



Density, viscosity and phase equilibria study of {ethylsulfate-based ionic liquid + water} binary systems as a function of temperature and composition



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ABSTRACT

This paper is a continuation of our investigation on physicochemical and thermodynamic properties of ionic liquids and its aqueous solutions. In this work the density, ρ and dynamic viscosity, η have been determined for binary mixtures of the ionic liquids: 1-ethyl-1-methyl-piperidinium ethylsulfate, [EMPIP][EtSO₄], 1-ethyl-1-methylmorpholinium ethylsulfate, [EMMOR][EtSO₄] and 1-ethyl-1-methylpyrrolidinium ethylsulfate, [EMPYR][EtSO₄] with water at wide temperature and composition range at atmospheric pressure. From experimental values of the density, ρ and dynamic viscosity, η the excess molar volumes, V^E and viscosity deviations, $\Delta\eta$ were calculated and correlated using Redlich–Kister polynomial equation. The (solid + liquid) phase equilibria, SLE for the tested binary mixtures have been determined by well-known dynamic method at a wide range of composition and temperature at atmospheric pressure. For comparison, the SLE data for {[EMPYR][EtSO₄] + water} binary mixtures have been determined using DSC technique. The experimental SLE data have been correlated by means of NRTL, UNIQUAC and Wilson equations. Additionally, the basic thermal properties of the pure ILs, that is, the glass-transition temperature, $T_{g,1}$ as well as the heat capacity at the glass-transition temperature, $\Delta C_{p(g),1}$, melting temperature, T_m and enthalpy of melting, $\Delta_m H$ have been measured using a differential scanning microcalorimetry technique (DSC). Decomposition of the ILs was detected by the simultaneous TG/DTA experiments. The choice of the ionic liquids allowed to determine the effect of cation structure on physicochemical and thermodynamic properties.

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1. Introduction

Due to the unique properties and large variety of applications in industry and applied chemistry, ionic liquids have attracted the attention of many research group around the world. The knowledge about physicochemical and the corresponding excess properties of the mixtures of ionic liquids with different solvents and their dependence on temperature and composition are very important from technological and industrial point of view. Moreover, the knowledge about the thermal and physical properties of ILs is extremely important in the design of ILs for different applications.

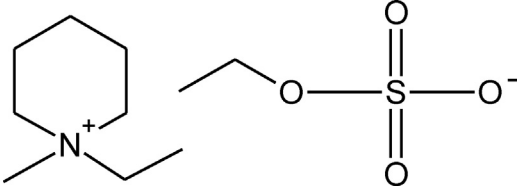
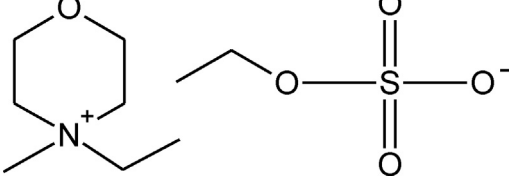
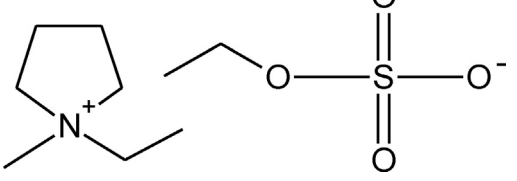
Although in the literature there are many information about physicochemical and thermodynamic study on binary mixtures of sulfate-based ionic liquids with various solvents [1–33] most of them are reported densities and viscosities of sulfate-based

ILs with alcohols [1–3,5,8–13,15–18,21,23,26,27,33] but just a few presented these properties for the binary mixtures of ethylsulfate-based ILs with water [1,2,6,12,16,24,25]. Moreover, more than a half of the ILs that have been studied based on 1-alkyl-3-methylimidazolium cation and there are only a few reports on sulfate-based ionic liquids with pyridinium [16,20,23], pyrrolidinium [20,28] and ammonium [28,29] cations. These data relate to the physicochemical properties of pure ionic liquids. According to the best of our knowledge, in the literature there are no data regarding the measurement of density, viscosity and phase equilibria for binary mixtures of ethylsulfate-based ILs with piperidinium, morpholinium and pyrrolidinium cation.

