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Volumetric and Viscosity Study for the Mixtures of 2-Ethoxy-2-methylpropane, Ethanol, and 1-Ethyl-3-methylimidazolium Ethyl Sulfate Ionic Liquid

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Densities, viscosities, refractive indices, and speeds of sound at the temperature of 298.15 K and atmospheric pressure are reported for the binary and the miscible ternary mixtures of 2-ethoxy-2-methylpropane, ethanol, and 1-ethyl-3-methylimidazolium ethyl sulfate ionic liquid. Excess molar volumes and viscosity, molar refraction, and isentropic compressibility changes of mixing have been calculated from the physical properties. The changes of mixing have been adequately fitted to the Redlich−Kister polynomial.

Introduction

Ionic liquids have been called designer solvents due to their ability to vary the ions, thereby modifying and optimizing the ionic liquid physical properties for a specific task. For this reason, a great number of applications are now being demonstrated for ionic liquids as catalysts, reactions media, and electrolytes in electrochemistry; in fuel and solar cells; as lubricants; as a stationary phase for chromatography; as matrixes for mass spectrometry, as supports for the immobilization of enzymes in separation technologies; as liquid crystals or templates for the synthesis of mesoporous, nanomaterials and ordered films; as materials for embalming and tissue preservation; etc.

The experimental measurement and theoretical modeling of physical properties of ionic liquids and mixtures are essential in the development and design of processes where they are involved. Moreover, knowledge of excess properties lets us develop an understanding of the interactions that determine the physical properties, making it easier to search for an optimal ionic liquid for a determined application. Nonetheless, ionic liquids research is still a relatively new field with insufficient available physical property data or work about correlation and prediction equations. Publications reporting physical or excess properties for mixtures of ionic liquids with more than one component are very scarce.[1−3]

In this paper densities ρ, dynamic viscosities η, refractive indices n_D, speeds of sound u, and isentropic compressibilities κ_s will be determined for the complete range of miscible mixtures of 2-ethoxy-2-methylpropane (ethyl *tert*-butyl ether or ETBE), ethanol, and 1-ethyl-3-methylimidazolium ethyl sulfate ([emim][EtSO$_4$] or EMISE) at 298.15 K and atmospheric pressure. For engineering use, density and viscosity would be the most appreciated properties. Moreover, excess molar volumes V^E, viscosity $\Delta\eta$, molar refraction ΔR, and isentropic compressiblity $\Delta\kappa_s$ changes of mixing calculations will be carried out using the measured properties and correlated with composition data using the Redlich−Kister polynomial. The interest of the mixture is due to the capability of ionic liquid as azeotrope breaker of (ETBE + ethanol) system in an extractive distillation. No comparable data in the surveyed literature for the binary system (ethanol + [emim][EtSO$_4$]) and for the ternary system (ETBE + ethanol + [emim][EtSO$_4$]) have been found.

Experimental Section

Materials: Synthesis of 1-Ethyl-3-methyl Imidazolium Ethyl Sulfate. Ionic liquid was prepared by reaction of equimolecular quantities of 1-methylimidazole (Aldrich, > 99 %, GC) and diethyl sulfate (Aldrich, > 98 %, GC) in toluene.[4,5] A solution of 1-methylimidazole in toluene was prepared first, and then diethyl sulfate was added dropwise under helium atmosphere. Reaction was done on an ice bath to avoid raising the temperature over 313.15 K. As [emim][EtSO4] is nonsoluble in toluene, it was separated from reagents and solvent and then washed three times with fresh toluene. Removal of residual volatile compounds in the ionic liquid was carried out first in a rotary evaporator (348.15 K, 1800 s) and later under vacuum (1333 Pa) at 353.15 K for 48 h.

The presence of trace amounts of impurities such as water or ions can have a dramatic effect on density and viscosity values as it was established in the studies of Seddon et al.[6] For this reason, it is usual to find several differences on physical properties even for pure compounds when we are talking about ionic liquids. In this work, synthesis of EMISE by alkylation of the imidazole nucleus with ethyl sulfate ensures the complete absence of halide ions,[4] and water content of ionic liquid is less than 100 ppm.

