

Apparent molar properties of aqueous protic ionic liquid solutions at $T = (293.15 \text{ to } 328.15) \text{ K}$

Gyanendra Sharma · Vickramjeet Singh · Ramesh L. Gardas

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Abstract Apparent molar volumes and apparent molar isentropic compressibilities of newly synthesized ammonium-based protic ionic liquid (PIL), namely diethylethanolammonium propionate, at $T = (293.15 \text{ to } 328.15) \text{ K}$ and at atmospheric pressure were obtained from experimentally measured density and speed of sound data. Redlich-Mayer type of equation was used to calculate infinite dilution apparent molar volumes, V_2^∞ , infinite dilution apparent molar isentropic compressibilities, $K_{s,2}^\infty$, and corresponding empirical parameters (S_v , B_v , S_k , and B_k). Positive magnitude of limiting volumetric slope S_v versus $m^{1/2}$ for protic ionic liquid (PIL) + water binary solutions, suggests that PIL-water interactions are weak. Further, the temperature dependence of V_2^∞ values has also been evaluated.

Keywords Protic ionic liquid · Diethylethanolammonium propionate · Apparent molar volumes · Apparent molar isentropic compressibilities

List of abbreviations

IL Ionic liquid
PIL Protic ionic liquid
DEEAP Diethylethanolammonium propionate

List of symbols

m Molality
 ρ Density
 u Speed of sound

V_ϕ Apparent molar volume
 $K_{s,\phi}$ Apparent molar isentropic compressibility
 V_ϕ^∞ Partial molar volume at infinite dilution
 K_ϕ^∞ Partial molar isentropic compressibility at infinite dilution
 κ_s Isentropic compressibility
 E_ϕ^∞ Apparent molar expansibility at infinite dilution

Introduction

Ionic liquids (ILs) are organic molten salts having melting point below 100°C . ILs are of two broad classes: protic ionic liquid (PIL) and aprotic ionic liquid (APIL); PILs are formed by equimolar (stoichiometric) combination of Bronsted acid with Bronsted base. PILs have an ability to form hydrogen bonds and as a result, they interact strongly with various polar solvents. The importance of IL as a better solvent, in comparison to volatile organic solvents, has been explored extensively [1–8]. ILs or binary solutions of IL with various solvents have been used for a number of applications. Moreover, thermodynamic properties characterizing the strength of interactions between IL and solvent are of paramount importance for applications in the field of refrigeration, design of process engineering, electrochemical applications, chemical processes, synthesis, etc. [9–13].

Simple ammonium-based PILs are easy to synthesized and have relatively low toxicity and low cost. In the last few years, a growing interest in ammonium-based PILs has been observed owing to their excellent properties [13–16]. Ammonium-based ILs are employed in a number of applications [17–21], e.g., dissolution of polymers, separation processes, organic synthesis, etc. Thermodynamic properties of ammonium-based IL and its mixture with various solvents

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G. Sharma · V. Singh · R. L. Gardas (✉)
Department of Chemistry, Indian Institute of Technology Madras,
Chennai 600036, India
e-mail: gardas@iitm.ac.in
URL: <http://www.iitm.ac.in/info/fac/gardas>

have been studied to provide reliable thermodynamic data which may be used for applications in industrial and chemical processes [22–25]. Volumetric and ultrasonic properties of ILs with solvents are important for elucidation of solute-solute and solute-solvent interactions occurring in these solutions [2, 7, 9, 10, 23, 24, 26–29]. Thermodynamic properties of binary solutions of ammonium-based ILs with water are scarce [9, 12, 13, 27, 30–33]. So, in this work, we report volumetric and acoustical properties of newly synthesized ammonium-based IL (diethylethanolammonium propionate [DEEAP]) in aqueous solutions at different temperatures, $T = (293.15 \text{ to } 328.15) \text{ K}$, and at atmospheric pressure. The results have been interpreted in terms of various molecular interactions occurring in binary solutions.

Experimental

Materials

Commercial available reagents were used as such without further purifications. *N,N*-diethylethanol amine, propanoic acid, and methanol were purchased from Sigma-Aldrich (>99 % purity).

