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Physical Properties of Aqueous Solutions of Ionic Liquid, 1-Propyl-3-methylimidazolium Methyl Sulfate, at $T = (298.15 \text{ to } 328.15)$ K

Hemayat Shekaari* and Elnaz Armanfar

Department of Chemistry, University of Mohaghegh Ardabili, Ardabil, Iran

Osmotic coefficients, electrical conductances, densities, viscosities, and refractive indices of ionic liquid, 1-propyl-3-methylimidazolium methyl sulfate [PrMIm]MSO_4, with water have been measured at $T = (298.15$ to $328.15)$ K and atmospheric pressure. Experimental osmotic coefficients have been measured using the vapor pressure osmometry (VPO) method and correlated with Pitzer-ion interaction and modified NRTL (MNRTL) electrolyte models. Molar conductances of this system have been correlated with the low concentration chemical model (lcCM) of conductance equation, and then ion association constants have been obtained at different temperatures. Ion association constant values increase with increasing temperature. The apparent molar volumes at infinite dilution are estimated by the Redlich−Mayer and Pitzer equations. Viscosity *B* coefficients have been calculated from Jones−Dole equation. It was found that in the investigated range of concentrations the values of viscosity *B* coefficients are positive. The *B* coefficient decreases with an increase in temperature. The results are explained in terms of hydrophobic hydration of ionic liquid.

Introduction

In the past decade, ionic liquids as innovative solvents have been used in many academic and industrial research areas because of unique properties such as wide liquid range, negligible vapor pressure, good thermal stability, excellent solvent power for organic and inorganic and polymeric compounds, suitable viscosity, and nonflammability.[1–3] Ionic liquids have been used in catalysis, chemical reactions, separations, electrochemistry, and nanoscience investigations. The most commonly studied ionic liquids are based on the imidazolium cation and fluorinated anions such as BF_4^-, PF_6^-, NTf^-, and so on. However, the hydrolytic instability of such anions has become obvious, and decomposition of these fluorinated anions leads to the formation of highly toxic and corrosive HF. The use of these ionic liquids will be limited.[4] Therefore, the synthesis and application of halogen-free ionic liquids is necessary. Several types of these systems have been described, e.g., alkyl sulfunates, organoborates, and alkyl sulfate anions. The most important properties of alkyl sulfate based ionic liquids are easy synthesis, using cheap alkylating agents, solvent-free synthesis, high reaction rate, excellent purity, large scale synthesis, safe and nontoxic chemical, low viscosities, and low melting points.[5] Physical properties of ionic liquids depend on structural factory, ion-association, and ion-solvation effects. The potential advantages of ionic liquids depend on studies of thermophysical properties such as activity coefficients, vapor−liquid equilibria, liquid−liquid phase equilibria, gas solubility, surface tensions, viscosities, electrical conductivities, and so on. Therefore, various research groups started with the systematic measurements of thermophysical properties. There have been numerous publications about physical properties of ionic liquids. Density, isobaric molar heat capacity, and excess molar enthalpy were experimentally determined at atmospheric pressure for a set of binary systems, 1-ethyl-3-methylimidazolium ethyl sulfate and 1-butyl-3-methylimidazolium methyl sulfate.[6] Measurements have been reported for osmotic coefficients of EmimEtSO4 (1-ethyl-3-methylimidazolium ethyl sulfate) and EmpyEtSO4 (1-ethyl-3-methylpyridinium ethyl sulfate) with water and ethanol.[7] Liquid−liquid equilibrium data for 1-ethyl-3-methylimidazolium ethyl sulfate + thiophene + 2,2,4-trimethylpentane and 1-ethyl-3-methylimidazolium sulfate + thiophene + toluene have been determined at 298.15 K.[8] Activity coefficients at infinite dilution were measured for eight alkanols in the ionic liquid 1-butyl-3-methylimidazolium methyl sulfate using the dilutor technique.[9] The ionic liquid 1-ethyl-3-methylimidazolium ethyl sulfate has been tested as a solvent for the separation of thiophene from aliphatic hydrocarbons. Liquid−liquid equilibrium data have been determined for ternary systems containing this type of ionic liquid, thiophene, and alkenes.[10] The surface tensions were measured of a series of alcoholic solutions of closely related ionic liquids, 1-methyl-3-methylimidazolium methyl sulfate, 1-butyl-3-methylimidazolium methyl sulfate, and 1-butyl-3-methylimidazolium octyl sulfate.[11] Viscosity and interfacial tension of 1-ethyl-3-methylimidazolium ethyl sulfate with water and ethanol were studied as a function of composition.[12] Solubility measurements of methane and of xenon in the ionic liquid 1-butyl-3-methylimidazolium methyl sulfate were performed with a high-pressure view-cell technique based on the synthetic method.[13] Dynamic viscosities, densities, speeds of sound, and refractive indices of 1-ethyl-3-methylimidazolium ethyl sulfate with methanol, 1-propanol, and 2-propanol have been measured over the whole composition range.[14] The solubility of alkali bromide MBr (M is Na or K) in 1-ethyl-3-methylimidazolium ethyl sulfate has been measured in the temperature range of (293.15 to 343.15) K.[15] The liquid−liquid equilibria of several binary systems containing 1,3-dimethylimidazolium methyl sulfate and dialkyl carbonates, ketones, or acetates were studied, and similar studies were made on alkyl sulfate containing ionic liquids.[16–18]

This paper is a continuation of our systematic measurement of thermophysical properties of binary mixtures containing an

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* Corresponding author. E-mail: hemayatt@yahoo.com. Tel.: +98-451-5514702. Fax: +98-451-5514701.

