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Synthesis, Spectral Studies and Anti-Microbial Activity of Novel Chalcones of 2-Acetyl Pyridine

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Abstract: ***Some new chalcones have been synthesized by the condensation of 2-acetyl pyridine with different aromatic aldehydes in 40% alkali. The synthesized compounds were identified by Physical data and Spectral data (IR, ^{1}HNMR). These Synthesized derivatives of Chalcones Screened for Antimicrobial Activities. Some of these compounds showed moderate to considerable anti-microbial activity.***

Keywords: Chalcones, Synthesis, Physical data, Spectral data, Anti-microbial activity.

I. INTRODUCTION

Chalcones are synthesized by Claisen – Schmidt condensation of aldehyde and ketone by base catalyzed followed by dehydration to yield chalcones [1]. The synthesis of chalcone compounds incorporating with heterocyclic become the great importance in medicinal chemistry (2, 3). The hetero atom products variety of application in the biological engineering and in other field of their specific structure (4). To the best of our knowledge acetyl pyridine involving different substituted aldehyde under basic condition reaction is unprecented. Incontinuation of our interest to developing novel synthetic methodologies and use of chalcones for organic synthesis. The compounds with the backbone of chalcones have been reported to possess various biological activities such as antimicrobial [5], anti-inflammatory [6], analgesic [7], antiulcerative [8], antimalarial [9], anticancer [10], antiviral [11] and antioxidant [12] activities. Antifungal activity of chalcones has been investigated by a number of researchers [13]. The present work indicates that, when a pyridine ring was incorporated into a chalcone structure, the molecule exhibited antifungal activity [15].

II. MATERIAL AND METHODS

The melting point of the compounds were determined in open capillaries, using Eligo digital melting point apparatus and Melting points were determined on a capillary melting point apparatus and are uncorrected expressed in degree Celsius and the values were uncorrected. IR spectra of the compounds were recorded on Shimadzu 8201 spectrophotometer using KBr and the values are expressed in 4000-400 cm-1. 1H and 13C NMR spectra were recorded on Bruker AV 400 MHz Spectrophotometer using TMS as an internal standard and the values are expressed in δ ppm. All the solvents used were analytical grade. The purity of the compound was checked by TLC using silica gel plates.

A. General Procedure for the Preparation of chalcones (1q-1x)

Equimolar quantity of 2-Acetylpyridine (0.01 mol) and substituted aromatic aldehyde (0.01 mol) were dissolved in 20 ml of ethanol was heated about 60^{o}C. In solution, 10 ml of 40% Sodium hydroxide solution was added drop wise. The reaction mixture was magnetically stirred for 1h. Allow the solution to cool and acidify with dil. HCl. A flocculent precipitate was formed. The precipitate was filtered and washed with cold water and recrystallize from ethanol.

III. RESULT AND DISCUSSION

Physical data of compounds (1q-1x) are obtained and given in following table.

Compound	Molecular Formula	Melting Point ^{o}C	Percentage Yield
1q	$C_{14}H_{10}OClN$	167	83
1s	$C_{14}H_{10}OBrN$	140	77
1t	$C_{14}H_{10}ONF$	146	85
1u	$C_{15}H_{13}O_2N$	126	80
1v	$C_{16}H_{15}O_2N$	110	82
1w	$C_{14}H_{10}O_3N_2$	184	80
1x	$C_{14}H_{13}ON$	120	96

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Spectral data of the compounds (**1q-1x**) are obtained using various a spectral methods. The results discussed are given below.

1q - (2*E*)-3-(4-chlorophenyl)-1-(pyridin-2-yl) prop-2-en-1-one:

IR (KBr) cm-1: 3527 (Ar C-Hstr), 2924 (C-Hstr pyridine), 2854(C-Hstr alkene), 1743 (C=Ostr), 1651(C=Cstr and 677 (C-Clstr).

1H NMR (DMSO) ppm; 7.1-7.6 (Ar), 7.7 (Hβ =CH-Ar), 6.6 (Hα –CO-C=s).

1s - (2*E*)-3-(4-bromophenyl)-1-(pyridin-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 3446 (ArC-Hstr), 2924 (C-Hstr pyridine), 2854(C-Hstr alkene), 1745 (C=Ostr), 1649 (C=Cstr), 671 (C-Brstr) and 889 (C-Hdef).

1H NMR (DMSO) ppm; 7.0-7.6 (Ar), 7.7 (Hβ =CH-Ar), 6.8 (Hα –CO-C=).

1t - (2*E*)-3-(4-flurophenyl)-1-(pyridin-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 3446 (ArC-Hstr), 2924 (C-Hstr pyridine), 2854(C-Hstr alkene), 1745 (C=Ostr), 1649 (C=Cstr), 671 (C-Brstr) and 889 (C-Hdef).

