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Synthesis, Characterization and Antimicrobial Activity of various Chalcone Derivatives

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Abstract: ***Chalcone are precursor of the natural occurring flavonoids family. Chalcone consists of two aromatic rings linked by three carbon carbonyl system. The chemistry and biological activities of Chalcone has motivate to synthesis Chalcone as a potential compound for antimicrobial activity. In the present work, a series of Chalcone derivatives were synthesized by Claisen-Schmidt Condensation reaction in which various substituted benzaldehyde and acetoaryl were condensed in the presence of aqueous alcoholic alkali solution. The structures of these compounds were confirmed on the basis of the spectral data. The compounds were screened for their antibacterial activity against B.subtilius, S.aureus, E.coli, P.aureoginosa and antifungal activity against A.niger. The results indicate that synthesized compounds have appreciable antibacterial and antifungal activity.***

Keywords: Chalcone, acetoaryl, antimicrobial activity, parallel synthesizer,MIC.

1. Introduction

The need of new antimicrobial agents has increased as misuse and overuse of antibiotic has created multidrug resistance, this has resulted in global health crisis. So, it has become inevitable for development of new and effective broad spectrum antimicrobial agent and with which there is no cross resistance with the available drugs.The pharmacological activities and chemistry of Chalcone has developed interest to synthesis various Chalcone derivatives for their antimicrobial activity.

Chalcone with antibacterial properties have been known since the 1940s(Deepa Gupta*et al*,2010).Chalcone is an open chain flavonoid in which two aromatic rings are linked three carbon α β unsaturated carbonyl system which is responsible for their biological activities(Ramesh *et al*,2012).Chalcone are well known intermediates for synthesizing various heterocyclic compounds like benzothiazepine(O Prakash*et al,*2005),pyrazolines(RY Prasad*et al*,2005), 1,4 diketones

(S Raghavan and K Anuradha,2002)and flavones(BA Bohn,1998).Chalcones and its derivatives have shown a wide variety of therapeutic activities likeantifungal(Bekhit A. A and Habib N. S,2001),anti-oncogenic(Lopez S. N,2001),anti-inflammatory(Kumar S.K*et al*,2003) ,anti-ulcerative(Hsieh H. K*et al* ,1998),analgesic(Murakami S*et al*,1991),antiviral(Viana G. S.;Bandeira M. A.; Matos F. J,2003)anti-bacterial(WuJ. H.; Wang X. H.; Yi Y. H.; Lee K. H,2003), antimalarial(Liu M.; Go P.; Wilairat M. L,2001), antiprotozoal (LunardiF*et al,*2003), antifilarial (AwasthiSK*et al,*2009), larvicidal (Begum NA; Roy N; Laskar RA; Roy K,2010),anti-convulsant(Kaushik S; Kumar N; Drabu S,,2010),anti-oxidant(Vogel S; Ohmayer S; Brunner G; Heilmann J, ,2008).They have also shown inhibition of the enzymes,like mammalian α-amylase(Najafian M*et al*,2010), cyclooxygenase (Zarghi A*et al* ,2006) and monoamine oxidase (Chimenti F*et al* ,2009).

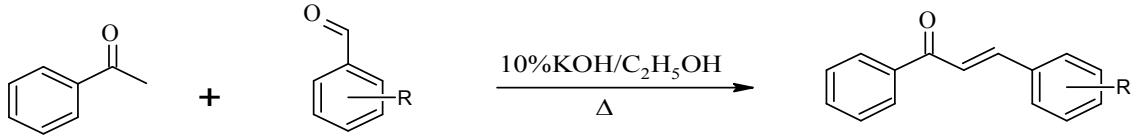
There are various methods to synthesis Chalcone and its derivatives. The most convenient and simplest method is Claisen –Schmidt Condensation Reaction. In the present work, we report reaction between various acetoaryl and substituted benzaldehyde in the presence of aqueous alcoholicalkali solution to form Chalcone derivatives.