ETBE with a purity of 99.4 %, checked by gas chromatography, was obtained from the Department of Chemical Engineering of the University of Barcelona, Spain. Ethanol with a nominal purity of 99.9 % was purchased from Merck. Densities, refractive indices, and water contents, determined by means of a Karl Fischer titration method carried out in a Metrohm 737 KF, are summarized along with published values[5,7−10] in Table 1 for pure compounds.

Chemicals used for the calibration of viscometer were acetone, 1-pentanol, 1,2-propanediol, and 1,3-butanediol (all chromatographic grade and purchased from Sigma-Aldrich and Fluka). Water used in densimeters and refractometer calibration was purified using a Milli-Q Plus system.

Experimental Apparatus and Procedure. The samples were prepared by filling glass vials with the liquids and weighing at once in a Mettler AE 240 balance, which measured to within ± 0.0001 g, for calculating the composition by weight difference. Vials are closed with screw caps to ensure a secure seal and to prevent evaporation. Additions are made with a syringe through a PTFE-faced silicone septum.

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Table 1. Water Content, Density ρ, Dynamic Viscosity η, and Refractive Index n_D of the Pure Components of the Ternary System Studied in This Work, at 298.15 K and Atmospheric Pressure

			ρ/g·cm^{-3}		η/mPa·s		n_D	
component	CASRN[a]	water content	exp.	lit.	exp.	lit.	exp.	lit.
ETBE	637-92-3	0.03 %	0.73551	0.7350[7]	0.371	not found	1.37298	1.3730[8]
ethanol	64-17-5	0.04 %	0.78522	0.78493[9]	1.077	1.0826[9]	1.35923	1.35941[9]
[emim][EtSO$_4$]	342573-75-5	98 ppm	1.23882	1.2296[5] 1.2423[10]	100.4	not found	1.47889	not found

[a] CASRN, Chemical Abstracts Service Registry Number.

Table 2. Physical and Excess Properties for Mixtures of ETBE (1) + Ethanol (2) + [emim][EtSO$_4$] (3) at 298.15 K and Atmospheric Pressure

