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Synthesis and antimicrobial studies of a novel series of piperazine chalcones

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ABSTRACT

A series of novel chalcones were synthesized via Claisen-Schmidt condensation of substituted ketones and 4-(4-Methyl-piperazin-1-yl)-benzaldehyde. These newly synthesized compounds were characterized by physical, chemical and spectral analysis data and are further screened for their antimicrobial activity. The newly synthesized chalcones showed moderate to good antimicrobial activity.

Key words: 4-(4-Methyl-piperazin-1-yl)-benzaldehyde, Ketones, Chalcones, Antimicrobial Activity.

INTRODUCTION

Chalcones are the α,β-unsaturated carbonyl compounds. Since a long time different researchers are utilizing their valuable time for synthesizing the chalcone moieties. This dedication of huge number of researchers supposed to be attracted due to the striking features of chalcones.

Chalcones exhibits various biological activities such as antimalerial [1], antiviral [2], anticancer [3] and other activities [4-5]. In addition to these features chalcones are also acting as an intermediate for the synthesis of various biologically active heterocycles such as pyrimidines [6-7], pyrazolines [8-9], isoxazolines [10-11], flavonoids [12-13], benzodiazepines [14] etc.

In continuation to this persuasion, we synthesized some novel chalcones via Claisen-Schmidt condensation of substituted acetophenones and 4-(4-Methyl-piperazin-1-yl)-benzaldehyde, so that this will be the precious addition to the existing biologically active chalcones.

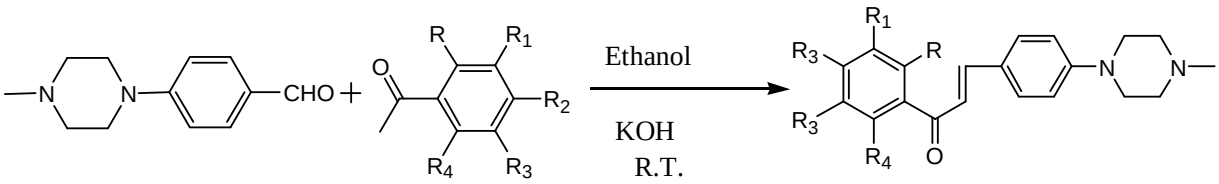
MATERIALS AND METHODS

Melting points were determined in open capillary tubes and are uncorrected. Purity of the compounds was checked by TLC on silica gel G. UV light or iodine vapour accomplished visualization. The IR Spectra were recorded on FTIR perkin-Elmer 1420 spectrometer and PMR spectra ($CDCl_3$) on a varian-300 MHZ spectrometer using TMS as internal standard. Mass spectra were recorded on VG 7070 H Mass spectrometer at 70 eV.

General Procedure for synthesis of chalcones

To a mixture of substituted acetophenones (0.01 mol) and 4-(4-Methyl-piperazin-1-yl)-benzaldehyde (0.01 mol) in ethanol (40 ml) was added 40% solution of sodium hydroxide (5ml). The reaction mixture was then stirred for few minutes after completion of reaction (monitored by TLC) the reaction mixture was poured into ice cold solution of water. The solid obtained washed with water and recrystalised from ethanol.

Physical data of all the synthesized compounds is mentioned in **table-1**.



(I-XVII)

Scheme-1. Synthesis of Chalcones

Entry	R	R_1	R_2	R_3	R_4
I	H	H	H	H	H
II	H	Br	H	H	H
III	H	H	Br	H	H
IV	H	H	OCH_3	H	H
V	H	F	OCH_3	H	H
VI	OCH_3	H	H	OCH_3	H
VII	H	H	OH	H	H
VIII	OH	H	H	H	H
IX	OH	H	OH	H	H
X	OH	Cl	H	H	H
XI	OH	H	H	Cl	H
XII	OH	I	H	Cl	H
XIII	OH	H	H	CH_3	H
XIV	OH	Br	H	CH_3	H
XV	OH	H	CH_3	Cl	H
XVI	OH	Cl	H	Cl	H
XVII	OH	H	CH_3	H	H

RESULTS AND DISCUSSION

A variety of novel chalcones were synthesized via Claisen-Schmidt condensation of substituted acetophenones and 4-(4-Methyl-piperazin-1-yl)-benzaldehyde. The reaction proceeded at room temperature. Work up procedure is simple and yield of the product is excellent.

All the newly synthesized chalcones were subjected for antimicrobial studies and exhibited moderate to good activity.

