thermophysical properties. The identification of degradation products of alkanolamines using gas chromatography (GC) and gas chromatography−mass spectrophotometry (GC-MS) has been reported by several researchers for MEA,[7] DEA,[8−10] di-2-propanolamine (DIPA)[11] and MDEA.[12−15] However, most of these works have been related to the analysis of degradation compounds of alkanolamines with the objective of understanding the mechanism and kinetics of degradation.

Table 1. Solubility of Piperazine (PZ) in Water

	PZ solubility				PZ solubility		
T (K)	g·L^{-1}	molarity	mole fraction	T (K)	g·L^{-1}	molarity	mole fraction
278	74.9418	0.87	1.5·10^{-2}	318	665.8622	7.73	0.124
283	105.9522	1.23	2.2·10^{-2}	323	682.2288	7.92	0.127
288	126.6258	1.47	2.5·10^{-2}	328	702.0410	8.15	0.130
293	148.1608	1.72	3.0·10^{-2}	333	714.1006	8.29	0.132
298	167.1116	1.94	3.4·10^{-2}	338	727.0216	8.44	0.134
313	644.3272	7.48	0.121	343	739.0812	8.58	0.136

In this work, we present the experimental density, viscosity, refractive index, and surface tension values of aqueous PZ and aqueous (MDEA + PZ) solutions that have been measured over a wide temperature range at atmospheric pressure and also correlated as a function of temperature. Thermal expansion coefficients (isobaric) for all of the compositions of the considered systems have been calculated from their experimental density data using an empirical correlation. Thermal stability of aqueous PZ and aqueous (MDEA + PZ) solutions has also been determined using a thermogravimetric analyzer (TGA).

Experimental Section

Chemicals. PZ with stated purity of 99.9 % (GC, area %), (catalogue no. 807325, CAS no. 110-85-0, batch no. S4663425) and MDEA with purity of 99.5 % (GC, area %), (catalogue no. 805851, CAS no. 105-59-9, batch no. S4605451) were supplied by Merck and were used without further purification. The aqueous PZ and aqueous (MDEA + PZ) solutions were prepared

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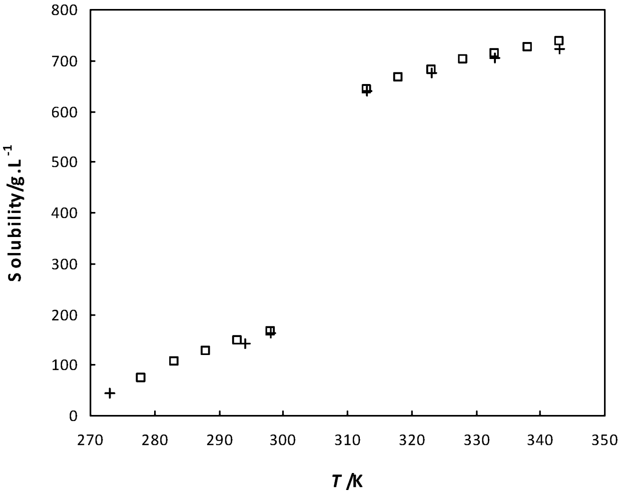

Figure 1. Solubility of anhydrous solid crystals of PZ in distilled water at temperatures (273 to 343) K: □, this work; +, Bishnoi et al.[16] (2002).

gravimetrically with a precision of ± 0.0001 g using an analytical balance (Mettler Toledo model AS120S). The possible uncertainty in calculating mole fraction and mass fraction of aqueous solutions was estimated to be around ± 0.001 and ± 0.0001, respectively. All measurements for each sample were performed in duplicate, and the average values are reported.

