

A Facile and Greener Way of Glycine Catalyzed Novel Synthesis of Triarylimidazoles- Synthesis and Characterization

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Abstract : The facile and green way to synthesis triaryl imidazole(s) was discussed. The glycine has been selected as a catalyst. The chemical structures of these compounds were identified by UV, FT-IR, ¹H NMR and Mass spectral techniques. The progress of the reaction time, purity was checked by TLC. Results of the bacterial studies reveal that the triaryl substituted compounds have considerable antibacterial activity in comparison with ciprofloxacin as model drug.

Key words: Imidazole-spectral studies- green synthesis- anti bacterial activity.

I. INTRODUCTION

Developing cleaner, safer and environmentally friendly chemical processes is an important goal for chemists in both academia and industry ^[1]. It is no longer acceptable to make products without being concerned about environmental pollution. Several strategies have been developed based on the idea that it is not only important what is produced but also how it is produced. To remove organic solvents from chemical processes, many old reactions have been revised and carried out in water ^[2], under solventless conditions ^[3], in supercritical fluids ^[4], in ionic liquids ^[5] However, a meager have been observed in amino acid catalyzed reactions . Based on the careful analysis of literature, the scope of the present investigation involves the glycine catalyzed reaction for the synthesis of triaryl substituted imidazole as starting material for the development of water soluble imidazole containing the antimicrobial polymers. On the outset the first stage have been synthesized and characterized.

II. EXPERIMENTAL

The chemicals benzil (1), ammonium acetate (2), Salicylaldehyde (3), para-Chlorobenzaldehyde (4), para-Methoxybenzaldehyde (5), and glycine were commercially available from Merck and Avra chemicals, ¹H NMR (300 MHz) spectra were recorded on a Bruker Advance III 300 MHz multi nuclei solution NMR. FTIR spectra (KBr pellets) were measured on the Alpha Bruker FTIR instrument scanning the entire region of 4000 - 400 cm⁻¹ with typical resolution of 1.0 cm⁻¹. Mass spectroscopy was recorded on ES-FIGIEAN ionization mass spectrometer.

A. Synthesis of 2-(2-Hydroxyphenyl)-4,5-diphenyl-1H-imidazole (6)

A mixture of benzil (0.525g; 2.5mmol), Salicylaldehyde (0.3053g;2.5mmol), ammonium acetate (0.5g;6mmol) and amino acid (0.05g) was stirred in ethanol (10ml) for 48 hours at room temperature. The completion of the reaction was monitored by TLC.

B. Synthesis of 2-(4-Chlorophenyl)-4,5-diphenyl-1H-imidazole (7)

A mixture of benzil (0.525g; 2.5mmol), para-Chlorobenzaldehyde (0.35g;2.5mmol), ammonium acetate (0.5g;6mmol) and amino acid (0.05g) was stirred in ethanol (10ml) for 48 hours at room temperature. The completion of the reaction was monitored by TLC.

C. Synthesis of 2-(4-methoxyphenyl) 4,5-diphenyl-1H-imidazole (8)

A mixture of benzil (0.525g; 2.5mmol), paramethoxybenzaldehyde (0.340g;2.5mmol), ammonium acetate (0.5g;6mmol) and amino acid (0.05g) was stirred in ethanol (10ml) for 48 hours at room temperature. The completion of the reaction was monitored by TLC.

III. RESULTS AND DISCUSSION

The condensation of equimolar mixture of benzil and substituted benzaldehyde along with ammonium acetate were carried out at 80 °C in the presence of glycine as catalyst for ten minutes to get triaryl substituted imidazoles. Hence, the present investigation was very much clear enough that the presence of the catalytic amount of glycine under solvent free condition resulted excellent yield.

2-(2-Hydroxyphenyl)-4,5-diphenyl-1H-imidazole(6)

FTIR (cm⁻¹) : Aromatic (3061),N-H(3316)
¹H NMR (ppm) : NH-proton (13.0), Aromatic 6.7-8.3 (m,15H),
Mass (m/z) : Calculated M.W. 312.75, Observed M.W 313.0
M.pt : 202- 204⁰C

2-(4-Chlorophenyl)-4,5-diphenyl-1H-imidazole(7)

FTIR (cm⁻¹) : Aromatic (3060, 3028),N-H (3318).
¹H NMR (ppm) : NH-proton-(s,14.4),Aromatic 6.6-7.9 (m,15H).
Mass (m/z) : Calculated M.W 330.827, Observed M.W 330.9
M.pt : 250- 253⁰C