Synthesis and characterization of diethylethanolammonium propionate [DEEAP]

[DEEAP] was synthesized by exothermic neutralization of propanoic acid by base (*N,N*-diethylethanol amine). To 10 ml of methanol, *N,N*-diethylethanol amine was added in round-bottom flask, and this mixture was kept in ice bath for a few minutes. Further, propanoic acid was added slowly and drop wise (by using dropping funnel) to the above reaction mixture. Addition of acid was completed in 2 h at 5 °C and then reaction mixture was stirred continuously for 24 h at room temperature. The excess amount of starting material and solvent were removed by putting the reaction mixture into rotavapor at $\approx 50 \text{ }^\circ\text{C}$ for 4 h. The resultant product (PIL) was dried at room temperature under high vacuum for 36 h, in order to remove moisture and excess of amine, and further IL was kept in nitrogen atmosphere. The synthesis of [DEEAP] is shown in Scheme 1.

The newly synthesized [DEEAP] has been characterized by various spectroscopic techniques and found to be having purity of >98 %.

^1H NMR analysis (using CDCl_3 as solvent and TMS as the internal standard) of [DEEAP], $\delta = 4.725 \text{ ppm}$ (broad, 2H, OH

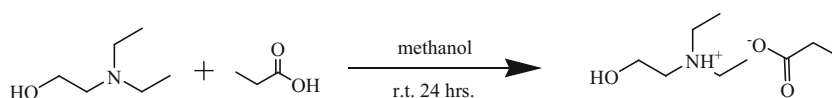
and NH^+), $\delta = 3.863 \text{ ppm}$ (t, 2H), $\delta = 3.049 \text{ ppm}$ (q, 3H), $\delta = 2.972 \text{ ppm}$ (t, 2H), $\delta = 2.287 \text{ ppm}$ (q, 2H), $\delta = 1.250 \text{ ppm}$ (t, 6H) and $\delta = 1.119 \text{ ppm}$ (t, 3H). JASKO FT/IR-4100 spectrometer having maximum resolution of 0.9 cm^{-1} and signal to noise ratio of 22,000:1 was used to record IR spectra. The IR broad band appeared in range of $3400\text{--}2800 \text{ cm}^{-1}$ corresponds to the characteristic ammonium peak, $\nu(\text{N-H})$ and $\nu(\text{O-H})$ stretching vibration. The broad band centered around 1600 cm^{-1} corresponds to the characteristic carbonyl, $\nu(\text{C=O})$ stretching and $\delta(\text{N-H})$ plane bending, vibrations. High-Resolution Mass Spectra recorded for m/z $[\text{M-CH}_3\text{CH}_2\text{COO}]^+$ value of $\text{C}_6\text{H}_{16}\text{NO}$ using Micromass Q-Tof micro (ESI mass spectrometer) and found to be 118.1228 (Supporting Information Fig. S1), which is in agreement with calculated value 118.1232. Thermograms for pure [DEEAP] is recorded using the TGA instrument (TA instruments Hi-Res. TGA Q500) with a weighing precision of $\pm 0.01 \%$ at a heating rate of $10 \text{ }^\circ\text{C min}^{-1}$ under nitrogen atmosphere with open aluminum pan. Thermograms of pure [DEEAP] shows (Supporting Information Figure S2) that the degradation of PIL starts at around $\approx 65 \text{ }^\circ\text{C}$ and it completely decompose at $\approx 110 \text{ }^\circ\text{C}$.

The water content of the synthesized PIL was measured by using Analab Karl Fischer Titrator (Micro AquaCal100) and is as follows: $[\text{DEEAP}] \approx 7000 \text{ ppm}$. The water content in IL has been taken in account for the molality correction of binary solutions.

