Identification of novel chemical structures of sulfo-imidazolium zwitterionic-type salt basis on 2D NMR analysis

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ABSTRACT

The reaction of the equimolar mixture of imidazole and 1,4-butanesultone was performed at 90°C in ethylbenzene for 12 h and the zwitterionic and ionic structures of the product investigated by the NMR analysis. The present work displayed the great importance of 2D NMR analysis on the structure elucidation of this sulfonic acid functionalized liquid salt. The results of NMR analysis demonstrate that 4-imidazole-1-yl-butane-1-sulfonic acid did not exist and really a mixture of zwitterionic and ionic compounds is produced.

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1. Introduction

Ionic liquids (ILs) are widely utilized in the multicomponent reactions as the solvent, catalyst or dual solvent-catalyst due to unique physicochemical and environmentally benign properties [1–9]. The organic products and the ionic liquids can be separated in the different phases and this can decrease the consumption of toxic and volatile organic solvents and avoid tedious and complex separation and purification technologies [10,11].

The imidazolium cations containing covalently-bound anionic sulfonate groups were synthesized through the equimolar reaction of N-allylimidazole and 1,3-propanesulfonate or 1,4-butanesulfone in acetone, and the NMR spectra data were presented [12,13]. Firstly, Hajra et al. showed a structure of the product of the reaction of imidazole with 1,4-butanesulfone and named it as 4-(1-imidazolium)butane sulfonate which was used in one-pot synthesis of β-nitroanilines [14]. 4-(1-Imidazolium)butane sulfonate was used in a three-component synthesis of 2-amidoalkyl and 2-carbamatesalkyl naphthols [15], and the mechanism reaction was illustrated using a chemical structure of 4-(1-imidazolium)butane sulfonate as previously reported in the literature [14]. Then, this compound was used as an organocatalyst for the synthesis of tetrazoles [16] and the regioselective ring-opening of aziridines [17]; nevertheless, no spectroscopic and physical data for the claimed structure entitled 4-(1-imidazolium)butane sulfonate was exhibited. Furthermore, this compound is observed in the chemical list of the chemical supplier “Accel Pharmtech” as 1H-imidazole-1-butanesulfonic acid with CAS No: 1094601-09-8 [18].

To the best of our knowledge, no spectroscopic and physical data were displayed for this zwitterionic-type salt in the literature. We interested in this reaction and its product when we observed the unusual 1D NMR of product, and proposed the equilibrium structures. We repeated the reaction several times, but the results were identical. On the other hand, the proposed equilibrium structures did not convince us; thus we used 2D NMR to describe the unusual 1D NMR and excess peaks. It was approved that the reaction of imidazole and 1,4-butanesulfone could not produce 4-(1-imidazolium)-butane sulfonate.

2. Experimental

2.1. Materials and methods

Unless specified, all chemicals were analytical grade and purchased from Merck, Aldrich, and Fluka Chemical Companies and used without further purification. Products were characterized by their physical constant and FT-IR, NMR and elemental analysis. The purity determination of the substrates and reaction monitoring were accompanied by TLC using silica gel 60 G/UV 254 plates. The purity determination and MS (EI) data of the products were accomplished by GC-MS on an Agilent 6890N GC system with a 5973N mass selective detector under 70 eV conditions. The FT-IR