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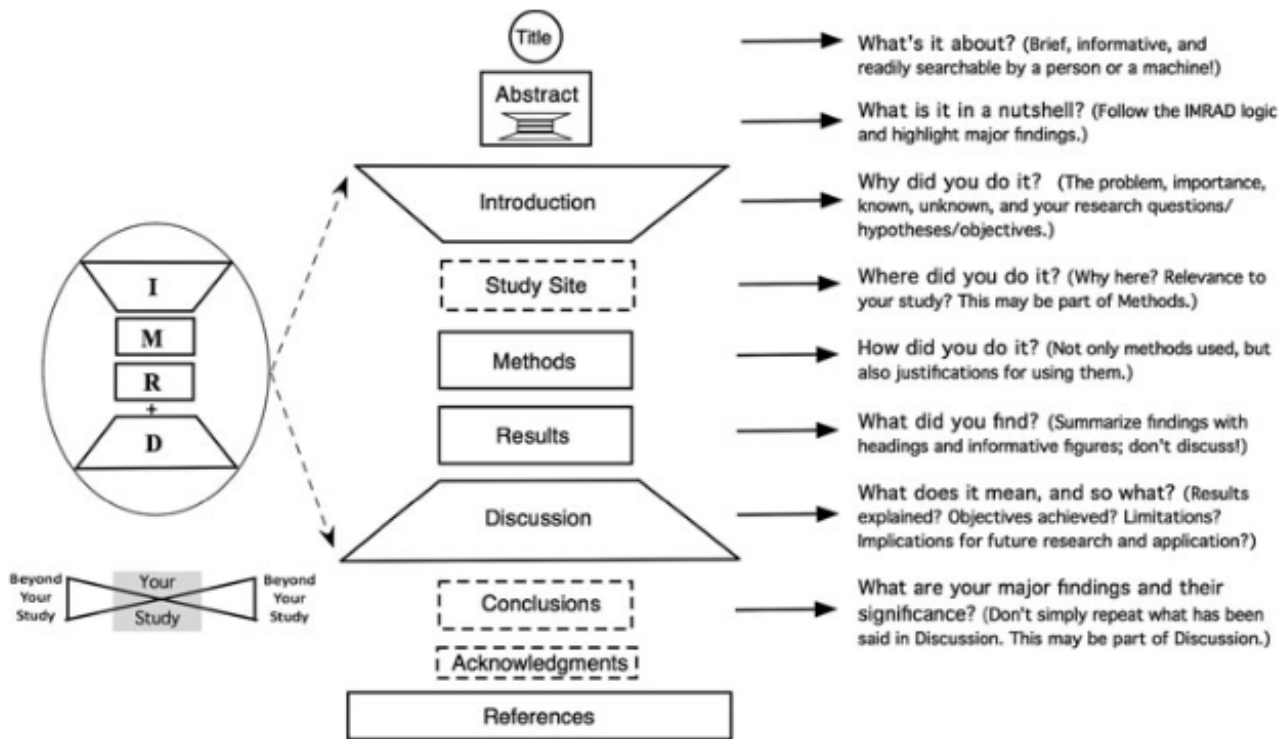
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Article

Ionogels in Aqueous Media: From Conductometric Probing of the Ionic Liquid Washout to the Design of More Stable Materials

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Abstract: Although the most promising applications of ionogels require their contact with aqueous media, few data are available on the stability of ionogels upon exposure to water. In this paper, a simple, easy-to-setup and precise method is presented, which was developed based on the continuous conductivity measurements of an aqueous phase, to study the washout of imidazolium ionic liquids (IL) from various silica-based ionogels immersed in water. The accuracy of the method was verified using HPLC, its reproducibility was confirmed, and its systematic errors were estimated. The experimental data show the rapid and almost complete (>90% in 5 h) washout of the hydrophilic IL (1-butyl-3-methylimidazolium dicyanamide) from the TMOS-derived silica ionogel. To lower the rate and degree of washout, several approaches were analysed, including decreasing IL content in ionogels, using ionogels in a monolithic form instead of a powder, constructing ionogels by gelation of silica in an ionic liquid, ageing ionogels after sol-gel synthesis and constructing ionogels from both hydrophobic IL and hydrophobic silica. All these approaches inhibited IL washout; the lowest level of washout achieved was ~14% in 24 h. Insights into the ionogels' structure and composition, using complementary methods (XRD, TGA, FTIR, SEM, NMR and nitrogen adsorption), revealed the washout mechanism, which was shown to be governed by three main processes: the diffusion of (1) IL and (2) water, and (3) IL dissolution in water. Washout was shown to follow pseudo-second-order kinetics, with the kinetic constants being in the range of $0.007\text{--}0.154\text{ mol}^{-1}\text{s}^{-1}$.

Keywords: ionogels; sol-gel; aerogels; silica; confined ionic liquids; conductivity; hydrophobicity



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1. Introduction

Ionic liquids (ILs) are salts with a melting point below 100 °C or near room temperature [1]. They usually comprise a large and highly asymmetric organic cation and a relatively small organic or inorganic anion. The chemical composition of ionic liquids hinders their crystallisation upon cooling and provides them with a set of unique properties [1,2]: high ionic conductivity [3], wide electrochemical stability windows [4], extremely low vapour pressures [5], high thermal stability [6] and great dissolving abilities for a wide range of ionic and molecular substances [7]. These characteristics make ionic liquids excellent solvents [8], electrolytes [9], catalysts and reaction media [10], extractants [11,12], lubricants [13], etc. However, ILs are generally expensive, relatively hard to synthesise and purify, and often toxic to the environment [14,15]. The latter drawback is exacerbated by a high level of solubility in aqueous media or industrial liquors of poorly soluble ionic liquids [16,17], resulting in uncontrolled and non-recoverable losses of high-cost and often harmful substances [18].



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