Piperazine Solubility. We investigated the extent of the solubility limit for PZ in water by adding a known quantity of anhydrous PZ (solid crystals) to a round-bottomed flask containing 20 mL of distilled water at temperatures (278 to 343) K. The aqueous PZ samples were placed in a thermostatted water bath (Polyscience, 28 L B/S/C) with temperature uniformity of ± 0.2 K and were operated at a shaking speed of 160 cycles$\cdot$min^{-1} to ensure proper mixing of PZ in water. The sample solutions were allowed to settle until the saturation was attained, which indicated the solubility limit of PZ in water. The total PZ contents were determined after taking out a small quantity of the solution from the upper portion of the flask and titrated with 2N HCl to an end point pH of 2, as mentioned by Bishnoi et al.[16]

Density. A digital vibrating glass U-tube densimeter (DMA5000, Anton-Paar) was used for density measurements with an accuracy of ± 0.003 kg$\cdot$m^{-3} at temperatures (298.15 to 338.15) K. The temperature accuracy of ± 0.02 K (traceable to the ITS-90 protocol) was maintained with a built-in platinum resistance thermometer. The experimental uncertainty for temperature and density measurements is estimated to be ± 0.03 K and ± 0.005 kg$\cdot$m^{-3}, respectively. The densimeter was calibrated using Millipore quality water samples before and after all density measurements, as followed in our previous article.[17]

Viscosity, Refractive Index, and Thermal Decomposition. The kinematic viscosity of the samples was measured at temperatures (303.15 to 333.15) K using calibrated Ubbelohde viscometers of appropriate sizes that were immersed in a thermostatic bath (Tamson, TVB445). The calibrated Ubbelohde viscometers were further verified with the standard solvents of known viscosity (provided by the equipment supplier) and found to be in good agreement. The bath temperature was regulated with a Pt-100 temperature probe with an accuracy of ± 0.02 K. The dynamic viscosity of the samples was calculated using kinematic viscosity and corresponding density values with an uncertainty of ± 0.3 %. The experimental viscosity at corresponding temperatures was measured with an uncertainty of ± 0.04 mPa$\cdot$s and ± 0.04 K, respectively. The refractive index (RI) of the samples was determined using a programmable digital refractometer (Atago, RX-5000 alpha) with a measuring accuracy of ± 4$\cdot$10^{-5}. The refractometer was calibrated using Millipore quality water before each series of measurements and checked for ethanol (absolute, Fischer scientific) with purity 99.4 % and known refractive index of 1.36242. The refractive indices were measured at (303.15 to 333.15) K with a temperature control accuracy of ± 0.05 K. The experimental uncertainty for refractive index measurement at corresponding temperatures was found to be ± 3$\cdot$10^{-5} and ± 0.05 K, respectively. Thermal stability of the samples was investigated using a TGA (Perkin-Elmer, Pyris V-3.81) at a heating rate of 10 K$\cdot$min^{-1} under a N_2 atmosphere supplied at a rate of 20 mL$\cdot$min^{-1}. Further details of the calibration and methodology of using equipments can be found in our recently published article.[18]

Surface Tension. The surface tension of samples was measured using an interfacial tension meter, IFT 700 (VINCI Technologies), with a precision of ± 0.03 mN$\cdot$m^{-1} and temperature accuracy of 0.2 K. The apparatus was calibrated

Table 2. Literature Review for the Determination of Physical Properties of Aqueous PZ and Aqueous (MDEA + PZ) Solutions at Atmospheric Pressure

year	composition	T (K)	ref	apparatus
		Density and Viscosity		
2006	aqueous PZ: ($100w$ = 1.74, 3.44, 5.16, 6.88)	(298 to 333)	5	Gay−Lussac pycnometer and Ostwald viscometer
2006	aqueous MDEA (1) + PZ (2): ($100w_1/w_2$ = 27/3, 24/6, 21/9, 18/12)	(288 to 333)	21	Gay−Lussac pycnometer Ostwald viscometer
2008	aqueous MDEA (1) + PZ (2): ($100w_1$ = 11.83, 23.39, 34.74, 45.84) and ($100w_2$ = 2.2, 4.3, 6.5, 8.6)	(293.15 to 333.15)	4	DMA 58 (Anton Paar) Ubbelohde viscometer
		Surface Tension		
2006	aqueous MDEA (1) + PZ (2): ($100w_1/w_2$ = 27/3, 24/6, 21/9, 18/12)	(293 to 323)	22	GBX tensiometer (ILMS-4, using Du Nouy ring method)
		Density, Viscosity, and Surface Tension		
2005	aqueous PZ:			
	($100w$ = 5.37, 8.65, 12.78, 14.45) for density and viscosity	(293.15 to 323.15)	20	DMA 58 (Anton Paar) and Ubbelohde viscometer (PSL)
	($100w$ = 4.3, 8.6, 12.9) for surface tension	(293.15, 313.15)		Krüss (K9) tensiometer
		Thermal Degradation		
1988	aqueous MDEA: ($100w$ = 50)	453	13	autoclave, gas chromatograph (GC), and gas chromatograph−mass spectrophotometer (GC-MS).
1997	aqueous MDEA: ($100w$ = 20 to 50)	(373 to 473)	14	autoclave and GC-MS.
2006	aqueous MDEA: ($100w$ = 30)	473	15	gas chromatograph (GC)