x_1	x_2	ρ g·cm^{-3}	η mPa·s	n_D	u m·s^{-1}	κ_S TPa^{-1}	V^E cm^{-3}·mol^{-1}	$\Delta\eta$ mPa·s	ΔR cm^{-3}·mol^{-1}	$\Delta\kappa_S$ TPa^{-1}
0.0000	0.9522	0.85234	1.589	1.37769	1200	814	−0.266	−4.234	0.020	−62
0.0527	0.9020	0.84187	1.428	1.37823	1183	848	−0.412	−4.107	0.021	−71
0.1407	0.8182	0.82609	1.208	1.37835	1163	895	−0.595	−3.848	0.009	−84
0.2556	0.7089	0.80827	0.974	1.37854	1141	950	−0.749	−3.456	0.016	−94
0.3132	0.6539	0.80017	0.878	1.37889	1116	1003	−0.779	−3.237	0.043	−70
0.0000	0.9062	0.90503	2.138	1.39213	1248	710	−0.440	−8.255	0.038	−90
0.0720	0.8410	0.88524	1.860	1.39070	1216	764	−0.628	−7.812	0.028	−98
0.1589	0.7622	0.86365	1.568	1.38882	1181	830	−0.787	−7.233	0.016	−97
0.2368	0.6916	0.84630	1.352	1.38711	1154	888	−0.883	−6.669	0.005	−91
0.0000	0.8666	0.94332	2.709	1.40265	1286	641	−0.553	−11.61	0.055	−102
0.0770	0.7999	0.91873	2.401	1.39974	1244	703	−0.768	−10.85	0.034	−110
0.1736	0.7162	0.88991	1.937	1.39636	1198	784	−0.873	−9.963	0.039	−107
0.0000	0.8060	0.99200	3.860	1.41553	1339	562	−0.649	−16.49	0.062	−109
0.0564	0.7605	0.97135	3.565	1.41298	1302	607	−0.832	−15.66	0.065	−118
0.1236	0.7064	0.94764	3.102	1.40981	1260	665	−0.970	−14.78	0.072	−120
0.0000	0.7508	1.02842	5.078	1.42508	1382	509	−0.692	−20.75	0.066	−107
0.0338	0.7254	1.01518	4.792	1.42310	1357	535	−0.816	−20.17	0.061	−114
0.0848	0.6872	0.99572	4.386	1.42047	1320	576	−0.972	−19.29	0.072	−121
0.0000	0.7174	1.04766	5.975	1.43017	1406	483	−0.707	−23.17	0.070	−104
0.0350	0.6923	1.03375	5.651	1.42838	1379	508	−0.844	−22.49	0.083	−112
0.0845	0.6567	1.01434	5.135	1.42536	1341	548	−1.005	−21.58	0.081	−120
0.0000	0.5951	1.10450	10.65	1.44516	1467	421	−0.702	−30.64	0.084	−79
0.0501	0.5653	1.08483	9.768	1.44179	1441	444	−0.983	−29.47	0.065	−103
0.0000	0.5103	1.13481	15.40	1.45298	1525	379	−0.664	−34.31	0.082	−73
0.0465	0.4866	1.11699	14.20	1.44977	1488	405	−0.970	−33.22	0.057	−90
0.0000	0.4147	1.16259	22.88	1.46005	1566	351	−0.577	−36.33	0.076	−57
0.0475	0.3950	1.14589	21.09	1.45757	1532	372	−1.033	−35.32	0.072	−78
0.0000	0.3256	1.18410	32.21	1.46557	1598	331	−0.491	−35.85	0.072	−43
0.0290	0.3162	1.17390	30.74	1.46442	1580	341	−0.737	−35.36	0.108	−57
0.0000	0.2039	1.20805	49.77	1.47144	1636	309	−0.320	−30.38	0.048	−26
0.0324	0.1973	1.19784	46.30	1.46984	1617	319	−0.695	−31.26	0.040	−42
0.0000	0.1271	1.22067	65.47	1.47457	1657	298	−0.183	−22.32	0.039	−16
0.0174	0.1248	1.21569	63.01	1.47383	1648	303	−0.434	−23.26	0.031	−25
0.0000	1.0000	0.78522	1.077	1.35923	1144	973	0.000	0.000	0.000	0
0.0000	0.9504	0.85474	1.628	1.37833	1202	810	−0.281	−4.379	0.019	−63
0.0000	0.9026	0.90892	2.189	1.39300	1253	700	−0.462	−8.564	0.031	−94
0.0000	0.7866	1.00544	4.312	1.41900	1352	544	−0.662	−17.96	0.062	−107
0.0000	0.7676	1.01799	4.678	1.42246	1369	524	−0.681	−19.48	0.070	−107
0.0000	0.7120	1.05050	6.132	1.43097	1407	481	−0.702	−23.55	0.074	−102
0.0000	0.5926	1.10555	10.83	1.44549	1480	413	−0.704	−30.71	0.087	−85
0.0000	0.5724	1.11325	11.74	1.44727	1490	405	−0.696	−31.81	0.074	−81
0.0000	0.5223	1.13096	14.78	1.45198	1516	384	−0.676	−33.75	0.081	−74
0.0000	0.4604	1.15007	18.98	1.45680	1545	364	−0.629	−35.69	0.073	−64
0.0000	0.4336	1.15767	21.39	1.45888	1558	356	−0.612	−35.94	0.080	−60
0.0000	0.3598	1.17632	28.75	1.46366	1586	338	−0.532	−35.91	0.079	−48
0.0000	0.3088	1.18770	34.69	1.46642	1603	328	−0.466	−35.04	0.066	−41
0.0000	0.2187	1.20533	47.69	1.47092	1631	312	−0.332	−30.99	0.064	−28
0.0000	0.1281	1.22061	66.09	1.47476	1655	299	−0.199	−21.59	0.056	−16
0.0000	0.0000	1.23882	100.4	1.47889	1683	285	0.000	0.000	0.000	0