Apparatus and procedure

All the solutions were made by weight basis in air-tight glass vials by using analytical balance with a precision of $\pm 0.01 \text{ mg}$ (Sartorius CPA225D). Millipore quality freshly degassed water was used for making binary solutions. The density, ρ and speed of sound, u of binary solutions were measured simultaneously by using vibrating-tube digital density and speed of sound analyzer (Anton Paar, DSA 5000 M) at $T = (293.15 \text{ to } 328.15) \text{ K}$ and at atmospheric pressure. The instrument is equipped with a density cell and a sound velocity cell, both the cells are temperature-controlled by a built-in Peltier thermostat (PT-100) having an accuracy of $\pm 0.01 \text{ K}$. The instrument was calibrated by double distilled, degassed and deionized water and with dry air at atmospheric pressure. The uncertainty in the measurement of density is $\pm 7 \times 10^{-3} \text{ kg m}^{-3}$ and speed of sound is $\pm 0.5 \text{ m s}^{-1}$. The water used for binary solutions and for the calibration of the instrument has a specific conductance less than $1.29 \times 10^{-6} \text{ } \Omega^{-1} \text{ cm}^{-1}$. The measured

Scheme 1 Synthesis of DEEAP



density and speed of sound of water at different temperatures agree well with the literature values [34–42] and are given Table 1.

Table 1 Comparison of experimentally measured density, ρ , and speed of sound, u , of pure water at $T = (293.15$ to $328.15)$ K with literature values

Component	T/K	$\rho \cdot 10^{-3} \text{ kg} \cdot \text{m}^{-3}$		$u/\text{m} \cdot \text{s}^{-1}$	
		Expt.	Literature	Expt.	Literature
Water	293.15	0.998206	0.9982062 ^a	1482.84	1482.96 ^c
			0.99820 ^b		
			0.9982041 ^c		
			0.998203 ^d		
	298.15	0.997049	0.997047 ^a	1496.85	1497.12 ^c
			0.9970449 ^c		1496.65 ^f
			0.997043 ^d		1496.69 ^g
			0.99704 ^e		
	303.15	0.995660	0.9956488 ^a	1509.25	1509.59 ^c
			0.99560 ^b		1509.01 ^f
			0.9956473 ^c		
			0.995645 ^d		
308.15	0.994045	0.9940326 ^a	1519.82	1520.17 ^c	
		0.9940319 ^c		1519.81 ^{f,g}	
		0.994029 ^d			
		0.99403 ^e			
313.15	0.992228	0.992152 ^a	1528.89	1529.25 ^c	
		0.99220 ^b		1528.83 ^f	
		0.992158 ^c			
		0.992212 ^d			
318.15	0.990223	0.9902132 ^c	1536.56	1536.78 ^c	
		0.990208 ^d		1536.36 ^f	
		0.99017 ^e		1536.41 ^g	
323.15	0.988030	0.9880363 ^c	1542.73	1542.66 ^c	
		0.99801 ^e		1542.53 ^f	
		0.988284 ^h			
328.15	0.985690	0.9856952 ^c	1547.61	1547.32 ^f	
		0.985710 ⁱ			

The standard uncertainties are $u(\rho)=7.00 \times 10^{-3} \text{ kg} \cdot \text{m}^{-3}$, $u(u)=0.5 \text{ m} \cdot \text{s}^{-1}$, $u(T)=0.01 \text{ K}$

^a Ref [34]

^b Ref [35]

^c Ref [36]

^d Ref [37]

^e Ref [38]

^f Ref [39]

^g Ref [40]

^h Ref [41]

ⁱ Ref [42]

Results and discussion

Measured density and speed of sound values for binary solutions at different temperatures, $T=(293.15, 298.15, 303.15, 308.15, 313.15, 318.15, 323.15$ and $328.15)$ K were used to calculate the apparent molar volumes, V_ϕ (Table 2) and apparent molar isentropic compressibilities, $K_{s,\phi}$ (Table 3) of DEEA P with the help of following equations:

$$V_\phi = [M/\rho] - [(\rho - \rho_o)/(m \cdot \rho \cdot \rho_o)], \quad (1)$$

$$K_{s,\phi} = (\kappa_s \cdot M/\rho) - [(\kappa_s^o \cdot \rho - \kappa_s \cdot \rho_o)/(m \cdot \rho \cdot \rho_o)] \quad (2)$$

where M and m are, respectively, the molar mass and molality of the ILs, ρ and ρ_o are the densities of binary solution and solvent (water), respectively, κ_s and κ_s^o are the isentropic compressibilities of solution and solvent, respectively. The isentropic compressibilities κ_s have been calculated by using Newton-Laplace's equation as: $\kappa_s = 1/(u^2 \cdot \rho)$. The densities, ρ , of binary (IL+water) solutions increase with concentration of IL and decrease with temperature (Fig. 1), whereas speeds of sound, u , increase with IL concentration as well as with temperature (Fig. 2). The combined uncertainties in the determined V_ϕ and $K_{s,\phi}$ values range from 0.154 to $0.04 \times 10^6 \text{ m}^3 \cdot \text{mol}^{-1}$ and 0.40 to $0.10 \times 10^{-15} \text{ m}^3 \cdot \text{mol}^{-1} \cdot \text{Pa}^{-1}$, for low- and high-concentration range of IL, respectively.