Table 3. Physical Properties of Aqueous PZ and Aqueous MDEA (1) + PZ (2)

T	aqueous PZ: (100*w*)			MDEA (1) + PZ (2) + water: 100(w_1/w_2)					
K	1.74	5.16	10.35	32.28/1.74	32.28/5.16	32.28/10.35	48.80/1.74	48.80/5.16	48.80/10.35
Density ($kg\cdot m^{-3}$)									
298.15	998.287	999.360	1001.285	1024.783	1025.327	1026.382	1036.829	1037.365	1038.328
303.15	996.868	997.913	999.758	1022.845	1023.638	1024.216	1034.515	1035.038	1035.960
308.15	995.233	996.252	998.022	1020.478	1021.327	1022.135	1032.086	1032.595	1033.480
313.15	993.399	994.396	996.095	1018.342	1018.923	1019.582	1029.570	1030.069	1030.918
318.15	991.382	992.359	993.991	1015.946	1016.452	1017.222	1026.868	1027.357	1028.172
323.15	989.188	990.147	991.722	1013.410	1013.896	1014.680	1024.008	1024.487	1025.274
328.15	986.804	987.769	989.288	1010.647	1011.128	1011.882	1021.093	1021.576	1022.335
333.15	984.320	985.241	986.131	1007.861	1008.326	1008.763	1018.037	1018.498	1018.942
338.15	981.667	982.506	983.952	1004.814	1005.210	1005.946	1014.921	1015.341	1016.063
100AAD	0.004	0.001	0.001	0.003	0.004	0.005	0.0005	0.004	0.001
Viscosity ($mPa\cdot s$)									
303.15	0.86	0.97	1.28	3.62	4.11	5.62	6.93	8.63	11.74
313.15	0.69	0.76	1.02	2.48	2.97	4.04	5.02	6.23	8.41
318.15	0.61	0.69	0.92	2.2	2.67	3.57	4.38	5.55	7.4
323.15	0.57	0.62	0.81	1.96	2.38	2.94	3.62	4.56	6.28
333.15	0.47	0.51	0.59	1.32	1.72	2.21	2.73	3.42	4.62
100AAD	0.28	1.24	0.50	1.46	0.3	0.09	0.05	0.01	0.02
Refractive Index (nD)									
303.15	1.33449	1.34019	1.34847	1.36487	1.37197	1.37818	1.37118	1.37752	1.38286
313.15	1.33366	1.33914	1.34797	1.35989	1.36458	1.37242	1.36578	1.37285	1.37764
318.15	1.33324	1.33831	1.34752	1.35718	1.36031	1.36734	1.36227	1.36942	1.37418
323.15	1.33287	1.33793	1.34699	1.35309	1.35702	1.36166	1.35984	1.36664	1.37124
333.15	1.33241	1.33638	1.34632	1.34624	1.35166	1.35506	1.35502	1.36139	1.36743
100AAD	0.007	0.63	0.10	0.63	0.35	0.03	1.04	1.07	0.62
Surface Tension ($mN\cdot m^{-1}$)									
303.1	71.48	70.24	69.10	60.32	59.70	58.86	57.52	56.72	56.23
313.2	69.34	68.51	67.92	58.86	58.32	57.90	56.13	55.50	55.22
318.2	68.76	67.35	66.75	58.14	57.60	56.81	55.36	54.82	54.38
323.1	67.84	66.70	65.37	57.63	57.06	56.23	54.62	54.14	53.67
333.3	66.21	65.26	64.14	56.44	55.85	55.32	53.72	53.05	52.49
100AAD	0.004	0.003	0.002	0.0003	0.0001	0.002	0.0003	0.0002	0.0008