Densities were measured in an Anton Paar DMA 60/602 densimeter. The uncertainty in the measurement is ± 0.00001 g·cm^{-3}. Speeds of sound were measured with an uncertainty of ± 1 m·s^{-1} in an Anton Paar DSA 48 densimeter and sound analyzer. Both densimeters were calibrated with air and water. Refractive indices were measured in an ATAGO RX-5000 refractometer calibrated with water. The uncertainty in the measurement is ± 0.00004. Temperature was controlled with a Heto Therm thermostat to maintain the temperature at (298.15 ± 0.02) K.

The viscosity was determined by micro Ubbelohde viscometer technique. Three micro Ubbelohde viscometers (capillaries I, II, and III) were used in the experiments according to the different viscosity values for the mixtures. The viscometer was calibrated using compounds with different viscosities: acetone, 1-pentanol, 1,2-propanediol, and 1,3-butanediol. Flow time measurements are performed by Lauda Processor Viscosity System PVS1 with a resolution of 0.01 s. The temperature of the viscometer was kept constant using a Lauda clear view thermostat D 20 KP with a through-flow cooler DLK 10 and a thermal stability of ± 0.005 K. Viscosity measurements were repeated at least three times for each sample and were found to be repeatable to within ± 0.03 s for times less than 100 s and ± 0.5 s for bigger times. Kinematic viscosity of solution υ is given by

$$\upsilon = Ct - K/t \tag{1}$$

where ρ is the solution density, t is the flow time, and C and K are the viscometer constants calculated from the measurements performed with the calibration chemicals at 298.15 K. Viscosity of these compounds was taken from Riddick et al.[9] and George and Sastry.[11] Dynamic viscosities are calculated from kinematic viscosities and densities. The uncertainty for the dynamic viscosity determination is estimated to be ± 0.5 %.

Results

Preliminary a cloud-point method[12] was used to determine the solubility curve of ternary system, aimed at defining the range of miscible mixtures. The experimental values obtained of densities ρ, dynamic viscosities η, speeds of sound u, and refractive indices n_D measured for the single phase mixtures of (ETBE + ethanol + [emim][EtSO$_4$]) and for the binary mixture (ethanol + [emim][EtSO$_4$]) are listed in Table 2. Data for the binary (ETBE + ethanol) system have already been published.[13] The same table includes the calculated values of isentropic compressibilities κ_s, excess molar volumes V^E, viscosity $\Delta\eta$, molar refraction ΔR, and isentropic compressibility changes of mixing $\Delta\kappa_s$.

The excess molar volumes V^E, viscosity $\Delta\eta$, and molar refraction ΔR changes of mixing are calculated as a function of mole fraction, x_i, using the following expressions:

$$V^E = V_M - \sum_i x_i V_i \tag{2}$$

$$\Delta\eta = \eta - \sum_i x_i \eta_i \tag{3}$$

$$\Delta R = R_M - \sum_i x_i R_i \tag{4}$$

where V_M is the molar volume of the mixture, η is the dynamic viscosity, R_M is the molar refraction of the mixture obtained from the Lorentz−Lorenz equation, and V_i, η_i, and R_i are the molar volume, the viscosity, and the molar refraction, respectively, for the component i.

Isentropic compressibilities κ_s and their changes of mixing $\Delta\kappa_s$ are calculated using the relationships:

$$\kappa_s = u^{-2}\rho^{-1} \tag{5}$$

$$\Delta\kappa_s = \kappa_s - \sum_i \phi_i \kappa_{si} \tag{6}$$

where κ_s and κ_{si} are the isentropic compressibilities of the mixture and component i, respectively, and ϕ_i is the volume fraction of component i in the mixture given by

$$\phi_i = x_i V_i / \sum_j x_j V_j \tag{7}$$

and j refers to all the components in the mixture.