1H NMR (DMSO) ppm; 7.0-7.6 (Ar), 7.7 (Hβ =CH-Ar), 6.8 (Hα –CO-C=).13CNMR (CDCl3) ppm: 127-142 (Ar), 181 (-CO-), 145(Cβ),122 (Cα).

1u - (2*E*)-3-(4-methoxyphenyl)-1-(pyridine-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 3454 (ArC-Hstr), 2922 (C-Hstr pyridine), 2854(C-Hstr alkene), 1743 (C=Ostr), 1649 (C=Cstr), 979 (C-Sstr), 734 (C-Hdef), and 675 (C-Clstr).

1H NMR (DMSO) ppm; 8 (Hβ =CH-Ar), 7.3-7.7 (Ar), 7.2 (Hα –CO-C=).

1v - (2*E*)-3-(4-ethoxyphenyl)-1-(pyridin-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 3454 (ArC-Hstr), 2922 (C-Hstr pyridine), 2854(C-Hstr alkene), 1743 (C=Ostr), 1649 (C=Cstr), 979 (C-Sstr), 734 (C-Hdef), and 675 (C-Clstr).

1H NMR (DMSO) ppm; 8 (Hβ =CH-Ar), 7.3-7.7 (Ar), 7.2 (Hα –CO-C=).

1w– (2*E*)-3-(4-Nitrophenyl)-1-(pyridin-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 2924 (C-Hstr pyridine), 2854(CHstr alkene), 1743 (C=Ostr), 1635 (C=Cstr), 974 (C-Sstr), 889 (C-Hdef) and 731(C-Hdef).

1H NMR (DMSO) ppm; 7.8 (Hβ =CH-Ar), 7.0-7.7 (Ar), 6.9 (Hα –CO-C=).

1x - (2*E*)-3-(4-methoxyphenyl)-1-(pyridin-2-yl) prop-2-en-1-one

IR (KBr) cm-1: 3454 (ArC-Hstr), 2922 (C-Hstr pyridine), 2854(C-Hstr alkene), 1743 (C=Ostr), 1649 (C=Cstr), 979 (C-Sstr), 734 (C-Hdef), and 675 (C-Clstr).

1H NMR (DMSO) ppm; 8 (Hβ =CH-Ar), 7.3-7.7 (Ar), 7.2 (Hα –CO-C=).

IV. ANTIMICROBIAL SCREENING

A. Antibacterial Activity

The purified products were screened for their antibacterial activity by using disc diffusion method. The nutrient agar broth prepared by the usual method, was inoculated aseptically with 0.5 ml of 24 hr old subculture of *Staphylococcus aureus* and *Escherichia coli* in separate conical flask at 40^0-50^0C and mixed well by gentle shaking. About 25 ml of the contents of the flask were poured and evenly spread in Petridis (90 mm in diameter) and allowed to set for two hrs. The cups (8mm in diameter) were formed by the help of borer in agar medium and filled with 0.1 ml (1mg/ml) solution of sample in acetone.

In antibacterial activity of chalcone derivatives (1q – 1x) were carried out using culture of *Klebsiella aerogenes* and *Proteus Vulgaris* by the disc diffusion method and the minimum inhibitory concentration (MIC) of these compounds were determined. Ciprofloxacin was used as the standard drug, whereas dimethyl sulphoxide (DMSO) as solvent. The minimum inhibitory concentration (MIC) was evaluated by the micro dilution method of test compounds. more active against *Klebsiella aerogenes*. Compound 1i was better antibacterial activity against *klebsiella aerogenes*.

B. Antifungal Activity

Aspergillus niger was employed for testing antifungal activity by disc diffusion method. The culture was maintained on Sabouraud dextrose agar slants. Sterilized Sabouraud dextrose agar medium was inoculated with 72 hr old 0.5 ml suspension of fungal spores in a separate flask. About 25 ml of the inoculated medium was evenly spreader in a sterilized Petridis and allowed to set for 2 hr. The cups(8 mm in diameter) were punched in Petridis and loaded with 0.1 ml (2 mg/ml) of solution of sample in acetone. The plates were incubated at 200 – 250^0C for 72 hr. After the completion of incubation period, the zones of inhibition growth is in the form of diameter in mm was measured. Along the test solution in each Petridis one cup was filled up with solvent which acts as control.

In antifungal activity of chalcone derivatives (1q – 1x) were carried out using the culture of *Mucor racemosus*, *A. flavous* and *A. fumigatous* by the disc diffusion method and the MIC of these compounds were determined. *Nystatin* used as the standard drug. The compound 1l shows high(35mm) antifungal activity against aspergillus fumigatous than other compounds.