Table 4. Coefficients of Thermal Expansion of Aqueous PZ and Aqueous MDEA (1) + PZ (2) Solutions using Equation *3*

	$\alpha_p\cdot 10^4/K^{-1}$								
T	aqueous PZ: (100*w*)			MDEA (1) + PZ (2) + water: 100(w_1/w_2)					
K	1.74	5.16	10.35	32.28/1.74	32.28/5.16	32.28/10.35	48.80/1.74	48.80/5.16	48.80/10.35
298.15	4.18	4.22	4.39	4.86	4.94	4.98	5.29	5.31	5.39
303.15	4.19	4.23	4.40	4.87	4.95	4.99	5.30	5.32	5.41
308.15	4.19	4.24	4.41	4.88	4.97	5.01	5.32	5.34	5.42
313.15	4.20	4.25	4.42	4.89	4.98	5.02	5.33	5.35	5.44
318.15	4.21	4.26	4.43	4.91	4.99	5.03	5.35	5.37	5.45
323.15	4.22	4.27	4.44	4.92	5.00	5.05	5.36	5.38	5.46
328.15	4.23	4.28	4.45	4.93	5.02	5.06	5.37	5.40	5.48
333.15	4.24	4.28	4.46	4.94	5.03	5.07	5.39	5.41	5.49
338.15	4.25	4.29	4.47	4.95	5.04	5.08	5.40	5.42	5.51

after each set of measurements with standard solutions provided by the supplier and also verified for ethanol samples (absolute, Fischer scientific) with purity 99.4 % and known surface tension value of 21.55 $mN\cdot m^{-1}$ at 303.15 K. The experimental uncertainty for surface tension measurement at corresponding temperatures was found to be ± 0.04 $mN\cdot m^{-1}$ and ± 0.3 K, respectively. A pendent drop method was used for surface tension measurements, which allows the formation of a regular drop suspended in a thermostated chamber. A camera connected to the computer focuses and records the shape of the liquid drop to derive the interfacial properties through fast calculating system software.

Results and Discussion

The solubility of PZ in distilled water was measured at temperatures of (278 to 343) K, and the experimental data is presented in Table 1. The limited PZ solubility in water may be explained by the formation of piperazine hexahydrate crystals ($C_4N_2H_{10}\cdot 6H_2O$), which melt at 317 K.[19] This fact is reinforced by the appearance of a discontinuity in PZ solubility at around 313 K, as shown in Figure 1. The solubility values of PZ are found to be in good agreement with the reported work of Bishnoi et al.[16] However, in this work, the solubility values of PZ in water appeared to be slightly higher for the whole temperature range, as compared with the reported data of Bishnoi et al.[16] Moreover, in the present work, PZ solubility at 293 K, that is, 148.16 $g\cdot L^{-1}$, is also found to be in close proximate with the reported value of Merck (PZ, MSDS), that is, 150 $g\cdot L^{-1}$.

A summary of the literature review[4,5,20−22] for the determination of physical properties such as density, viscosity, and surface tension of aqueous PZ and aqueous (MDEA + PZ) solutions and identification of degradation products of aqueous MDEA solutions[13−15] is presented in Table 2, which reveals that the experimental data for these systems are widely scattered and limited to specific temperatures and compositions. Moreover, the experimental data for the refractive index, thermal decomposition, and the values for the coef-

Table 5. Thermal Decomposition Values of Aqueous PZ and Aqueous MDEA (1) + PZ (2)

	aqueous PZ: (100w)			MDEA (1) + PZ (2) + water: 100(w_1/w_2)					
	1.74	5.16	10.35	32.28/1.74	32.28/5.16	32.28/10.35	48.80/1.74	48.80/5.16	48.80/10.35
T_{start} (°C)	32	32	30	30	44	47	29	31	31
T_{onset} (°C)	79	81	81	48 and 137	76 and 162	85 and 163	67 and 163	69 and 167	74 and 171
T_{f} (°C)	161	196	134	204	235	223	227	226	246

ficients of thermal expansions of the studied systems are not available in open literature. Therefore, new experimental measurements for thermophysical properties of industrially important solvents, MDEA and (MDEA + PZ), have been made in the present work. The experimental values for the density, viscosity, refractive index, and surface tension of aqueous PZ and aqueous (MDEA + PZ) solutions as a function of temperature as well as amine concentration have been presented in Table 3 along with their percent average absolute deviations (100AAD) using eq 1.

