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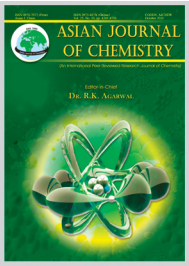

Synthesis and Antibacterial Activity of Novel Hydrazones Derived from 4,5-Diazafluoren-9-hydrazone

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An environmentally benign series of novel hydrazones were synthesized by condensation of 4,5-diazafluoren-9-hydrazone with various aldehydes and ketones by using acetic acid as a catalyst under solvent free condition. This protocol gives excellent yield of the products. All synthesized products were characterized by IR, NMR, mass and also tested for antibacterial (*Escherichia coli*, *Staphylococcus aureus*, *Bacillus subtilis* and *Klebsiella pneumonae*) activities by disc diffusion method.

Key Words: 4,5-Diazafluoren-9-hydrazone, Grinding, Acetic acid, Hydrazones, Antibacterial.

INTRODUCTION

Hydrazone is an important class in organic chemistry. These compounds have interesting biological activities such as antimycobacterial[1], antimicrobial[2-4], antituberculosis[5], anti-tumorals[6], antiinflammatory[7,8], antimalarial[9], anticonvulasnt[10] and anticancer-anti HIV[11].

At present, a broad range of methods for synthesis of hydrazones were reported such as microwave irradiation [polystyrene sulfonic acid][12], ultrasound irradiation[13], Ball-mill process[14], grinding method [PTSA][15] and also by refluxing method[16-18]. But these known methods of hydrazones synthesis suffers from one or other limitations such as harsh reaction conditions, expensive reagents, low yields and relatively long reaction time.

Because of that the researcher still continuous to synthesize novel hydrazones with better methodology in terms of simplicity, eco-friendly and economic viability which is achieved by using acetic acid. Thus, in this article, we report the synthesis of novel hydrazones derived from 4,5-diazafluorene-9-hydrazone in acetic acid as a catalyst by grinding method. Synthesis of organic compounds by grinding method has the advantages of shorter time, higher yield, mild reaction condition as well as being environmentally friendly[19]. Thus grinding method comes under the title of green chemistry. This synthesized hydrazones are also assayed for antibacterial activities.

EXPERIMENTAL

Melting points of the synthesized compounds were determined in open-glass capillaries on a stuart-SMP10 melting point apparatus and are uncorrected. IR absorption spectra were recorded on a Perkin Elmer 1650 FTIR using KBr pellets in the range of 4000-450 cm^{-1}. ^1H NMR was recorded on a Bruker spectrometer operating at 300 MHz. The ^1H NMR chemical shifts are reported as parts per million (ppm) downfield from TMS (Me_4Si) used as an internal standard. Mass spectra were recorded on LCQ ion trap mass spectrometer. Purity of the compounds were checked by thin layer chromatography (TLC) on Merck silica gel 60 F_{254} pre-coated sheets in benzene/methanol mixture and spots were developed using iodine vapour as visualizing agents.

General procedure

Synthesis of 4,5- diazafluoren-9-one (2): The 4,5-diazafluoren-9-one was synthesized using method reported in the literature[20].

Synthesis of 4,5-diazafluoren-9-hydrazone (3): A mixture of 4,5-diazafluoren-9-one (1 mmol) and hydrazine hydrate (1.1 mmol) was crushed in mortar with a pestle at room temperature and acetic acid (20 mol %) was added and crushed then the progress of reaction was monitored by TLC. After completion of reaction (3 min) the crude product was washed with water, dried and purified by column chromatography.

Synthesis of hydrazones on grinding (4a-o and 5a-e): A mixture of 4,5-diazafluoren-9-hydrazone (1 mmol) and aldehyde/ketone (1 mmol) was crushed in mortar with a pestle at room temperature and acetic acid (20 mol %) was added and crushed then the progress of reaction was monitored by TLC. After completion of reaction (3 min) the crude product