This work is a continuation of our systematic research on physicochemical and thermodynamic properties of {IL + water} binary mixtures [34–39]. The purpose of this work is to present the densities, dynamic viscosities and corresponding excess properties at wide range of temperature and composition as well as the phase equilibrium data for three binary mixtures of water and ethylsulfate-based ionic liquids:

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Table 1
Structures, abbreviations and names for the ionic liquids studied in this work.

Structure	Name, abbreviation
	1-Ethyl-1-methylpiperidinium ethylsulfate, [EMPIP][EtSO ₄]
	1-Ethyl-1-methylmorpholinium ethylsulfate, [EMMOR][EtSO ₄]
	1-Ethyl-1-methylpyrrolidinium ethylsulfate, [EMPYR][EtSO ₄]

1-ethyl-1-methylpiperidinium ethylsulfate, [EMPIP][EtSO₄], 1-ethyl-1-methylmorpholinium ethylsulfate, [EMMOR][EtSO₄] and 1-ethyl-1-methylpyrrolidinium ethylsulfate, [EMPYR][EtSO₄] and to discuss the effect of ILs structure on the presented properties.

2. Experimental

2.1. Materials

The ionic liquids: 1-ethyl-1-methylpiperidinium ethylsulfate, [EMPIP][EtSO₄], 1-ethyl-1-methylmorpholinium ethylsulfate, [EMMOR][EtSO₄] and 1-ethyl-1-methylpyrrolidinium ethylsulfate, [EMPYR][EtSO₄] were synthesized (with mass fraction purities higher than 97%) on special request by Liquid Technologies (Iolitec GmbH&Co. KG, Denzlingen, Germany). The water content, which has a significant impact on the IL properties, such as density, viscosity and phase equilibria measurements, was analyzed by Karl Fischer titration technique (model SCHOTT Instruments TitroLine KF). It was shown that the water content was less than 800 ppm for [EMPIP][EtSO₄], 1050 ppm for [EMMOR][EtSO₄] and 1200 ppm for [EMPYR][EtSO₄]. Ultrapure water was deionized by a reverse osmosis unit with an ion-exchange system (Cobrabid-Aqua, Poland) and

next degassed in an ELMA Germany ultrasonic bath at about 320 K before each measurement. The structures of the ionic liquids tested in this work are presented in Table 1. The specifications of the studied chemicals are presented in Table 2.

2.2. Methods and procedures

Binary mixtures of IL and water were prepared with uncertainty of $\pm 1 \times 10^{-4}$ g by mass using a high precision analytical balance (Mettler Toledo AB204-S). In order to minimize the variation in composition, due to evaporation or absorption of water by ionic liquid, each sample was prepared immediately before the experiment.

2.2.1. Differential scanning microcalorimetry (DSC)

Thermophysical characterization of pure ionic liquids, that is the glass transition temperature ($T_{g,1}$), the heat capacity at the glass-transition temperature ($\Delta C_{p(g),1}$), temperature (T_{fus}) and enthalpy of melting ($\Delta_{fus}H$) have been done using differential scanning microcalorimetry technique (DSC). The experiments were performed with DSC 1 STAR[®] System (Mettler Toledo) calorimeter equipped with liquid nitrogen cooling system and operating in a

Table 2
Specifications of chemical samples.

Chemical name	Source	Initial mass fraction purity	Purification method	Final mass fraction purity	Analysis method
1-Ethyl-1-methylpiperidinium ethylsulfate, [EMPIP][EtSO ₄]	Io-li-tec	>0.970	Vacuum heating	0.999	Karl–Fischer
1-Ethyl-1-methylmorpholinium ethylsulfate, [EMMOR][EtSO ₄]	Io-li-tec	>0.970	Vacuum heating	0.999	Karl–Fischer
1-Ethyl-1-methylpyrrolidinium ethylsulfate, [EMPYR][EtSO ₄]	Io-li-tec	>0.970	Vacuum heating	0.999	Karl–Fischer
Water	Own source	–	Reversed osmosis, ion exchange	0.9999	Density

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