A Facile Synthesis of Novel 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate Salts

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Abstract: Quaternary ammonium salts are used extensively as precursors of photochromic spiropyrans, RNA antagonists, various cyanine dyes. Alkoxymethylene-N,N-dimethyliminium salts, the adduct fromed from DMF and N-alkylating agents such as dialkylsulphates were used for quaternization of benzazole deriviatives which results the formation quaternary ammonium methosulphate salts. These salts were used under green conditions to condense with various N-alkyl-indole-3-carbaldehydes resulted formation of novel 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate salts.

Keywords: 2-Methylbenzimidazole, Dimethylsulphate, Dimethylformamide.

1. Introduction

Quaternary Ammonium Salts (QAS) play an important role in the living organisms and many functions of prokaryotic and eukaryotic cells have been shown to be alkyl ammonium salt dependant [1,2]. Despite the fact that several N-alkyl quaternary ammonium salts bearing a 2-methylbenzothiazole, -benzoselenazole, -benzoxazole, -indole, -quinoline and –quinazoline nuclei are easily found in the literature, mainly as unisolated precursors of cyanine derivatives [3,4].

2. Experimental (or Theoretical)

Alkoxymethylene-N,N-dimethyliminium salt, the adduct formed from DMF and N-alkylating agent such as dimethylsulphate was used for the quaternization of 2-methylbenzimidazole derivatives which results the formation of 1,2,3-trimethyl-1H-benzo[*d*]imidazol-3-ium methosulphate salt. These quaternary benzimidazolium salts were condensed with various N-alkyl-Indole-3-carbaldehydes under green conditions resulted formation of novel 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate salts.

3. Results and discussion

Introduction Alkoxymethylene-N,N-dimethyliminium salts were prepared by the reaction of DMF and alkylating agents such as dimethylsulphate and diethylsulphate respectively under Microwavw assisted synthesis about 2 minutes. The obtained methosulphate salts were added with equal amounts of N-alkyl-indole-3-carbaldehyde using ethanol as solvent and L-proline as catalyst.

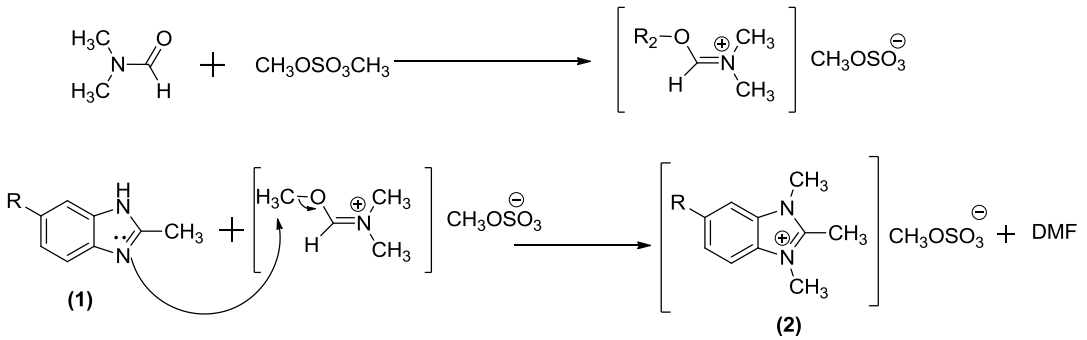

Figure 1. Synthesis of methoxymethylene-N,N-dimethylimethylbezimidazolium methosulphate salts.

Experimental Section Melting points are uncorrected and were determined in open capillary tubes in sulphuric acid bath. TLC was performed on silicalgel-G and spotting was done using iodine or UV light. IR

spectra were recorded using Perkin-Elmer 1000 instrument in KBr phase. 1H NMR on VARIAN 400 MHz instrument and mass spectra on Aglient-LC-MS instrument giving only $M^{+\cdot}$+1 and $M^{+\cdot}$-1 values.

Synthesis of quaternary salts (Microwave method): A solution of DMS (0.03 mol) in dry DMF (10 ml) was prepared by taking in a 50 mil Erlenemeyer Flask and stirred well for 10 min. To this compound **1** (0.01 mol) was added and subjected to microwave irradiation at 450W level for a period of 2 Min. The reaction mixture was cooled to RT and allowed to stand for 5 min at RT. The separated crystals were filtered, washed with cold ether and dried to obtain crude **2**.

Synthesis 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate Salts: To the compound **2** (0.01 mol) in ethanol (10 ml), compound **3** (0.01 mol) added in a round bottom flask and catalytic amount of L-Proline was added. The reaction mixture was subjected to microwave irradiation at 450W level for a period of 2 Min. The completion of the reaction was monitored by checking TLC. The reaction mixture was cooled to RT and allowed to stand for 5 min at RT. The separated solid was filtered and dried to obtain pure **4**.

Results and discussion

Methoxy-methylene-N,N-dimethyliminium methosulphate salts were prepared by the adduct formed with DMF which was then treated with N-alkyl-2-methylbenzimidazole under microwave irradiation method. The obtained salts were condensed with N-alkyl-indole-3-carbaldehyde under various green conditions such as Microwave irradiation method using ethanol as solvent and L-proline as catalyst. The reaction was also done using green reaction mediums like PEG-600 under both conventional and Microwave irradiation methods.

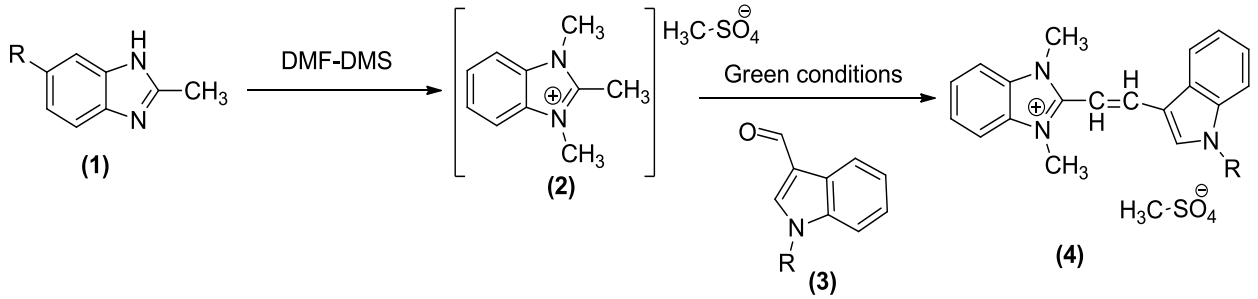

Figure 2. Synthesis of 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate salts.

4. Conclusions

To the best of our knowledge, the benzimidazolium methosulpahe salts have not been extensively used for the synthesis of target compounds, namely 1,3-dimethyl-2-(2-(1-alkyl-1H-indol-3-yl)vinyl)-1H-benzo[*d*]imidazol-3-ium methosulphate salts by microwave irradiation methods and other green conditions. The high yields were obtained in less time and temperatures. The collection of the product and purification is also simple which makes the method more convenient and feasible to obtain the desired target compounds.

References

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