The infinite dilution apparent molar volumes, V_ϕ^∞ , of PIL have been evaluated by using Redlich-Mayer type of equation as:

$$V_\phi = V_\phi^\infty + S_v \cdot m^{1/2} + B_v \cdot m \quad (3)$$

where S_v and B_v are the empirical parameters, evaluated by the least square analysis of the above equation. The V_ϕ^∞ , S_v , and B_v values for binary solutions (PIL+water) are given in supporting information Table S1. At infinite dilution, each ion is surrounded only by the solvent molecules; therefore, V_ϕ^∞ is unaffected by ion-ion interaction and it is a measure only of the ion-solvent interactions. The V_ϕ^∞ values increase with increase in temperature of binary solutions. Similar trend in V_ϕ^∞ values with temperature has also been reported [32] for binary system of imidazolium-based ionic liquid (1-alkyl-3-methylimidazolium bromide) with water. The magnitude of S_v parameter for aqueous solutions of IL is positive at all studied temperatures, which suggests that the ion-ion interactions are stronger than ion-water interactions. Further, magnitude of B_v parameter for (IL+water) binary solutions are negative at all temperatures studied.

Table 2 The densities ρ and apparent molar volumes V_ϕ of ammonium-based protic ionic liquid in water at $T = (293.15 \text{ to } 328.15) \text{ K}$ and at atmospheric pressure

m mol·kg ⁻¹	$\rho \cdot 10^{-3}$ kg·m ⁻³	$V_\phi \cdot 10^6$ m ³ ·mol ⁻¹	$\rho \cdot 10^{-3}$ kg·m ⁻³	$V_\phi \cdot 10^6$ m ³ ·mol ⁻¹	$\rho \cdot 10^{-3}$ kg·m ⁻³	$V_\phi \cdot 10^6$ m ³ ·mol ⁻¹
DEEAP+water						
$T/K=293.15$		$T/K=298.15$		$T/K=303.15$		
0.09506	0.999851	173.96	0.998662	174.49	0.997235	175.11
0.13496	1.000338	175.39	0.999134	175.93	0.997689	176.58
0.21523	1.001781	174.32	1.000542	174.90	0.999067	175.54
0.30310	1.003235	174.09	1.001952	174.70	1.000441	175.35
0.40667	1.004923	173.87	1.003579	174.54	1.002027	175.19
0.55082	1.007143	173.77	1.005724	174.48	1.004107	175.15
$T/K=308.15$		$T/K=313.15$		$T/K=318.15$		
0.09506	0.995593	175.66	0.993716	176.60	0.991649	177.60
0.13496	0.996033	177.15	0.994124	178.16	0.992030	179.18
0.21523	0.997384	176.12	0.995423	177.12	0.993277	178.14
0.30310	0.998721	175.98	0.996704	176.97	0.994502	177.99
0.40667	1.000267	175.83	0.998184	176.83	0.995919	177.85
0.55082	1.002287	175.82	1.000114	176.82	0.997765	177.84
$T/K=323.15$		$T/K=328.15$				
0.09506	0.989396	178.62	0.987007	179.55		
0.13496	0.989749	180.23	0.987334	181.21		
0.21523	0.990944	179.19	0.988485	180.17		
0.30310	0.992115	179.04	0.989610	180.02		
0.40667	0.993470	178.90	0.990911	179.88		
0.55082	0.995232	178.89	0.992602	179.87		

The standard uncertainties are $u(m)=6.90 \times 10^{-6} \text{ mol} \cdot \text{kg}^{-1}$, $u(\rho)=7.00 \times 10^{-3} \text{ kg} \cdot \text{m}^{-3}$, $u(T)=0.01 \text{ K}$, combined uncertainty, $U(V_\phi)=(0.13 \text{ to } 0.06) \times 10^6 \text{ m}^3 \cdot \text{mol}^{-1}$ for low- and high-concentration range of IL, respectively, ($k=0.95$, level of confidence)