2. Experimental

The melting point was determined by Lab India Visual Melting point Apparatus and uncorrected. The IR spectra of the synthesized compounds were recorded on Bruker ATR spectrophotometer. The ^{1}H NMR were recorded in $CDCl_3$using Bruker NMR spectrometer and chemical shifts are reported as parts per million(ppm) using tetramethylsilane (TMS) as internal standard. All the compounds were synthesized by Rodleys Tech Parallel Synthesizer. Reactions were monitored using thin layer chromatography (TLC).The visualization was done using iodine vapour.

3. Scheme of Synthesis

3.1 Scheme 1

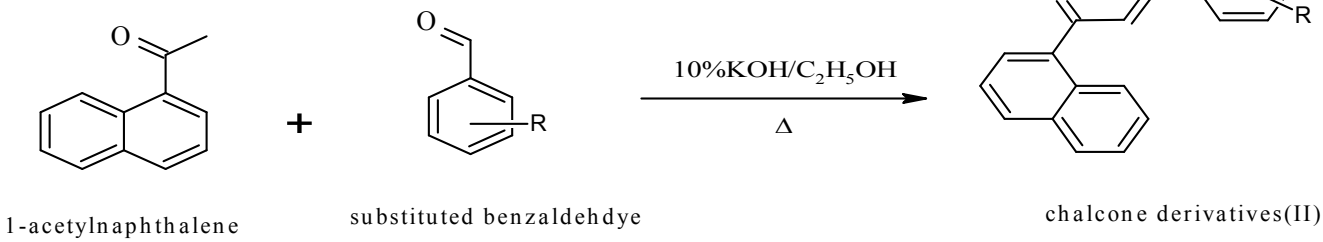


acetophenone substituted benzaldehyde chalcone derivatives (I)

3.1.1 List of substituted benzaldehdye

1. benzaldehyde
2. 2-nitrobenzaldehdye
3. p-chlorobenzaldehdye
4. 3-nitrobenzaldehdye
5. p-methoxybenzaldehyde
6. 2,4-dichlorobenzaldehdye
7. p-dimethylaminobenzaldehdye
8. cinnamaldehdye

3.1.2 Synthetic procedure



1-acetylnaphthalene substituted benzaldehdye chalcone derivatives(II)

To a solution of acetophenone(0.02mol) taken in a 250ml round bottom flask,added cooled mixture of 10% potassium hydroxide and ethanol, stirred thoroughly, then solution of substituted benzaldehyde (0.02mol)was added The reaction mixture was kept for reflux for 3hour on parallel synthesizer at 110°C,75rpm.The solvent was drained and lump obtained was washed with cold water till it become neutral and recrystallized from ethanol. The recrystallized product was kept in cold water for overnight.

3.2 Scheme 2

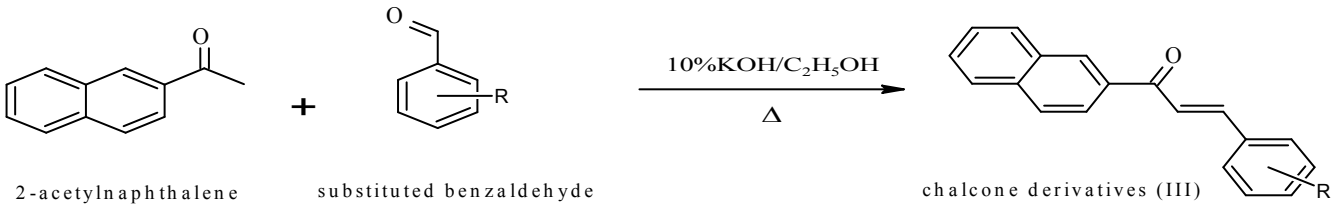
3.2.1 List of Substituted Benzaldehdye

1. benzaldehdye
2. 2-nitrobenzaldehdye
3. p-chlorobenzaldehdye
4. 3-nitrobenzaldehdye
5. p-methoxybenzaldehyde
6. 2,4-dichlorobenzaldehdye
7. p-dimethylbenzaldehdye
8. cinnamaldehdye

3.2.2 Synthetic Procedure

To a solution of 1-acetylnaphthalene(0.02mol) taken in a 250ml round bottom flask ,added cooled mixture of 10% potassium hydroxide and ethanol , stirred thoroughly, then solution of substituted benzaldehyde (0.02mol)was added .The reaction mixture was kept for reflux for 2hour on parallel synthesizer at 115°C,85rpm.The solvent was drained and lump obtained was washed with cold water till it become neutral and recrystallized from ethanol. The recrystallized product was kept in cold water for overnight.