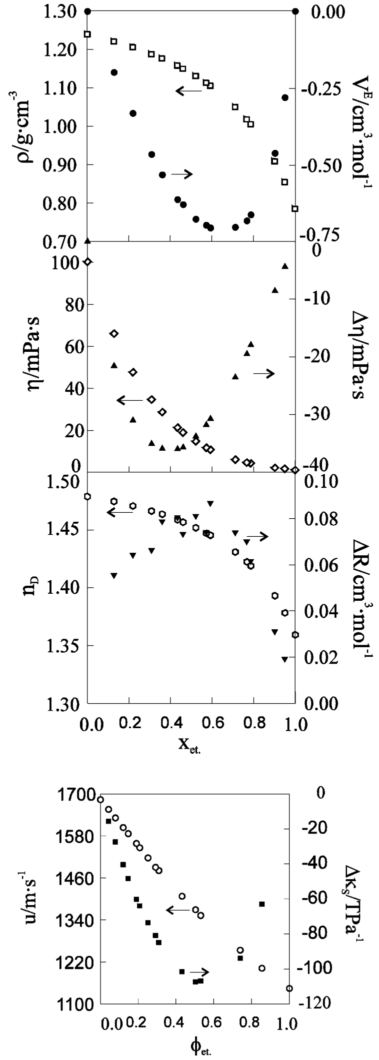

Figure 1. Physical and excess properties for (ethanol + 1-ethyl-3-methylimidazolium ethyl sulfate) system at 298.15 K and atmospheric pressure: □, density ρ; ◇, dynamic viscosity η; ⬠, refractive index n_D; ○, speed of sound u; ●, excess molar volume V^E; ▲, viscosity change of mixing $\Delta\eta$; ▼, molar refraction change of mixing ΔR; ■, isentropic compressiblity change of mixing $\Delta\kappa_s$.

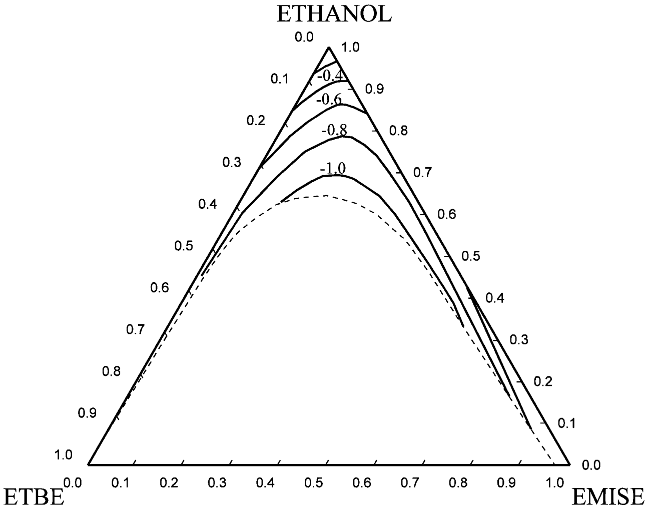

Figure 2. Excess molar volume (cm^3·mol^{-1}) isolines for (ethyl *tert*-butyl ether + ethanol + 1-ethyl-3-methylimidazolium ethyl sulfate) at 298.15 K and atmospheric pressure (system compositions in mole fraction).

