

Short communication

Isotachophoretic separation of selected imidazolium ionic liquids

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Abstract

Results of determination of selected imidazolium ionic liquids by isotachopheresis (ITP) with conductometric detection was presented. The effects of the molar mass of different ionic liquids on electrophoretic mobility was observed. The presented method was validated and basic validation parameters were determined. Limit of detection (LOD) in a 10 and 25 ng/L for anions and cations, respectively, is very satisfied. Thanks to its low cost and high rate, the presented method can be used in qualitative routine analysis as an alternative technique to liquid chromatography. © 2007 Elsevier B.V. All rights reserved.

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1. Introduction

Room-temperature ionic liquids (RTIL), salts that are liquid at ambient temperature, are normally composed of relatively large organic cations and inorganic or organic anions. Unlike molecular liquids, ionic liquids, being polar solvents, are environmentally benign, nonvolatile, nonflammable; most of them have good solubility in water and are stable in air [1]. In addition, by varying the lengths and branching of alkyl chains of the anionic core and the cationic precursor, it is possible to design solvents for specific applications. Because of these characteristic properties, ionic liquids are widely used as new solvent media in heterogeneous catalysis, organic synthesis, electrochemistry, sensors, battery applications, analysis and separation [1,2]. Typical ionic liquids consist of an organic cation with delocalized charges and a small anion, most often weakly coordinating fluoroanions such as BF₄ or PF₆ [3]. Table 1 presents primary structures of the room-temperature ionic liquids. Millions of possible cation and anion combinations offer their widely tunable properties with regard to polarity, hydrophobicity and solvent miscibility behavior [3,4].

Currently, scientists are interested in the applications of ionic liquids in separation and analysis. Reports on ionic liquids used

as a modifier of background electrolytes (BGEs) in capillary electrophoresis (CE) appeared frequently [5–7]. Other scientists systematically studied the stationary phase of gas chromatography based on ionic liquids. Ionic liquids were used as additives in eluents to separate in high performance liquid chromatography (HPLC) [1,4,8] and countercurrent chromatography (CCC) [9].

Ionic liquids are more toxic xenobiotics and probably can be rating to persistent organic pollutants (POPs) [10]. From this point of view, analyses of ionic liquids in different parts of ecosystem (water, soils, sediments) are important and necessary. Therefore, the development of new analytical methods for the rapid and reproducible separation and identification of ionic liquids is a prerequisite for future biological and environmental research into these substances. Additionally, ionic liquids can yield photodegradation or biodegradation and analytical control of secondary products of these processes is also necessary.

Ionic liquids are already associated with the term “green” and “green chemistry”, but only because they have got no measurable vapour pressure, it means that they emit no volatile organic compounds (VOCs). Knowledge about their toxicological and ecotoxicological properties is deficient in spite of great importance from ecological point of view.

Isotachopheresis (ITP) with direct conductivity detection is useful and powerful method for high-sensitive determination of both anionic and cationic species in environmental analysis

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Table 1
Primary structures of the room-temperature ionic liquids

Quaternary ammonium salts $[R_yNH_{4-y}]^+X^-$	
Imidazolium salts $[RR'IM]^+X^-$	
Pyrolidinium salts $[RR'Pyr]^+X^-$	
Phosphonium salts $[R_yPH_{4-y}]^+X^-$	
Pirydinium salts $[RPy]^+X^-$	

R, R', alkyl substituent; X, anion (e.g. $AlCl_4^-$, BF_4^- , PF_6^- , Cl^- , Br^- , CH_3COO^- , CF_3COO^-); $y = 1, 2, 3, 4$.

of waters, wastewaters, sewage sludges and composts [11–20] and can be successfully used in ionic species (eg. ionic liquids) analysis.

The main aim of this study is to demonstrate the possibility of using a new isotachophoretic method to determination of selected room-temperature ionic liquids. The effects of the molar mass on different ionic liquids in the separation was observed. The methods quantitative analytical performance was satisfying and basic validation parameters were assigned.

2. Materials and methods

2.1. Instrumentation

An isotachophoretic analyzer EA 102 (Villa Labeco, Spis-ska Nova Ves, Slovakia) was used. This analyzer is provided with two FEP (fluoroethylenpropylene polymer) columns (a pre-separation one: $0.8\text{ mm} \times 90\text{ mm}$ and an analytical one: $0.3\text{ mm} \times 160\text{ mm}$), two contact conductometric detectors, a sample loop $30\text{ }\mu\text{L}$. Driving current in detection step was $50\text{ }\mu\text{A}$ in analytical column on both modes (anionic and cationic).

The isotachophoretic data were acquired and processed by an ITPPro 32 software (KasComp, Bratislava, Slovakia) installed on a Pentium computer.

2.2. Chemicals

Ammonium acetate was obtained from P.O.Ch. (Gli-wice, Poland). Hydroxyethylcellulose, polyethylene glycol 600, tetrabutylammonium perchlorate were purchased from Sigma–Aldrich (Steinheim, Germany). All chemicals utilized for electrolytes, standard solutions, and sample preparation were pure per analysis (*p.p.a.*) class.

Deionised water was used from Milli-Q RG (Millipore, Mol-sheim, France).

The ionic liquids selected for these studies were obtained from the working group of Dr. Piotr Stepnowski (Faculty of Chemistry, Waste Management Laboratory, University of Gdańsk, Poland).

3. Results and discussion

Table 2 presents basic physicochemical properties of selected ionic liquids used in isotachophoretic experiments [21]. Five from them have got the same anion (BF_4^-) and different homol-ogous imidazolium cations:

- PMIM (1-*n*-propyl-3-methyl-imidazolium cation),
- BMIM (1-butyl-3-methyl-imidazolium cation),
- AMIM (1-*n*-amyl-3-methyl-imidazolium cation),
- HMIM (1-*n*-hexyl-3-methyl-imidazolium cation),
- OMIM (1-*n*-octyl-3-methyl-imidazolium cation).

These abbreviations should be used in whole text.

Additionally, in experiments we used one ionic liquid with chloride anion.

Table 3 presents composition of leading (LE) and terminating (TE) electrolytes used to separate ionic liquids by isotachophoresis. Isotachophoresis worked in cationic and anionic analysis mode. Electrolytic systems were known earlier, but in this study we optimize driving current.

3.1. Cationic mode

Isotachophoresis working in cationic mode enables identifi-cation of cationic core of room-temperature ionic liquids. Fig. 1 shows results of identification of ionic liquids in model mixture using conductivity detection.

In Fig. 1, steps from all analytes are sharp and clear. Con-ductivity detection enables identification of [PMIM], [BMIM], [AMIM], [HMIM] and [OMIM], relative step heights (RSH) (parameter for qualitative analysis in isotachophoresis) of these cations are for [PMIM] 0.369, for [BMIM] 0.418, for [AMIM] 0.476, for [HMIM] 0.527 and for [OMIM] 0.626.

The advantage of conductivity detection is small error assign of RSH value. Standard deviation (S.D.) 0.023, 0.035, 0.03, 0.025 and 0.028 for [PMIM], [BMIM], [AMIM], [HMIM] and [OMIM], respectively, is admissible. Additionally, signal from conductivity detection is easy to interpretation.

In results of experiments we find interesting correlation between molar mass M_r of the RTIL and RSH value (Fig. 2).

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