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ionic liquid with molecular solvents.[19–23] In this context, the procedure to synthesize an ionic liquid is described, and the osmotic coefficient, electrical conductance, density, viscosity, and refractive index data of aqueous ionic liquid 1-propyl-3-methylimidazolium methyl sulfate [PrMIm]MSO$_4$ solutions have been measured at T = (298.15 to 328.15) K. Experimental osmotic coefficients have been measured using vapor pressure osmometry (VPO). The results have been correlated with Pitzer-ion interaction[25] and one local composition model, the modified NRTL (MNRTL) electrolyte model.[26] These models have been successfully used to correlate the osmotic coefficients in electrolyte solutions. Molar conductances of this system have been correlated with the low concentration chemical model (lcCM) for conductance, and ion association constants have been obtained at different temperatures.[27] Apparent molar volumes of studied mixtures were calculated at temperatures T = (298.15 to 318.15) K from density data. The corresponding limiting apparent molar quantities were found by extrapolation to infinite dilution using the Redlich−Mayer equation. The viscosity B coefficients have been calculated using the Jones−Dole equation. Experimental results of refractive indices for the systems studied are reported and have been fitted by a polynomial expansion equation.

Experimental Section

Chemicals. Imidazole (>99 % w), sodium ethoxide (>99 % w), 1-bromopropane (>99 % w), toluene (>99 % w), absolute ethanol (>99.5 % w), ethyl acetate (>99 % w), and dimethyl sulfate (>99 % w) were obtained from Merck. These reagents were used without any further purification. The doubly distilled water with a specific conductivity less than 1.3 μS·cm^{-1} was used for preparation of solution. All the solutions were prepared in fresh doubly distilled water by mass and converted to the molarity scale when required using density data at different temperatures.

Synthesis of Ionic Liquid. The synthesis of 1-propyl-3-methylimidazolium methyl sulfate [PrMIm]MSO$_4$ occurred in two steps.[28] First, the imidazole diluted in absolute ethanol was deprotonized using sodium ethoxide and then alkylated with 1-bromopropane to make 1-propylimidazole in a 2 L two-necked round-bottom flask fitted a reflux condenser. This mixture was then stirred at room temperature for one day. During this time a yellow liquid formed (1-propylimidazole) and then purified as standard methods given in the literature. Second, dimethyl sulfate was added dropwise to a solution of equal molar amounts of 1-propylimidazole in toluene, cooled in an ice-bath under nitrogen at a rate to maintain the reaction temperature below 313.15 K. The reaction mixture was stirred at room temperature for (1 to 4) h depending on the amount of starting materials. The upper organic phase of the resulting mixture was decanted, and the lower ionic liquid phase was washed with ethyl acetate. The ionic liquid was dried by heating and stirring under high vacuum, and the ionic liquid was kept in bottles under an inert gas. The ionic liquid was characterized using ^{1}H NMR (Bucker Av-300) and IR (Buck Scientific) spectra to confirm the absence of any major impurities, and the densities of pure water and pure ionic liquid at different temperatures are given in Table 1.

Osmotic Coefficient Measurements. Osmotic coefficient measurements were made with a vapor pressure osmometer (VPO) K-7000 (Knauer). With this method, vapor pressure was determined indirectly by using the voltage of thermistors caused by changes in temperatures. This type of osmometer, its principle of operation, and its application to the estimation of osmotic coefficients of ionic liquid solutions have been described in an earlier study.[22] For aqueous solutions, the instrument was calibrated using NaCl reference solutions with known osmotic coefficients in the appropriate concentration range, yielding a function that correlates the voltage readings to the corresponding concentrations of the reference solutions and their osmotic coefficients, and then in the same conditions, the readings were measured for the studied ionic liquid solutions.

Table 1. Density d and Refractive Index n_D of Water and Pure Ionic Liquid [PrMIm]MSO$_4$ as a Function of Temperature

	d/g·cm^{-3}		n_D*	
T/K	H$_2$O	[PrMIm]MSO$_4$	H$_2$O	[PrMIm]MSO$_4$
298.15	0.99704	1.5114	1.3325	1.4761
308.15	0.99403	1.5075	1.3313	1.4725
318.15	0.99021	1.4930	1.3298	1.4701
328.15	0.98569	1.4851	1.3279	1.4664

Electrical Conductance Measurements. Conductance measurements were carried out with a digital conductivity meter (Metrohm 712) with a sensitivity of 0.1 % and a dipping type conductivity cell with platinized electrodes with a cell constant of 0.824 cm^{-1} under nitrogen atmosphere and at a frequency of 1 MHz. The cell constant was determined with aqueous KCl solution. About 50 cm^3 of pure water was placed in the conductivity cell, and the cell was closed. Known massing pure ionic liquids were added with a syringe to the cell containing the solvent. Circulating water from a thermostatically regulated bath was around the sample holder with a double wall to maintain the temperature with a precision of 0.02 K. The conductivity cell was purged with nitrogen during each run.[23]

Density Measurements. Density measurements of these solutions were measured using an Anton Paar vibrating tube digital densimeter (model DMA-4500M) with precision of ± 5·10^{-5} g·cm^{-3} at different temperatures from (298.15 to 328.15) K. Density is extremely sensitive to temperature, so temperature was kept constant within ± 1.0·10^{-3} K using the built-in Peltier device. The instrument was calibrated with ultra pure water and dry air. From the densities, apparent molar volumes V_ϕ of [PrMIm]MSO$_4$ in water were calculated. Details of the measurement method were given elsewhere.[20]

Viscosity Measurements. Kinematic viscosities of [PrMIm]MSO$_4$ + H$_2$O binary mixtures were determined using an Ostwald-type viscometer which had a flow time of about 200 s for water at 298.15 K. Gravity fall is the principle on which this viscometer is based. The viscometer was placed in a JULABO ED (Germany) thermostat with temperature stability ± 0.03 K. The viscometer was calibrated using the efflux time of double distilled water at T = (298.15 to 328.15) K. Flow time measurement was performed by a digital stopwatch with a resolution of 0.01 s. The estimated uncertainty of the experimental viscosity was ± 0.02 mPa·s.[29] The dynamic viscosity η was calculated by using the expression

$$\frac{\eta}{d} = ct - \frac{k}{t} \tag{1}$$

where c and k are the viscometer constants; t is the flow time; and d is the density of solution.