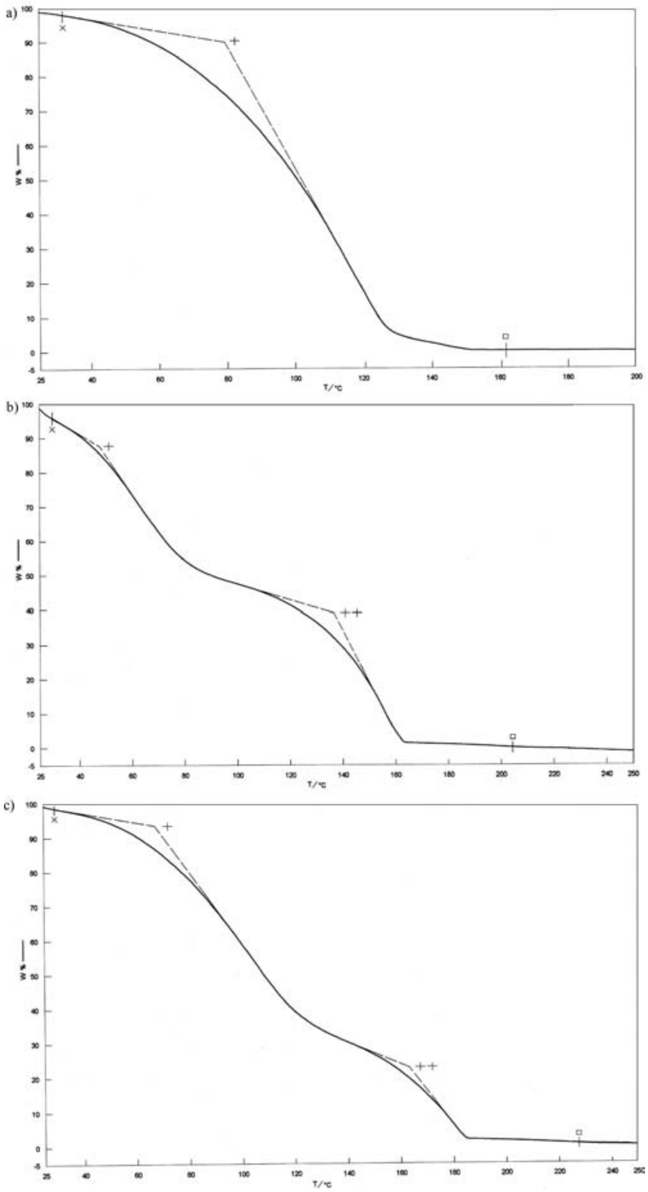

Figure 2. Plots for thermal decomposition as weight percent loss (W %) versus temperature (T/°C) of (a) aqueous PZ: 100w = 1.74; ×, T_{start} (start decomposition temperature) = 31.927 °C at 97.9387 (W %); +, T_{onset} (intersection of baseline weight and the tangent of weight vs temperature curve (---)) = 79.402 °C at 89.8039 (W %); □, T_{f} (final decomposition temperature) = 160.681 °C at 0.0997 (W %); and MDEA (1) + PZ(2) + water: (b) 100(w_1/w_2) = 32.28/1.74; ×, T_{start} = 29.737 °C at 95.9760 (W %); +, 1st T_{onset} = 47.887 °C at 87.4634 (W %); ++, 2nd T_{onset} = 136.552 °C at 38.9671 (W %); □, T_{f} = 204.211 °C at 0.0953 (W %); and (c) 100(w_1/w_2) = 48.80/1.74; ×, T_{start} = 29.211 °C at 98.5573 (W %); +, 1st T_{onset} = 67.121 °C at 93.3463 (W %); ++, 2nd T_{onset} = 163.136 °C at 22.7240 (W %); □, T_{f} =2 27.368 °C at 0.5125 (W %) at heating rate of 10 °C·min^{-1}.