The variation of V_ϕ^∞ data with temperature can be expressed by using the following equation:

$$V_\phi^\infty = a + bT + cT^2 \quad (4)$$

where a , b , and c are the empirical parameters, and T is temperature of the solution. Infinite dilution apparent molar expansibility, $E_\phi^\infty = (\partial V_\phi^\infty / \partial T)_P$ values (supporting information Table S1) for binary mixtures (IL+water) are positive and increase with the increase in temperature. Similarly, positive magnitude of E_ϕ^∞ values (supporting information Table S2) for binary solutions of (1-butyl-3-methylimidazolium bromide+water) at different temperatures has also been reported [26].

Apparent molar isentropic compressibility, K_ϕ^∞ at infinite dilution for binary system (IL+water) has also been calculated by using Redlich-Mayer type equation as:

$$K_{s,\phi} = K_\phi^\infty + S_k \cdot m^{1/2} + B_k \cdot m \quad (5)$$

where S_k and B_k are the empirical parameters (supporting information Table S3). The magnitude of K_ϕ^∞ values increase

with temperature and is found to be both negative and positive. The magnitude of K_ϕ^∞ values becomes positive after 298.15 K. The K_ϕ^∞ values are resultant of positive and negative effects, where the solvent intrinsic compressibility is positive effect and the solute intrinsic penetration of the solvent molecules is the negative effect [25]. The positive K_ϕ^∞ values of ILs in water indicate the dominance of intrinsic compressibility over the penetration effect. The positive magnitude of K_ϕ^∞ values for {methyltrioctylammonium bis(trifluoromethylsulfonyl) imide+ethylacetate/ethanol} systems have also been reported [43], and this also suggest the dominance of solvent intrinsic compressibility over the penetration effect.

Conclusions

Volumetric and acoustical properties of protic ionic liquid [DEEAP] synthesized by neutralization method of bronsted acid and base were studied in water at $T = (293.15 \text{ to } 328.15) \text{ K}$ and at atmospheric pressure. The infinite dilution values (V_ϕ^∞ and K_ϕ^∞), slopes (S_v and S_k), and empirical

Table 3 The speeds of sound u and apparent molar isentropic compressibilities $K_{s,\phi}$ of ammonium based protic ionic liquid in water at $T = (293.15$ to $328.15)$ K and at atmospheric pressure

m $\text{mol}\cdot\text{kg}^{-1}$	u $\text{m}\cdot\text{s}^{-1}$	$K_{s,\phi}\cdot 10^{14}$ $\text{m}^3\cdot\text{mol}^{-1}\cdot\text{Pa}^{-1}$	u $\text{m}\cdot\text{s}^{-1}$	$K_{s,\phi}\cdot 10^{14}$ $\text{m}^3\cdot\text{mol}^{-1}\cdot\text{Pa}^{-1}$	u $\text{m}\cdot\text{s}^{-1}$	$K_{s,\phi}\cdot 10^{14}$ $\text{m}^3\cdot\text{mol}^{-1}\cdot\text{Pa}^{-1}$
DEEAP+Water						
$T/\text{K}=293.15$			$T/\text{K}=298.15$		$T/\text{K}=303.15$	
0.09506	1498.40	-0.02	1511.47	0.72	1523.03	1.63
0.13496	1505.67	-0.13	1518.30	0.62	1529.35	1.52
0.21523	1517.23	0.40	1529.18	1.09	1539.63	1.83
0.30310	1531.05	0.41	1542.20	1.09	1551.87	1.79
0.40667	1546.87	0.45	1557.15	1.11	1565.79	1.79
0.55082	1567.10	0.64	1576.11	1.28	1583.61	1.92
0.09506	1498.40	-0.02	1511.47	0.72	1523.03	1.63
$T/\text{K}=308.15$			$T/\text{K}=313.15$		$T/\text{K}=318.15$	
0.09506	1532.40	1.99	1541.17	2.92	1548.04	3.01
0.13496	1538.22	2.00	1546.81	2.76	1553.33	3.00
0.21523	1548.54	2.15	1555.98	2.95	1562.41	3.15
0.30310	1560.06	2.17	1566.82	2.89	1572.59	3.17
0.40667	1573.11	2.21	1579.09	2.88	1584.08	3.21
0.55082	1589.84	2.36	1594.76	2.97	1597.57	3.46
0.09506	1532.40	1.99	1541.17	2.92	1548.04	3.01
$T/\text{K}=323.15$			$T/\text{K}=328.15$			
0.09506	1553.62	3.98	1556.02	4.78		
0.13496	1558.62	3.83	1560.56	4.60		
0.21523	1566.80	3.93	1568.00	4.63		
0.30310	1576.38	3.86	1576.23	4.62		
0.40667	1587.24	3.83	1585.75	4.60		
0.55082	1601.02	3.90	1598.01	4.65		