Refractive Index Measurements. The refractive indices were measured using an automatic digital refractometer, ATAGO-DRA1, with an uncertainty of ± 1·10^{-4} and an uncertainty in temperature of ± 0.03 K that calibrated with doubly distilled water and tetrachloroethylene (supplied by the company) before each series of measurements according to manual instruction.

Table 2. Experimental Osmotic Coefficient Data ϕ of [PrMIm]MSO$_4$ + H$_2$O as a Function of Ionic Liquid Molality, *m*, at Different Temperatures

	ϕ		
m/mol·kg^{-1}	T = 308.15 K	T = 318.15 K	T = 328.15 K
0.0122	0.914	1.004	1.015
0.0381	0.889	0.983	0.984
0.0508	0.861	0.960	0.977
0.0732	0.845	0.924	0.910
0.0893	0.826	0.924	0.881
0.1374	0.789	0.848	0.848
0.2999	0.761	0.812	0.823
0.4396	0.747	0.790	0.810
0.5689	0.733	0.768	0.803
0.7404	0.727	0.755	0.793
0.8644	0.724	0.753	0.788
1.0960	0.721	0.752	0.793
1.3471	0.731	0.755	0.836

Temperature was controlled with a JULABO ED (Germany) circulating bath. A procedure called "zero setting" was always performed before the actual measurements of the sample's refractive index, to ensure that the refractometer is working properly.[23,24]

Results and Discussion

The experimental osmotic coefficients of the studied aqueous solutions of ionic liquid with molality m in equilibrium were obtained according to

$$\phi = (\nu_r m_r \phi_r)/(\nu m) \tag{2}$$

where ϕ_r, m_r, and ν_r are the osmotic coefficients and the molality of the reference solution showing the same instrument reading as the ionic liquid solution, which means the vapor pressure (and therefore the solvent activity) is equal in both solutions and stoichiometric numbers of reference electrolytes (NaCl), which are set equal to 2, respectively.

Table 2 gives the experimental osmotic coefficients of the aqueous solutions of [PrMIm]MSO$_4$ at T = (308.15 to 328.15) K. Figure 1 shows the molal concentration dependence of the experimental osmotic coefficients for aqueous solutions of [PrMIm]MSO$_4$ at experimental temperatures T = (308.15 to 328.15) K. The osmotic coefficients increase only slightly when the temperature increases from (308.15 to 328.15) K. The behavior of the osmotic coefficients can be interpreted in terms of ion-solvation and ion-association effects. On examination of Figure 1, it is noted that the osmotic coefficients in the dilute region decrease with increasing concentration of ionic liquids showing the presence of nonideality due to ion−ion interactions.

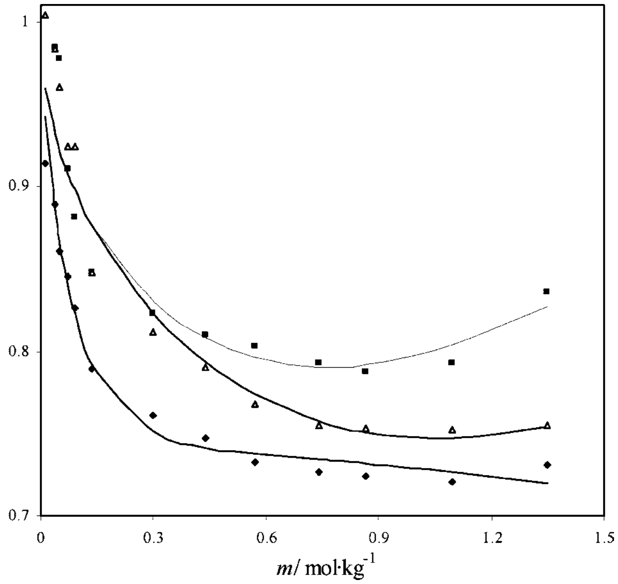

Figure 1. Osmotic coefficient data, ϕ, of [PrMIm]MSO$_4$ as a function of ionic liquid molality, m: lines, MNRTL model; points, experimental data; ■, T = 328.15 K; △, T = 318.15 K; and ◆, T = 308.15 K.

Several models are available in the literature for the correlation of osmotic coefficients as a function of molalities. The three-parameter Pitzer-ion interaction model has been successfully used for aqueous electrolyte solutions and has the following form for a binary 1:1 electrolyte solution[25]

$$\phi - 1 = f_\phi + mB_\phi + m^2 C_\phi \tag{3a}$$

where

$$f_\phi = \frac{-A_\phi I^{1/2}}{1 + bI^{1/2}} \tag{3b}$$

$$B_\phi = \beta^{(0)} + \beta^{(1)} \exp(-\alpha I^{1/2}) \tag{3c}$$

$$A_\phi = \left(\frac{1}{3}\right)\sqrt{2\pi N_A d_0}\left(\frac{e^2}{2\pi\varepsilon_0 D_e kT}\right)^{3/2} \tag{3d}$$

In these equations, $\beta^{(0)}$, $\beta^{(1)}$, and C_ϕ are ion-interaction parameters of the Pitzer model that are dependent on temperature and pressure, and A_ϕ is the Debye−Hückel constant for the osmotic coefficient in the molality scale. The Pitzer-ion interaction model parameters are given in Table 3 derived using a nonlinear least-squares method. The other quantities are described elsewhere.[25]