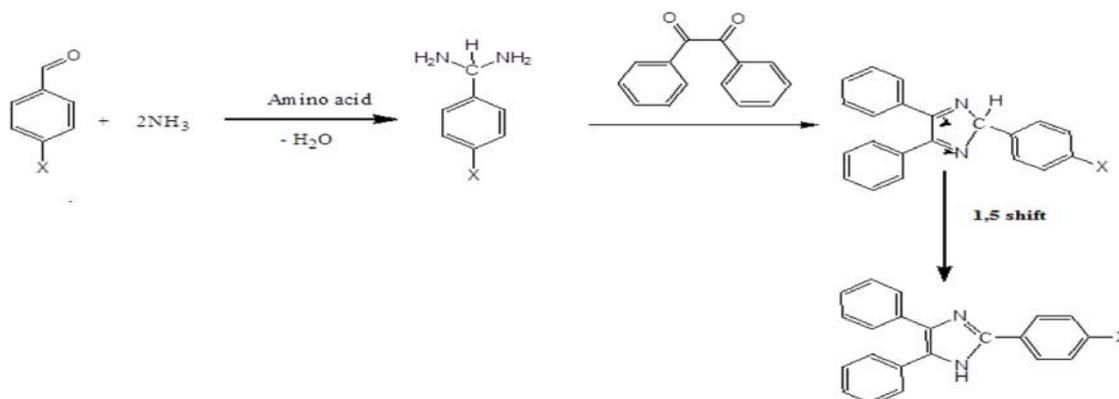
2-(4-methoxyphenyl) 4,5-diphenyl-1H-imidazole (8)

FTIR (cm⁻¹) : Aromatic (3060, 3002, 2956) , N-H (3316)
¹H NMR (ppm) : NH-proton(s)(13.9), Aromatic Proton (m,15H)
Mass (m/z) : Calculated M.W 326.402, Observed M.W 327.0
M.pt : 253- 255⁰C

FTIR provided a preliminary idea in confirmation the formation of product. Absence of peak at 1730 cm⁻¹ clearly generates the utilization of starting materials transforms into the product. Further the corresponding peaks at 3314, 3318 and 3316 cm⁻¹ for N-H stretching in respect of compound 6, 7 and 8 respectively. All such stretching and bending peaks have also been supported for the formation of the product.

Similarly, proton NMR strongly empowered for the formation of the product by its value at 13.0, 14.4 and 13.9 corresponding to the N-H protons of compound 6, 7 and 8 respectively. The concern mass of compound 6, 7, 8 are in good agreement with the observed (313, 330.9, 327.0) and calculated values (312.75, 330.75, 326.402) respectively .

Mechanism:



Scheme. 1 Synthesis of triaryl substituted Imidazole

Substituted benzaldehydes on treatment with two moles of ammonia (obtained from heating of ammonium acetate) in the presence of glycine as a green catalyst resulted sp^3 hybridized diamines. Which on further treatment with benzil on continuous refluxing undergoes 1,5 shift followed by cyclization resulted triaryl substituted imidazoles (Scheme. 1).

IV. Biological activity

The biological activity of the three triaryl substituted imidazoles(s) was subjected to three representative numbers of pathogenic organism's viz., *E.coli*, *S.aureus*, and *Bacillus* respectively. The monitoring of antibacterial activity is usually performed by the determination of the MIC, the smallest amount of the agent that inhibits the multiplication of the pathogen. It is found that the MIC for compound **6** having certain activity against the pathogens than compound **7** and **8** is presented in Table 1. Compound **6** has excellent activity than the model drug ciprofloxacin against all the pathogens of present investigation. However, compound **7** has mild activity against *Bacillus* while comparing with model drug whereas no zone of inhibition was observed for compound **7** and **8** beside *E.coli* and *S.aureus*.

V. CONCLUSIONS

In the present work, three aromatic aldehydes were used to check the practicability of amino acid especially neutral amino acid (glycine) catalyzed synthesis of triaryl substituted imidazole and characterized using FTIR, NMR and Mass techniques. The required stretching and bending peaks along with NMR and Mass have been suggested for the formation of the products. The general mechanism for the formation of the product has also been recommended. Among the three aldehydes, salicylaldehyde based **6** has superior antibacterial properties than the ciprofloxacin used as a model drug. The yields are comparable. Hence, the glycine may strongly be suggested as green catalyst in the synthesis of imidazoles derivatives. The catalyst with various diketones and aldehydes has also been accomplished to enhance the scope of utilization of the green catalyst.

ACKNOWLEDGMENTS

One of the authors, **Dr.S. Guhanathan**, gratefully acknowledge University Grants Commission, Government of India, New Delhi for financial support [Major Research Project: F. No. 40-50 / 2011(SR)].

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