3.3 Scheme 3



2-acetylnaphthalene substituted benzaldehyde chalcone derivatives (III)

3.3.1 List of substituted benzaldehdye

1.benzaldehdye
2. 2-nitrobenzaldehdye
3. p-chlorobenzaldehdye
4. 3-nitrobenzaldehdye
5. p-methoxybenzaldehyde
6. 2,4-dichlorobenzaldehdye
7. p-dimethylbenzaldehdye
8. Cinnamaldehdye

3.3.2 Synthetic procedure:-

To a solution of 2-acetylnaphthalene(0.02mol) taken in a 250ml round bottom flask ,added cooled mixture of 10% potassium hydroxide and ethanol , stirred thoroughly, then solution of substituted benzaldehyde (0.02mol)was added .The reaction mixture was kept for reflux for 2hour on parallel synthesizer at 120°C,95rpm.The solvent was drained and lump obtained was washed with cold water till it become neutral and recrystallized from ethanol. The recrystallized product was kept in cold water for overnight.

Table 1: Physical Data of synthesized compounds

Compound	Molecular Formula	Molecular Weight	M.P(°C)	Yield (%)
A1(1,3-diphenylprop-2-en-1-one)	$C_{15}H_{12}O$	208	55-59	68.05
A2(3-(4-chlorophenyl)-1-phenylprop-2-en-1-one)	$C_{15}H_{11}ClO$	242.5	116-118	85.41
A3(3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one)	$C_{16}H_{14}O_2$	238	62-65	72.18
A4(3-[4-(dimethylamino)phenyl]-1-phenylprop-2-en-1-one)	$C_{17}H_{17}NO$	251	82-86	54.68
A5(3-(2-nitrophenyl)-1-phenylprop-2-en-1-one)	$C_{15}H_{11}NO_3$	253	93-96	71.75
A6(3-(3-nitrophenyl)-1-phenylprop-2-en-1-one)	$C_{15}H_{11}NO_3$	253	70-73	80.10
A7(3-(2,4-dichlorophenyl)-1-phenylprop-2-en-1-one)	$C_{15}H_{10}Cl_2O$	277	105-108	92.64
A8(1,5-diphenylpenta-2,4-dien-1-one)	$C_{17}H_{14}O$	234	96-100	79.87

A9(1-(naphthalen-1-yl)-3-phenylprop-2-en-1-one)	$C_{19}H_{14}O$	258	101-105	93.73
A10(3-(4-chlorophenyl)-1-(naphthalen-1-yl)prop-2-en-1-one)	$C_{19}H_{13}ClO$	292	94-97	75.32
A11(3-(4-methoxyphenyl)-1-(naphthalen-1-yl)prop-2-en-1-one)	$C_{20}H_6O$	288	77-81	74.43
A12(3-[4-(dimethylamino)phenyl]-1-(naphthalen-1-yl)prop-2-en-1-one)	$C_{21}H_9NO$	301	87-90	79.43
A13(1-(naphthalen-1-yl)-3-(2-nitrophenyl)prop-2-en-1-one)	$C_{19}H_{13}NO_3$	303	125-129	74.67
A14(1-(naphthalen-1-yl)-3-(3-nitrophenyl)prop-2-en-1-one)	$C_{19}H_{13}NO_3$	303	112-115	67.35
A15(3-(2,4-dichlorophenyl)-1-(naphthalen-1-yl)prop-2-en-1-one)	$C_{19}H_{12}Cl_2O$	327	78-82	84.43
A16(1-(naphthalen-1-yl)-5-phenylpenta-2,4-dien-1-one)	$C_{21}H_{16}O$	284	130-132	89.06
A17(1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one)	$C_{19}H_4O$	258	119-121	70.20
A18(3-(4-chlorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one)	$C_{19}H_{13}ClO$	292	111-114	59.79
A19(3-(4-methoxyphenyl)-1-(naphthalen-2-yl)prop-2-en-1-one)	$C_{20}H_{16}O_2$	288	83-86	74.43
A20(3-[4-(dimethylamino)phenyl]-1-(naphthalen-2-yl)prop-2-en-1-one)	$C_{21}H_{19}NO$	301	62-66	90.16
A21(1-(naphthalen-2-yl)-3-(2-nitrophenyl)prop-2-en-1-one)	$C_{19}H_{13}NO_3$	303	123-126	61.80
A22(1-(naphthalen-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one)	$C_{19}H_{13}NO_3$	303	89-92	62.66
A23(3-(2,4-dichlorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one)	$C_{19}H_{12}Cl_2O$	327	129-133	63.41
A24(1-(naphthalen-2-yl)-5-phenylpenta-2,4-dien-1-one)	$C_{21}H_{16}O$	284	68-72	69.22