The MNRTL equation gives the best empirical description and has real physical meaning for the correlation of osmotic and activity coefficients. In the MNRTL model, the excess Gibbs free energy of an electrolyte solution and consequently the activity coefficients of solvent have been written as the sum of long-range (LR) and short-range (SR) contributions, where subscript s refers to solvent.

$$\ln(\gamma_S) = \ln(\gamma_S^{LR}) + \ln(\gamma_S^{SR}) \tag{4}$$

The long-range contribution is represented by the Pitzer−Debye−Hückel (PDH) equation. The PDH equation for the activity coefficient of component i is

$$\ln(\gamma_i^{PDH}) = -\left(\frac{1}{M_s}\right)^{1/2} A_\phi \left\{\frac{2z_i^2 \ln(1 + \rho I_x^{1/2})}{\rho} + \frac{z_i^2 I_x^{1/2} - 2I_x^{3/2}}{1 + \rho I_x^{1/2}}\right\} \tag{5}$$

where I_x is ionic strength on mole fraction base $I_x = (1/2)\sum z_i^2 x_i$; ρ is the closest distance parameter; and its value is 14.9 for aqueous solutions.

The MNRTL model represents the short-range contribution for the solvent activity coefficient. In this equation, there are four fitting parameters

Table 3. Parameters of the Pitzer Model for the [PrMIm]MSO$_4$ + H$_2$O System at Different Temperatures

T/K	$\beta^{(0)}$	$\beta^{(1)}$	C_ϕ	$\sigma(\phi)$
308.15	0.43397	−2.35801	−0.19815	0.013
318.15	−0.35729	0.55979	0.20409	0.024
328.15	−0.24211	0.31727	0.17750	0.029

Table 4. Parameters of the MNRTL Model for the [PrMIm]MSO$_4$ + H$_2$O System at Different Temperatures

T/K	$\tau_{s,ca}$	$\tau_{ca,s}$	$\omega_{s,ca}$	$\omega_{ca,s}$	$\sigma(\phi)$
308.15	0.02674	4.66537	−2.30969	−5.81940	0.011
318.15	−1.36396	1.43695	8.61940	−0.80884	0.030
328.15	−1.48220	1.42618	8.58447	−0.89772	0.034

$$\ln \gamma_{s,MNRTL} = 4X_c^2[\tau_{ca,s}W_{ca,s}/(X_s + 2W_{ca,s}X_c)^2 + \tau_{s,ca}(W_{s,ca} - 1)/(X_c + X_sW_{ca,s})^2] \quad (6)$$

in which $\tau_{ca,s}$ (salt−solvent parameter) and $\tau_{s,ca}$ (solvent−salt parameter) are the model parameters, and the following definition has been carried out

$$W_i = \exp(-\alpha\tau_i + \omega_i) \quad (7)$$

where the $\omega_{ca,s}$ and $\omega_{s,ca}$ are also adjustable parameters; α is the nonrandomness factor equal to 0.2; and $X_c = z_cx_c$, $X_a = z_ax_a$, and $X_s = x_s$.

The activity coefficients equation was converted to the osmotic coefficient

$$\phi = -[\ln(\gamma_s^{LR}) + \ln(\gamma_s^{SR}) + \ln(x_s)]/\nu mM_s \quad (8)$$

In the preceding equation, ν is the stoichiometric number of the ionic liquid; M_s is the molar mass of the solvent; and x_s is the mole fraction of the solvent. The MNRTL model parameters of the studied systems are given in Table 4. These values show that the Pitzer-ion interaction and MNRTL models correlate satisfactorily the experimental osmotic coefficients of aqueous solutions of studied ionic liquids.

Conductometry provides us information concerning association of ions. Conductivity measurements yield both the association constant as well as information about the relative solvating ability of solvents for the various ions. There are many different equations, which are preferred by authors for the treatment of conductance data.[30] The low concentration chemical model (lcCM) of the conductivity equation has been widely recently applied for the correlation of conductance data in aqueous and nonaqueous electrolyte solution.[27]

The calculated molar conductivities Λ in the aqueous solutions of ionic liquid are given in Table 5. Figure 2 shows the dependence of the molar conductivity Λ on the molar concentration c for the investigated system at different experimental temperatures. It is shown that the molar conductivity decreases with increasing amount of ionic liquid. Increasing ionic liquid concentration causes the formation of ion-pair in the dilute region and the stronger ion association in the studied mixtures. Analysis of conductivity data in the framework of the low-concentration chemical model (lcCM) uses the set of equations

$$\Lambda = \alpha[\Lambda_0 - S(c\alpha)^{1/2} + Ec\alpha\ln(c\alpha) + J_1c\alpha + J_2(c\alpha)^{3/2}] \quad (9)$$

$$K_A = \frac{1-\alpha}{\alpha^2c\gamma_\pm^2} \quad (10)$$

$$\ln \gamma_\pm = -\frac{\kappa q}{1+\kappa R} \quad (11)$$

where

$$\kappa^2 = \frac{2\cdot10^3N_Az^2e^2\alpha c}{\varepsilon_0\varepsilon k_BT} \quad (12)$$

$$q = \frac{z^2e^2}{8\pi\varepsilon_0\varepsilon k_BT} \quad (13)$$

where Λ and Λ_0 are the molar conductivities at molarity c and infinite dilution; α is the fraction of oppositely charged ions acting as ion pairs; K_a is the ion association constant of ionic liquids; R is the distance parameter; $\gamma_\pm$ is the corresponding mean activity coefficient of the free ions. The coefficients of eq 9 reflect the relaxation (rel) and electrophoretic (el) effect. The coefficients E, J_1, and J_2 required for calculation were taken from ref 27. The rest of the parameters have the usual meaning.