3-[4-(4-Methyl-piperazin-1-yl)-phenyl]-1-phenyl-propenone (I)
IR (KBr): 1651cm^{-1}(C=O)**,** 1584cm^{-1}(C=C)**;** ^{1}HNMR: δ 2.2 (s,3H,CH_3), δ 2.4 (t,4H,CH_2), δ 3.3 (t,4H,CH_2), δ 6.9 (d,1H,H_α), δ 8.10 (d,1H,H_β), δ 7.0-8.2 (m,9H,Ar-H); M.S. (m/z): m+1= 307.2

1-(4-Bromo-phenyl)-3-[4-(4-methyl-piperazin-1-yl)-phenyl]-propenone (III)
IR (KBr): 1650cm^{-1}(C=O)**,** 1582cm^{-1}(C=C)**;** ^{1}HNMR:δ 2.3 (s,3H,CH_3), δ 2.5 (t,4H,CH_2), δ 3.1 (t,4H,CH_2), δ 6.7 (d,1H,H_α), δ 7.9 (d,1H,H_β), δ 7.0-8.2 (m,8H,Ar-H); M.S. (m/z): m+1= 385

1-(4-Methoxy-phenyl)-3-[4-(4-methyl-piperazin-1-yl)-phenyl]-propenone (IV)
IR (KBr): 1656cm^{-1}(C=O)**,** 1590cm^{-1}(C=C)**;** ^{1}HNMR:δ 2.1 (s,3H,CH_3), δ 2.4 (t,4H,CH_2), δ 3.4 (t,4H,CH_2), δ 3.8 (s,3H,OCH_3), δ 6.8 (d,1H,H_α), δ 8.0 (d,1H,H_β), δ 7.1-8.2 (m,8H,Ar-H); M.S. (m/z): m+1= 337.

Table1. Physical data of synthesized compounds (I-XVII)

Entry	Molecular formula	Yield (%)	Melting point (°C)
I	$C_{20}H_{22}N_2O$	92	110
II	$C_{20}H_{21}BrN_2O$	89	199
III	$C_{20}H_{21}BrN_2O$	88	169
IV	$C_{21}H_{24}N_2O_2$	85	147
V	$C_{21}H_{23}FN_2O_2$	89	154
VI	$C_{22}H_{26}N_2O_3$	84	74
VII	$C_{20}H_{22}N_2O_2$	88	220
VIII	$C_{20}H_{22}N_2O_2$	85	125
IX	$C_{20}H_{22}N_2O_3$	88	>300
X	$C_{20}H_{21}ClN_2O_2$	87	115
XI	$C_{20}H_{21}ClN_2O_2$	83	138
XII	$C_{20}H_{20}ClIN_2O_2$	83	165
XIII	$C_{21}H_{24}N_2O_2$	82	160
XIV	$C_{21}H_{23}BrN_2O_2$	87	123
XV	$C_{21}H_{23}ClN_2O_2$	86	172
XVI	$C_{20}H_{20}Cl_2N_2O_2$	90	202
XVII	$C_{21}H_{24}N_2O_2$	78	135

Antimicrobial activity
Antimicrobial screening was done by using cup plate method [15-16] at a concentration of 100µg/ml. The compounds were evaluated for antibacterial activity against Bacillus subtilis gr +ve, Pseudomonas aeruginosa gr –ve, Staphylococcus aureus gr +ve, Escherichia coli gr –ve and antifungal activity against Aspergillus niger, Aspergillus Flavus, Curvularia, Alternaria. DMSO

was used as solvent control. The results of antimicrobial data are summarized in **table 2.** All compounds show the moderate to good activity against bacteria and fungi.

Table 2: Antimicrobial activity of synthesized compounds (I-XVII)

Products	Bacteria (Zone of Inhibition in mm)				Fungi (Zone of Inhibition in mm)			
	A	B	C	D	E	F	G	H
I	11	15	12	---	---	---	---	---
II	---	13	---	---	---	---	16	---
III	---	15	---	---	---	---	16	---
IV	---	17	14	---	---	---	17	---
V	---	16	---	---	---	---	17	---
VI	12	21	13	---	---	---	15	---
VII	11	---	09	11	---	---	---	---
VIII	13	14	21	19	15	---	21	---
IX	---	---	14	10	---	---	---	---
X	15	---	12	18	12	---	17	---
XI	10	12	10	15	11	---	19	---
XII	12	10	14	09	25	---	---	---
XIII	14	12	14	14	---	---	27	---
XIV	12	10	09	14	---	---	---	---
XV	---	---	11	16	---	---	20	---
XVI	12	---	16	18	16	---	27	---
XVII	14	---	14	18	21	---	17	---

A= *Bacillus subtilis gr +ve*, B= *Pseudomonas aeruginosa gr –ve*,C= *Staphylococcus aureusgr +ve*, D= *Escherichia coli gr –ve*, E= *Aspergillus niger*, F= *Aspergillus Flavus*, G= *Curvularia* H= *Alternaria*.

CONCLUSION

In conclusion, here we have reported some novel chalcones using 4-(4-Methyl-piperazin-1-yl)-benzaldehyde for the first time possessing good to moderate antimicrobial activity via simple procedure within minutes at room temperature. The newly synthesized chalcones were confirmed by spectral analysis.

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