Table 2: Analytical Data of the synthesized compounds

Compounds	R_f value	ATR	NMR(CDCl3) δ
A1	0.82	3059cm^{-1} (Ar-C-H str), 1680 cm^{-1}(C=O str), 1448 cm^{-1} (Ar-C=C str)	7.82-7.94 (d,2H,CO-CH=CH-), 7.15-7.97 (m, 10H, Ar-H)
A2	0.64	1667 cm^{-1}(C=O str), 1489 cm^{-1} (Ar-C=C str),758 cm^{-1}(C-Clstr)	7.83-7.93 (d,2H,CO-CH=CH-), 7.32-7.97 (m, 9H, Ar-H)
A3	0.73	1682 cm^{-1}(C=O str), 1594 cm^{-1} (Ar-C=C str), 1257cm^{-1}(C-O-C)	7.83-7.94 (d,2H,CO-CH=CH-), 7.39-7.96 (m, 9H, Ar-H), 3.89(s, 3H,O-CH3)
A4	0.43	2905 cm^{-1}(Ar C-Hstr),1650 cm^{-1}(C=O str), 1483 cm^{-1} (Ar-C=C str)	8.11-8.26 (d,2H,CO-CH=), 7.32-7.90 (m,9H,Ar-H), 2.73(s,6H,CH3)
A5	0.80	2920 cm^{-1}(Ar C-H str),1673 cm^{-1}(C=O str), 1463 cm^{-1} (Ar-C=C str), 1362 cm^{-1}(NO_2str)	8.15-8.23 (d,2H, CO-CH=CH-), 7.35-8.87 (m, 9H, Ar-H)
A6	0.68	3059 cm^{-1}(Ar C-H str),1666 cm^{-1}(C=O str), 1468 cm^{-1} (Ar-C=C str), 1349 cm^{-1}(NO2 str)	8.01-8.08 (d,2H,CO-CH=CH-), 7.30-7.99 (m, 9H, Ar-H)
A7	0.87	1679 cm^{-1}(C=O str), 1471 cm^{-1} (Ar-C=C str),692 cm^{-1}(C-Clstr)	7.90-7.94(d,2H,CO-CH=CH-), 7.32-7.95 (m, 6H, Ar-H)
A8	0.67	3028cm^{-1}(Ar C-H str),1657 cm^{-1}(C=O str), 1493 cm^{-1} (Ar-C=C str)	7.83-7.86 (d,4H,CO-CH=CH-), 7.22-7.89 (m, 10H, Ar-H)
A9	0.86	3053cm^{-1}(Ar C-H str),1663cm^{-1} (C=O str), 1449cm^{-1}(Ar-C=C str)	7.93-8.01 (d,2H,CO-CH=CH-), 7.23-7.97 (m, 12H, Ar-H)
A10	0.80	3028cm^{-1}(Ar C-H str), 1657 cm^{-1} (C=O str), 1493 cm^{-1} (Ar-C=C str),776 cm^{-1} (C-Clstr)	8.01-8.30 (d,2H,CO-CH=CH-), 7.23-8.75 (m, 11H, Ar-H)
A11	0.67	3049cm^{-1}(Ar C-H str),1660 cm^{-1}(C=O str), 1459 cm^{-1} (Ar-C=C str), 1173 cm^{-1} (C-O-C str)	8.00-8.09(d,2H,CO-CH=CH-), 7.25-7.96 (m, 11H, Ar-H) 3.89(s,3H,O-CH3)
A12	0.59	2906cm^{-1}(Ar C-H str),1675cm^{-1}(C=O str), 1440 cm^{-1} (Ar-C=C str)	8.26-8.37(d,2H,CO-CH=CH-),7.25-8.09(m,11H,Ar-H), 2.73(s,6H,CH3)