The standard uncertainties are $u(m)=6.90\times 10^{-6}\text{ mol}\cdot\text{kg}^{-1}$, $u(u)=0.5\text{ m}\cdot\text{s}^{-1}$, $u(T)=0.01\text{ K}$, combined uncertainty, $U(K_{s,\phi})=(0.60\text{ to }0.20)\times 10^{-15}\cdot\text{m}^3\cdot\text{mol}^{-1}\cdot\text{Pa}^{-1}$ for low and high concentration range of IL, respectively ($k=0.95$, level of confidence)

parameters (B_v and B_k) were determined by using Redlich-Mayer-type equation. Positive magnitude of S_v parameter for

studied binary solutions at all temperatures strengthens the view that ion-solvent interactions are weaker than ion-ion interactions. Positive K_ϕ^∞ values of (PILs+water) binary

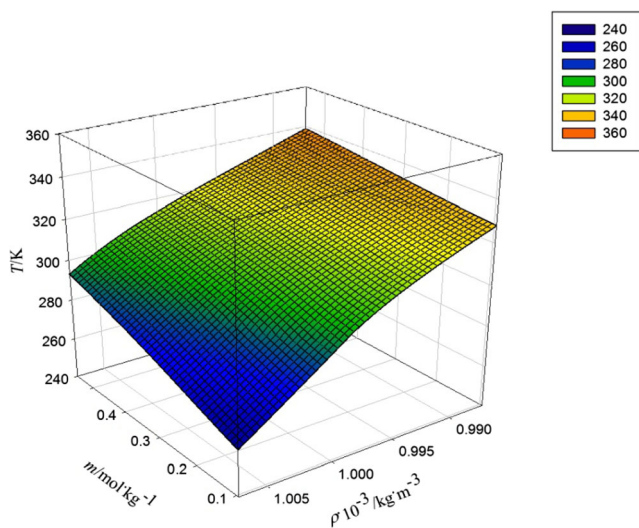


Fig. 1 Density ρ versus molality m of (DEEAP+water) binary solutions at different temperatures

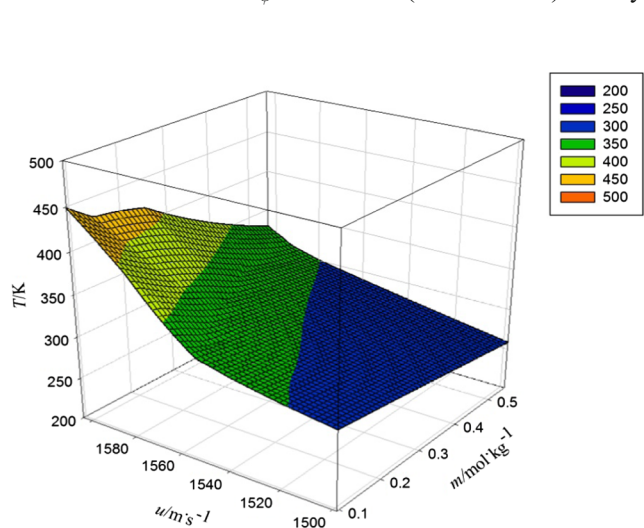


Fig. 2 Speeds of sound u versus molality m of (DEEAP+water) binary solutions at different temperatures

solutions suggest the dominance of the intrinsic compressibility over the penetration effect.

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