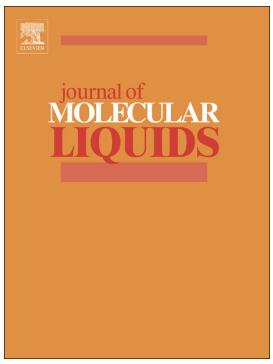

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Glycerol-based ionic liquids: crucial microwaves-assisted synthetic step for solketal amines

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Keywords
Ionic liquids; glycerol; microwaves; solketal amine; meerwein salt.

Abstract
Glycerol-based ionic liquids were prepared by alkylation of solketal amines. These ones were easily obtained from tosyl solketal compound through a microwaves assisted amination without using solvent. This crucial step allowed the formation of various solketal amines in 50 minutes at 85°C with high yields. The alkylation with Me_3OBF_4 led next to ionic liquids with BF_4^- as anion presenting a good thermal stability.

Introduction

Ionic liquids (ILs) are ionic and salt-like materials which are liquid below 100 °C. Their numerous properties make them an attractive choice of solvent in many important chemical processes [1]. ILs are of great interest in (bio)catalysis [2], organic synthesis [3], electrochemistry [4] or for the metallic extraction [5]. Appropriate reviews detailed recently the advantages of using ILs as new solvents in various fields [6].
However, ILs are nor green solvent considering thermodynamic and physical behaviours as mentioned in different publications of Marlair's group [7]. Indeed, their low biodegradability (or the toxicity of their degradation products) and their high (eco)toxicity led the scientific community to reduce their use or to find other greener alternatives [7]. Due to their biodegradability and non-toxicity, the use of renewable resources such as amino acids, amino alcohols or sugars, could improve the green character of ILs [8].
Our group developed a few years ago various ILs based on ammonium or phosphonium with anion from natural acids (*L*-lactic, *L*-tartaric, pyruvic, malic, malonic, succinic and osidic acids), but also from *L*-proline and its derivatives. Even if they were not readily biodegradable, these compounds showed in general lower toxicity towards various organisms than usual chlorinated and commercial ILs [9a,b,c].
In this present study, the starting material is the glycerol and more precisely a derivative of glycerol, the solketal.
Glycerol is used extensively in cosmetics, toiletries and pharmaceutical products. In a closed bottle test (performed according to OECD 301), 92% biodegradation was reported after 30 days. Furthermore, glycerol is of a low order of acute oral and dermal toxicity with LD50 values in excess of 4000 mg/kg [10].
Due to the high growth of biodiesel production, glycerol, a major by-product, is produced at the same growing rate, resulting in its oversupply [11]. So it needs to be valorized towards various purposes, as solvents for example [12].