Three-parameter fits of molar conductivity data yield the association constant K_a, the limiting molar conductivity Λ_0, and distance parameter R by nonlinear least-squares iteration. K_a, Λ_0, and R parameters are summarized in Table 6. The standard deviation $\sigma(\Lambda)$ of the experimental molar conductivities Λ(exp.) and the calculated one Λ(cal.) was computed as follows

$$\sigma(\Lambda) = (\sum[\Lambda(\text{exp.}) - \Lambda(\text{calc.})]^2/(n-p))^{1/2} \quad (14)$$

where n and p show the number of the experimental data and parameters, respectively.

Increasing K_a values with increasing temperature in Table 6 show that the extent of ion association in [PrMIm]MSO$_4$ + H$_2$O solutions increases with increasing temperature. Therefore, strong ion solvation was observed for [PrMIm]MSO$_4$ + H$_2$O solutions at low temperature which is similar to alkylammonium halide, which occurs by the occupation of intermolecular cavities of an icelike water structure by organic chains of these ions.[31]

Experimental results of refractive indices for the studied aqueous solutions of ionic liquid are reported in Table 7. The values of the refractive indices of the binary systems have been fitted by a polynomial expansion of the form

$$n_D - n_D^* = \sum_{i=1}^{3} A_im^i \quad (15)$$

where m is the molality of the ionic liquid and A_i is the fitting parameter given in Table 8 together with standard deviations of refractive index values. Figure 3 shows the refractive indices of [PrMIm]MSO$_4$ + H$_2$O system plotted against molality of ionic liquid. As can be observed in this figure, the n_D values decrease as the temperature increases. The refractive indices of pure water and pure ionic liquid at different temperatures are given in Table 1.

The experimental density, d, values as a function of ionic liquid concentration, m, for [PrMIm]MSO$_4$ + H$_2$O system are reported in Table 9. The apparent molar volumes V_ϕ of [PrMIm]MSO$_4$ were calculated from the densities of the solutions using the following equation

$$V_\phi = \frac{M}{d} - 1000\frac{(d-d_0)}{mdd_0} \quad (16)$$

where m is the molality of [PrMIm]MSO$_4$ in water; d and d_0 are densities of solution and pure solvent, respectively; and M is the molar mass of [PrMIm]MSO$_4$. The calculated apparent molar volume, V_ϕ, values for the solutions of [PrMIm]MSO$_4$ are given in Table 9 at different temperatures. Figure 4 shows

Table 5. Molar Conductivities Λ and Molar Concentrations *c* of Aqueous Solutions of Ionic Liquid, [PrMIm]MSO$_4$ + H$_2$O, at Different Temperatures

T = 298.15 K		T = 308.15 K		T = 318.15 K		T = 328.15 K	
c (mol·m^{-3})	Λ (S·cm^2·mol^{-1})	c (mol·m^{-3})	Λ (S·cm^2·mol^{-1})	c (mol·m^{-3})	Λ (S·cm^2·mol^{-1})	c (mol·m^{-3})	Λ (S·cm^2·mol^{-1})
0.8737	215.29	0.8029	260.35	1.0101	266.24	0.80469	286.24
1.6260	205.28	1.6707	250.82	1.7383	259.12	1.68827	276.58
2.4027	199.69	2.7412	244.03	2.5996	249.55	2.57975	267.79
2.6615	198.12	3.6009	241.17	3.5549	241.99	3.42388	257.82
3.8588	192.62	4.4443	238.63	4.6511	234.92	4.29169	249.28
5.1046	187.91	5.5879	236.14	5.4889	231.13	5.28572	237.74
6.1482	184.25	6.7395	233.48	6.2406	225.88	6.24031	230.84
7.1594	179.87	7.8100	232.85	7.1175	220.67	7.28167	222.28
8.3486	175.09	8.9779	230.29	7.9867	215.31	8.31515	215.57
9.2547	172.76	9.9754	227.71	8.9028	210.90	9.44329	208.88
10.2983	168.26	10.9081	225.57	9.6702	207.61	10.47677	204.50
11.3661	167.06	11.9949	222.64	10.8055	204.77	11.52603	199.16
12.3773	161.65	13.1546	219.51	11.6747	199.55	12.55161	192.53

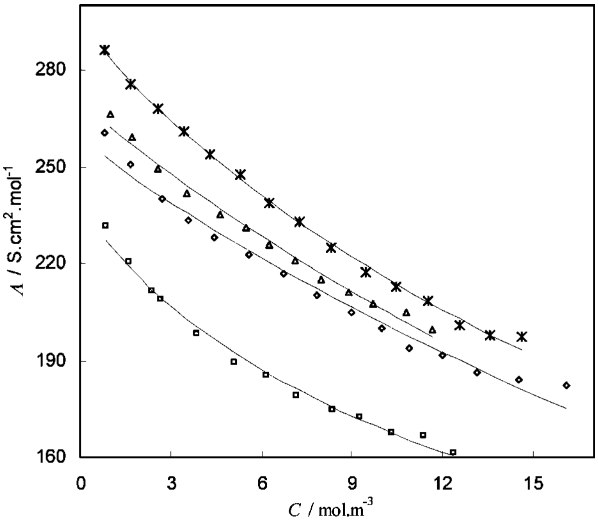

Figure 2. Molar conductivity of aqueous solutions of ionic liquids, [PrMIm]MSO$_4$, at experimental temperatures. Lines were generated using the lcCM model. Data: □, 298.15 K; ◇, 308.15 K; △, 318.15 K; *, 328.15 K.