$$100\text{AAD} = \frac{1}{N_{\text{p}}}\sum_{i=1}^{N_{\text{p}}} 100|(x_{\text{calcd}} - x_{\text{exptl}})/x_{\text{exptl}}|_i \qquad (1)$$

where, x_{calcd} and x_{exptl} are calculated and experimental physical properties studied here, respectively, and N_{p} is the number of data points. After careful analysis of the experimental data, the density, refractive index, and surface tension values were correlated using eq 2, whereas the viscosity data were correlated using eq 3.

$$Z = A_0 + A_1T \qquad (2)$$

$$\ln(\eta) = A_0 + A_1/T \qquad (3)$$

where, Z = (density, refractive index, and surface tension), η = dynamic viscosity, and A_0 and A_1 are the fitting parameters, which were estimated by a least-squares method and presented in Table S1 of the Supporting Information. As expected, an overall decrease in all measured system properties was observed with an increase in temperature. The values for standard deviations (SD) of density and viscosity of aqueous PZ (100w = 1.74, 5.16) measured in this work and that of Samanta and Bandyopadhyay[5] were calculated after correlating the experimental data using eqs 2 and 3, respectively, and are also presented in Table S1 of the Supporting Information. The coefficients of thermal expansion values for all compositions of the studied systems were calculated using eq 4 on the basis of the experimental density data with an estimated uncertainty of ± $0.03 \cdot 10^{-4}$/K^{-1}.

$$\begin{aligned} \alpha_{\text{p}} &= -1/\rho(\partial\rho/\partial T)_P \\ &= -A_1/(A_{\text{o}} + A_1T) \end{aligned} \qquad (4)$$

where α_{p} is the coefficient of thermal expansion, ρ is the density, T is the temperature, and A_i is the fitting parameter for experimental densities (eq 2) taken from Table S1 of the Supporting Information. It is obvious from Table 4 that the change in coefficients of thermal expansion values is not significant and the variation of volume expansion of the studied system could be considered to be independent of temperature. Thermal stability of aqueous PZ and aqueous (MDEA + PZ) solutions was evaluated using a TGA. The values for thermal decomposition of aqueous PZ and aqueous (MDEA + PZ) solutions are shown in Table 5 in terms of T_{start} (start temperature for decomposition), T_{onset} (intersection of baseline weight and the tangent of weight vs temperature curve), and T_f (final decomposition temperature). In Figure 2, the thermograms (percent weight loss with increase in temperature) for the decomposition of aqueous PZ, that is, $100w = 1.74$ (a), and aqueous MDEA (1) + PZ (2), $100(w_1/w_2) = 32.28/1.74$ (b) and $100(w_1/w_2) = 48.80/1.74$ (c), are shown. Thermograms for all other compositions of alkanolamine systems are presented in Figure S1a−f of the Supporting Information. The one-step degradation of aqueous PZ solutions shows the strong binding interactions between PZ and water molecules. The onset temperature and two-step degradation of aqueous (MDEA + PZ) solutions indicate that PZ molecules were initially decomposed, followed by the degradation of MDEA molecules in the second step.

Conclusions

Density, viscosity, refractive index, and surface tension values for aqueous PZ and aqueous (MDEA + PZ) solutions have been measured over a wide temperature range and also correlated. A decrease in all measured physical properties was observed with an increase in temperature. The coefficients of thermal expansion values reveal that the volume expansions of the studied systems do not depend on temperature. Thermal stability of aqueous PZ and aqueous (MDEA + PZ) solutions has also been investigated using thermogravimetric analysis.

Supporting Information Available:

Fitting parameters of eqs 2 and 3, and thermograms for aqueous PZ with mass fractions ($100w = 5.16, 10.35$) and aqueous blends of MDEA/PZ with mass fractions $\{100(w_1/w_2) = 32.28/5.16, 32.28/10.35, 48.80/5.16, 48.80/10.35\}$. This material is available free of charge via the Internet at http://pubs.acs.org/.

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