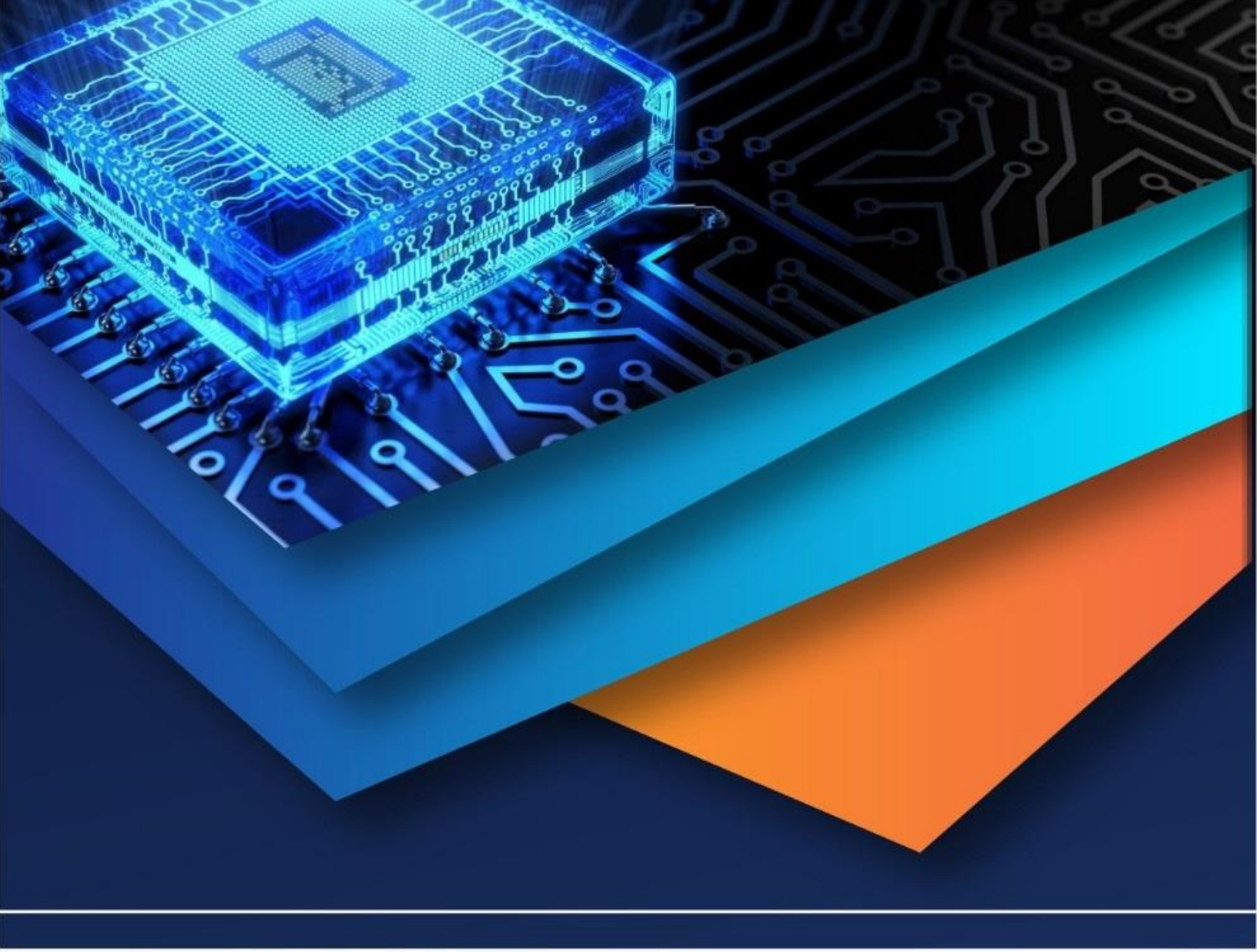
V. CONCLUSION

The present study of an efficient protocol for the Chalcones can be synthesized in good yields from aromatic aldehyde and ketone using the catalytic system of NaOH/ EtOH. The synthesized compounds were characterized by TLC, melting point, IR, NMR spectroscopy and elemental analysis. The results obtained from this study confirmed that the product has formed. The synthesized compounds 1a and 1b show significant antibacterial activity against *Klebsiella aerogenes* and *Proteus vulgaris.* Compounds 1c and 1d shows significant antifungal activityagainst *Mucor rcemosus*, *A. flavous*and *A. fumigatous*. Hence, it is concluded that thereis ample scope for further study in developing these as good lead compounds.

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