Table 6. Ion Association Constant K_a, Limiting Molar Conductance Λ$_0$, Distance *R*, and Standard Deviation of [PrMIm]MSO$_4$ + H$_2$O in Low Concentrations at Different Temperatures

T/K	K_a (dm^3·mol^{-1})	Λ$_0$ (S·cm^2·mol^{-1})	10^{10}·R m	$\sigma(\Lambda_0)$
298.15	14.69 ± 0.62	213.30 ± 0.05	21.84	0.78
308.15	20.46 ± 0.75	259.75 ± 0.06	21.45	0.98
318.15	21.76 ± 0.25	271.14 ± 0.06	22.36	0.40
328.15	32.08 ± 0.43	295.34 ± 0.08	24.11	0.51

the apparent molar volumes of [PrMIm]MSO$_4$ + H$_2$O system plotted against molality of ionic liquid. In solvents for which the Debye−Hückel limiting slopes for apparent molar volume, A_V, are known, the concentration dependence of V_ϕ at fixed temperature and pressure can be described using the following Redlich−Mayer equation in the dilute region[32]

$$V_\phi = V_\phi^0 + S_V m^{1/2} + b_V m \tag{17}$$

where V_ϕ^0 is the limiting apparent molar volume (equal to the partial molar volume at infinite dilution, V_2^0); b_V is an empirical parameter; and S_V is defined as

$$S_V = A_V\left[\frac{1}{2}\left(\sum_i \nu_i z_i^2\right)\right]^{3/2} \tag{18}$$

where ν_i and z_i are the stiochiometric number and absolute charge of ion i, respectively. The value of A_V is the Pitzer−Debye−Hückel limiting slope for apparent molar volumes in water at different temperatures, which were calculated by Ananthaswamy and Atkinson,[33] which are shown in Table 10. The values of V_ϕ^0 and b_V values were obtained by a least-squares analysis of eq 17, and their values for each mixture at the studied experimental temperatures together with standard deviations are listed in Table 10. The calculated V_ϕ values of this ionic liquid in aqueous solutions at the experimental

Table 7. Refractive Indices n_D of Aqueous Solutions of [PrMIm]MSO$_4$ as a Function of Ionic Liquid Concentration at Different Temperatures

m/mol·kg^{-1}	T = 298.15 K	T = 308.15 K	T = 318.15 K	T = 328.15 K
0.0122	1.3329	1.3319	1.3300	1.3289
0.0381	1.3334	1.3325	1.3308	1.3294
0.0509	1.3338	1.3328	1.3313	1.3299
0.0732	1.3345	1.3334	1.3318	1.3303
0.0894	1.3348	1.3337	1.3324	1.3307
0.1377	1.3359	1.3348	1.3335	1.3318
0.3004	1.3398	1.3386	1.3371	1.3352
0.4427	1.3429	1.3417	1.3401	1.3382
0.6101	1.3468	1.3452	1.3437	1.3417
0.7409	1.3496	1.3481	1.3469	1.3445
0.8646	1.3522	1.3506	1.3493	1.3470
0.9948	1.3549	1.3535	1.3519	1.3498
1.0961	1.3569	1.3557	1.3537	1.3519

Table 8. Parameters of Polynomial Expansion (eq 15) Fitting and Standard Deviation of Refractive Indices of the System [PrMIm]MSO$_4$ + H$_2$O at Different Temperatures

T/K	n_D*	10^4A_1	10^4A_2	10^4A_3	$10^4\sigma(n_D - n_D^*)$
298.15	1.3325	251.6829	−33.8033	6.8092	0.8350
308.15	1.3316	242.7067	−43.6549	20.8233	0.4942
318.15	1.3301	234.1238	−7.1179	−8.5762	1.9805
328.15	1.3284	250.1996	−75.6717	39.8836	1.4202

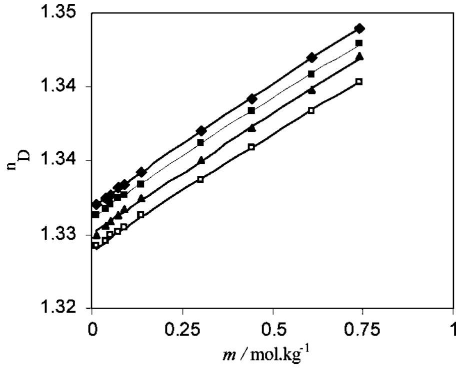

Figure 3. Refractive indices of aqueous solutions of [PrMIm]MSO$_4$ at experimental temperatures. □, 328.15 K; ▲, 318.15 K; ■, 308.15 K; ♦, 298.15 K.

Table 9. Molal Concentrations *m*, Density *d*, and Apparent Molar Volume V_ϕ of Aqueous Solution of [PrMIm]MSO$_4$ at Different Temperatures

	T = 298.15 K		T = 308.15 K		T = 318.15 K		T = 328.15 K	
m mol·kg^{-1}	d g·cm^{-3}	V_ϕ cm^3·mol^{-1}	d g·cm^{-3}	V_ϕ cm^3·mol^{-1}	d g·cm^{-3}	V_ϕ cm^3·mol^{-1}	d g·cm^{-3}	V_ϕ cm^3·mol^{-1}
0.0130	0.99775	181.92	0.99468	186.99	0.99076	195.371	0.98552	200.35
0.0410	0.99928	181.63	0.99601	188.46	0.99188	196.755	0.98658	200.56
0.0490	0.99968	182.31	0.99634	189.56	0.99228	195.136	0.98686	201.01
0.0720	1.00075	184.47	0.99730	191.12	0.99318	195.971	0.98764	202.04
0.0790	1.00108	184.80	0.99765	190.64	0.99338	197.073	0.98789	202.05
0.1270	1.00328	186.40	0.99965	191.84	0.99511	198.298	0.98939	203.70
0.2540	1.00838	189.92	1.00404	195.86	0.99891	201.922	0.99322	205.08
0.4090	1.01354	193.21	1.00875	198.35	1.00345	202.900	0.99704	207.23
0.5060	1.01612	195.32	1.01100	200.35	1.00582	203.950	0.99888	208.86
0.6560	1.01999	197.26	1.01450	201.97	1.00904	205.447	1.00200	209.73
0.8730	1.02508	199.09	1.02005	202.25	1.01346	206.615	1.00528	211.74
1.0210	1.02837	199.85	1.02271	203.41	1.01621	207.216	1.00774	212.18
1.3670	1.03403	202.27	1.02927	204.38	1.01888	211.126	1.00999	215.69
1.5690	1.03595	204.08	1.03046	206.64	1.02031	212.600	1.01224	216.13