Figure 1 shows the physical and excess properties for (ethanol + [emim][EtSO$_4$]) at 298.15 K and atmospheric pressure. For the ternary system, Figures 2 and 3 show the excess molar volume and the dynamic viscosity changes of mixing isolines (system compositions in mole fraction, x_i). Figure 4 shows the

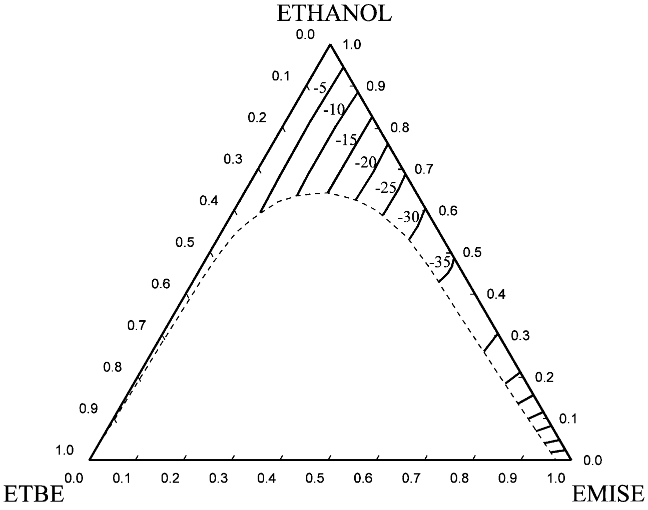

Figure 3. Dynamic viscosity changes of mixing (mPa·s) isolines for (ethyl *tert*-butyl ether + ethanol + 1-ethyl-3-methylimidazolium ethyl sulfate) at 298.15 K and atmospheric pressure (system compositions in volume fraction).

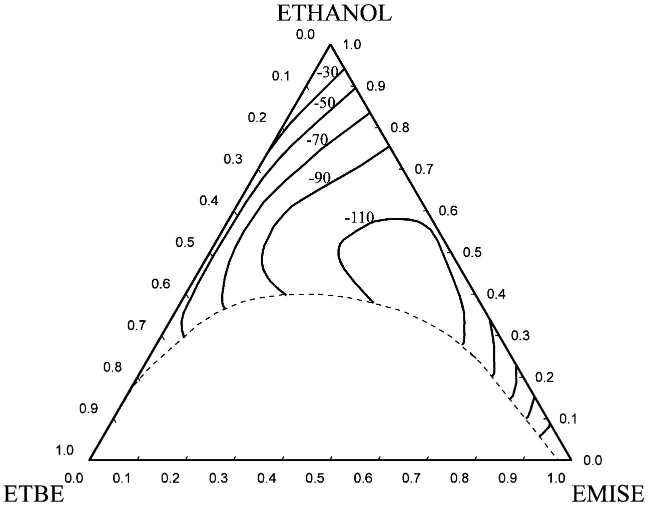

Figure 4. Isentropic compressibility changes of mixing (TPa^{-1}) isolines for (ethyl *tert*-butyl ether + ethanol + 1-ethyl-3-methylimidazolium ethyl sulfate) at 298.15 K and atmospheric pressure (system compositions in volume fraction).

Table 3. Polynomial Coefficients and Standard Deviations σ Obtained for Fits of Equation 8 to the V^E, $\Delta\eta$, ΔR, and $\Delta\kappa_s$ Composition Data for the Binary Systems[a]

property	A_0	A_1	A_2	A_3	σ
	[emim][EtSO$_4$] (1) + Ethanol (2)				
$V^E/cm^3 \cdot mol^{-1}$	−2.6203	1.2458	−1.0965	1.5579	0.007
$\Delta\eta$/mPa·s	−138.15	−63.15	−16.72		0.15
$\Delta R/cm^3 \cdot mol^{-1}$	0.3396				0.007
$\Delta\kappa_s/TPa^{-1}$	−426.3	93.8	−47.0		0.5
	Ethanol (1) + ETBE (2)				
$V^E/cm^3 \cdot mol^{-1}$	−2.8420	0.2218	−0.7658		0.003
$\Delta\eta$/mPa·s	−0.8173	−0.3399			0.005
$\Delta R/cm^3 \cdot mol^{-1}$	−0.1435				0.002
$\Delta\kappa_s/TPa^{-1}$	−168.0	25.1	−60.3	112.3	0.6

[a] For $\Delta\kappa_s$, system compositions were in volume fractions, ϕ_i.

isentropic compressibility changes of mixing isolines (system composition in volume fraction, ϕ_i).