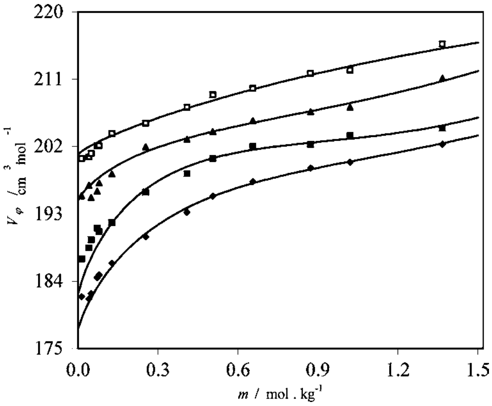

Figure 4. Apparent molar volume, V_ϕ, of aqueous solutions of [PrMIm]MSO$_4$ at experimental temperatures. Lines were generated using the Pitzer model. □, 328.15 K; ▲, 318.15 K; ■, 308.15 K; ◆, 298.15 K.

temperatures were also plotted in the form of $V_\phi - A_V\sqrt{m}$ against molality of ionic liquid (Figure 5). At infinite dilution, each ion is surrounded only by the solvent molecules and being infinitely distant with other ions. It follows, therefore, that V_ϕ^0 is unaffected by ion−ion interaction, and it is a measure only of the ion−solvent interaction. Table 10 shows that b_V values decrease with increasing temperature indicating the increased nonelectrostatic interactions of [PrMIm]MSO$_4$ at high temperatures.[34]

We have also tested the reliability of the Pitzer equation in the correlation of the experimental apparent molar volume data for the solutions of ionic liquid with water. The Pitzer equation for 1:1 salts has the form

$$V_\phi = V_\phi^0 + \nu|z_c z_a|\left(\frac{A_V}{2b}\right)\ln(1 + bI^{1/2}) + 2RT\nu_c\nu_a m[B_V + m\nu_c z_c C_V] \quad (19)$$

where ν_c and ν_a are the number of cations and anions and $\nu = \nu_c + \nu_a$; the subscripts c and a are used for cation and anion, respectively; and z_c and z_a are the number of charges on cations and anions; and b is the parameter for which the value of 1.2 kg$^{1/2}$·mol$^{-1/2}$ is usually used in water. The coefficient B_V is defined as

$$B_V = \beta_V^0 + \beta_V^1\left(\frac{2}{\alpha^2 I}\right)[1 - (1 + \alpha I^{1/2})\exp(-\alpha I^{1/2})] \quad (20)$$

where β_V^0, β_V^1, and C_V are pressure derivative parameters of Pitzer ion interaction parameters for the osmotic coefficient expression. The results of fitting V_ϕ values to eq 19 are also given in Table 10. This table shows that the difference between V_ϕ^0 values obtained from eq 17 and those obtained from the Pitzer equation in most cases is less than 1 cm^3·mol^{-1}. Furthermore, small standard deviations of the fit reported in this table prove that the Pitzer model is a powerful tool for correlation of volumetric properties of solutions of the ionic liquid [PrMIm]MSO$_4$.

The viscosity of [PrMIm]MSO$_4$ + H$_2$O solutions were measured at temperatures from (298.15 to 328.15) K and reported in Table 11. Figure 6 shows the viscosity of ionic liquid + H$_2$O system plotted against the molality of ionic liquid. The viscosities of aqueous solutions of ionic liquid were obtained using the Jones−Dole equation[35]

$$\frac{[(\eta/\eta_0) - 1]}{C^{1/2}} = A + BC^{1/2} \quad (21)$$

where C is the molar concentration of the ionic liquid in mol·dm^{-3}; the A coefficient is a measure of ion−ion interactions; the B coefficient is a measure of ion−solvent interactions; and η and η_0 are viscosity of solution and viscosity of pure water, respectively. The viscosity A and B coefficients have been calculated as the intercept and the slope from plots of $[(\eta/\eta_0) - 1]/C^{1/2}$ against $C^{1/2}$. It is seen that the viscosity A coefficient is negative in some cases, but this has no physical significance. For salts involving multivalent ions and salts involving large hydrophobic ions in aqueous solutions and for salts in nonaqueous solvents, B-coefficients are commonly large and almost always positive.[36] As expected, the viscosity of ionic liquid and the B coefficients decrease with an increase in temperature. The Jones−Dole A, B-coefficients for [PrMIm]MSO$_4$ + H$_2$O solutions are reported in Table 12 at different temperatures.

The coefficient B has two contributions: one is negative, and the other is positive. The positive contribution comes from the tendency of the ionic liquid to attract the H$_2$O molecule, while the negative contribution comes from the fact that the structure of the H$_2$O molecule itself goes against this tendency. So, these two opposing influences can cause a breakdown in the H$_2$O structure beyond the solvation shell. Table 12 shows the positive contribution prevails over the negative one, producing an increase in the viscosity of the solution with respect to that of the pure H$_2$O (Figure 6). The B-coefficient is positive throughout all experimental temperatures, indicating a structure-making behavior of the ionic liquid. The thermal coefficient (dB/dT) is negative. This change may be due to the strongest solute−solvent interactions in the lower temperature.

Table 10. Coefficients of the Redlich−Mayer and Pitzer Equations and the Corresponding Standard Deviations $\sigma(V_\phi)$ of $[PrMIm]MSO_4 + H_2O$ Mixtures for the Apparent Molar Volumes[a]

T/K	(Redlich)V_ϕ^0 $cm^3 \cdot mol^{-1}$	S_V $(cm^3 \cdot kg^{0.5} \cdot mol^{-1.5})$	b_V $(cm^3 \cdot kg \cdot mol^{-2})$	$\sigma(V_\phi)$	(Pitzer)V_ϕ^0 $cm^3 \cdot mol^{-1}$	$\beta_V{}^0 10^3$	$\beta_V{}^1 10^3$	$C_V 10^3$	$\sigma(V_\phi)$
298.15	181.72 ± 0.47	1.874	23.95 ± 1.52	0.93	177.53 ± 0.87	−0.62	2.91	0.18	0.98
308.15	188.15 ± 0.48	2.055	20.49 ± 1.55	0.95	182.19 ± 1.28	−1.01	3.72	0.29	1.45
318.15	195.23 ± 0.45	2.260	14.18 ± 0.88	0.88	194.67 ± 0.68	−0.31	1.34	0.13	0.77
328.15	200.07 ± 0.27	2.495	12.65 ± 0.22	0.54	200.84 ± 0.65	0.17	0.2	−0.03	0.74

[a] $\sigma(V_\phi) = [\sum_i (V_\phi(\exp) - V_\phi(\text{cal}))]^2/n]^{1/2}$.

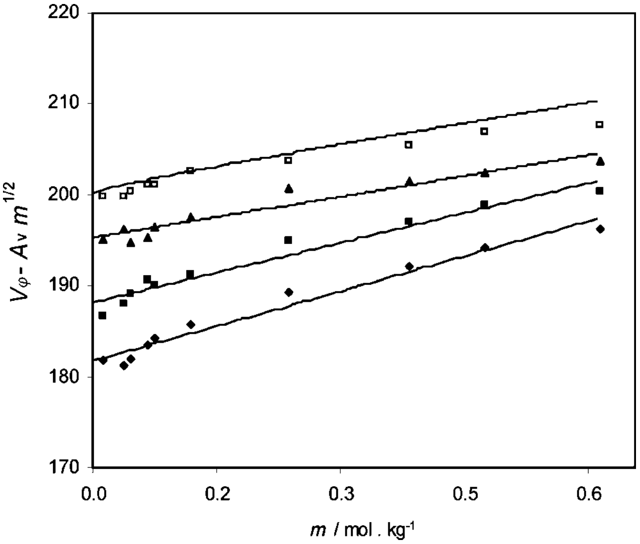

Figure 5. Apparent molar volume V_φ of aqueous solutions of $[PrMIm]MSO_4$ at experimental temperatures. Lines were generated using the Redlich−Mayer model. □, 328.15 K; ▲, 318.15 K; ■, 308.15 K; ◆, 298.15 K.

Table 11. Experimental Dynamic Viscosities η of Aqueous Solutions of $[PrMIm]MSO_4 + H_2O$ at Different Temperatures

	η/(mPa·s)			
m/mol·kg^{-1}	T = 298.15 K	T = 308.15 K	T = 318.15 K	T = 328.15 K
0.0156	0.799	0.699	0.620	0.566
0.0429	0.805	0.709	0.638	0.570
0.0519	0.814	0.717	0.642	0.575
0.0760	0.819	0.727	0.647	0.580
0.0905	0.813	0.735	0.660	0.588
0.1342	0.829	0.758	0.658	0.602
0.2693	0.838	0.796	0.702	0.647
0.4332	0.885	0.847	0.757	0.701
0.5359	0.931	0.888	0.810	0.736
0.6952	0.999	0.930	0.865	0.790
0.9249	1.113	1.021	0.963	0.878
1.0817	1.182	1.072	1.018	0.953
1.4481	1.387	1.205	1.167	1.054
1.6624	1.480	1.268	1.247	1.122

Conclusions

Osmotic coefficients of aqueous solutions of ionic liquid 1-propyl-3-methylimidazolium methyl sulfate $[PrMIm]MSO_4$ were determined by vapor pressure osmometery method at temperature ranges T = (298.15 to 328.15) K. The osmotic coefficients were reliably correlated by the Pitzer-ion interaction and MNRTL models. Electrical conductance properties of ionic liquids in water have been reported at temperatures from T = (298.15 to 328.15) K. Ion association constant values increase with increasing temperature showing that the extent of ion association increases with increasing temperature and ion−solvent interactions decreases. Measurements of viscosity, density and Refractive indices were made. The B coefficient for viscosity was positive at all temperatures, indicating a structure making behavior of ionic liquid. The thermal coefficient (dB/dT) was negative. This change may be due to the strongest solute−solvent interactions in the lower temperature.

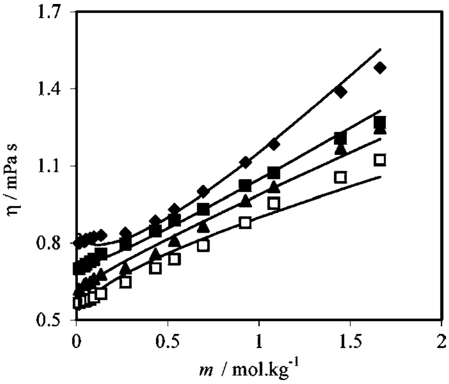

Figure 6. Viscosity of aqueous solutions of $[PrMIm]MSO_4$ at □, 328.15 K; ▲, 318.15 K; ■, 308.15 K; ◆, 298.15 K.

Table 12. Jones−Dole A, B-Coefficients for $[PrMIm]MSO_4 + H_2O$ Solutions at Different Temperatures

T/K	A/(dm$^{3/2}$·mol$^{-1/2}$)	B/(dm^3·mol^{-1})
298.15	−0.6494 ± 0.054	0.9211 ± 0.075
308.15	−0.1631 ± 0.030	0.6041 ± 0.041
318.15	0.2017 ± 0.029	0.4454 ± 0.040
328.15	0.5626 ± 0.073	0.2161 ± 0.100

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