Correlation

The V^E, $\Delta\eta$, ΔR, and $\Delta\kappa_s$ calculated data were correlated with the composition data by means of the Redlich−Kister polynomial,[14] which for binary mixtures is

$$Q_{ij} = x_i\, x_j \sum_k A_k (x_i - x_j)^k \tag{8}$$

where Q_{ij} is V^E, $\Delta\eta$, or ΔR and x_i is the mole fraction of component i, Q_{ij} is $\Delta\kappa_s$, with x_i as the volume fraction of component i. A_k is the polynomial coefficient, and k is the number of the polynomial coefficient. For ternary systems the corresponding equation, as a function of the composition, x_i (in mole or volume fraction), is

$$Q_{123} = Q_{21} + Q_{32} + Q_{13} + x_1x_2x_3\,(A + B\,(x_2 - x_1) + C\,(x_3 - x_2) + D\,(x_1 - x_3) + E\,(x_2 - x_1)^2 + F\,(x_3 - x_2)^2 + G\,(x_1 - x_3)^2 + \ldots \tag{9}$$

where Q_{123} represents V^E, ΔR, or $\Delta\kappa_s$ for the ternary mixture (ETBE + ethanol + [emim][EtSO$_4$]), Q_{ij} is the Redlich−Kister polynomial for the same property fitted to the binary systems data, being in this case Q_{13} equal to zero because of the immiscibility of ETBE and [emim][EtSO$_4$].

The A_k values for Q_{21} (ethanol + ETBE) and Q_{32} ([emim][EtSO$_4$] + ethanol) are summarized in Table 3 together with the corresponding standard deviations of fit. The parameters of the fitted polynomials for the binary (ethanol + ETBE) system were previously obtained by this group and have already been published,[13] except for the viscosity changes of mixing, which was included in this work. The Redlich−Kister coefficients for the ternary system are listed in Table 4 together with the corresponding standard deviations of fit. All of these coefficients were obtained by fitting, to the appropriate parameters, eqs 7 and 8 by least-squares regression. Fisher's *F*-test was used to decide the degree of the polynomial.

Conclusions

Excess molar volumes V^E and viscosity $\Delta\eta$, molar refraction ΔR, and isentropic compressibility $\Delta\kappa_s$ changes of mixing at 298.15 K were calculated for the binary system (ethanol + [emim][EtSO$_4$]) and also for the ternary system (ETBE + ethanol + [emim][EtSO$_4$]) from the measured densities, viscosities, refractive indices, and sound velocities. For the binary and

Table 4. Polynomial Coefficients and Standard Deviations σ Obtained for Fits of Equation 9 to the V^E, $\Delta\eta$, ΔR, and $\Delta\kappa_s$ Composition Data for the Ternary System ETBE (1) + Ethanol (2) + [emim][EtSO$_4$] (3)[a]

property	A	B	C	D	E	F	G	σ
$V^E/cm^3 \cdot mol^{-1}$	−67.7872	92.4374	−77.4518	−19.8907	−99.8499	−39.4061	40.1155	0.015
$\Delta\eta$/mPa·s	−341.36	300.21	−493.53	188.06	−31.69	−632.59	488.37	0.27
$\Delta R/cm^3 \cdot mol^{-1}$	1.7765							0.010
$\Delta\kappa_s/TPa^{-1}$	−574.75	−1552.68	791.30	749.66	5043.77	−2372.57	−3888.10	2.6

[a] For $\Delta\kappa_s$, system compositions were in volume fractions, ϕ_i.

ternary systems, over the miscible composition range, V^E values show negative deviations from ideal behavior. $\Delta\eta$ has negative and very large values due to the big differences between ionic liquid and other compounds viscosity. A minimum is found for the (ethanol + EMISE) binary system. Also $\Delta\kappa_S$ is negative over the miscible composition range. Values of molar refraction changes of mixing are so small that it is difficult to establish any conclusion. The Redlich−Kister equation was applied successfully for the correlation of all these excess properties.

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