A13	0.80	3053cm^{-1}(Ar C-H str),1663cm^{-1}(C=O str), 1499 cm^{-1} (Ar-C=C str), 1178 cm^{-1}(NO_2str)	8.15-8.23(d,2H,CO-CH=CH-), 7.25-8.17 (m, 11H, Ar-H)
A14	0.75	3054cm^{-1}(Ar C-H str),1675 cm^{-1}(C=O str), 1431 cm^{-1} (Ar-C=C str), 1193 cm^{-1}(NO_2str)	8.34-8.37 (d,2H,CO-CH=CH-), 7.38-8.23 (m, 11H, Ar-H)
A15	0.87	3056cm^{-1}(Ar C-H str),1672cm^{-1}(C=O str),1463 cm^{-1} (Ar-C=C str), 753 cm^{-1}(C-Clstr)	7.95-8.00 (d,2H,CO-CH=CH-),7.25-7.88 (m,10HAr-H)
A16	0.77	3050cm^{-1}(Ar C-H str),1650 cm^{-1}(C=O str), 1450 cm^{-1} (Ar-C=C str)	7.83-7.86 (d,2H,CO-CH=CH-),7.22-7.72 (m,14H Ar-H)
A17	0.78	3053cm^{-1}(Ar C-H str),1663 cm^{-1}(C=O str), 1499 cm^{-1}(Ar-C=C str)	7.94-8.01(d,2H,CO-CH=CH-),7.25-7.89(m,12H Ar-H)
A18	0.84	1660 cm^{-1}(C=O str), 1448 cm^{-1} (Ar-C=C str),756 cm^{-1}(C-Clstr)	7.89-7.95 (d,2H,CO-CH=CH-),7.25-7.84 (m,11H Ar-H)
A19	0.80	1663 cm^{-1}(C=O str), 1510 cm^{-1} (Ar-C=C str),1173 cm^{-1}(C-O-C str)	8.00-8.04 (d,2H,CO-CH=CH-),7.34-7.99 (m,11H Ar-H), 3.89 (s,3H,OCH3)
A20	0.69	2899cm^{-1}(Ar C-H str),1674 cm^{-1}(C=O str), 1437cm^{-1} (Ar-C=C str)	8.11-8.27(d,2H,CO-CH=CH-),7.25-7.99(m,11H.Ar-H), 2.73(s,6H,CH3)
A21	0.78	1676 cm^{-1}(C=O str), 1364 cm^{-1} (Ar-C=C str),1278 cm^{-1}(NO_2str)	8.15-8.23(d,2H,CO-CH=CH-),7.25-7.98 (m,11H Ar-H)
A22	0.68	1669 cm^{-1}(C=O str), 1526 cm^{-1} (Ar-C=C str),1179cm^{-1}(NO_2str)	8.00-8.06 (d,2H,CO-CH=CH-),7.25-7.98 (m,11H Ar-H)
A23	0.78	1662 cm^{-1}(C=O str), 1469cm^{-1} (Ar-C=C str),752 cm^{-1}(C-Clstr)	8.02-810 (d,2H,CO-CH=CH-),7.25-7.99 (m,10H Ar-H)

A24	0.69	3054cm^{-1}(Ar C-H str),1661 cm^{-1}(C=O str), 1499cm^{-1}(Ar-C=C str)	7.83-7.89 (d,4H,CO-CH=CH-),7.24-7.68 (m,12H Ar-H)

4. Antimicrobial Activity

All the synthesized compounds have been screened for anti-bacterial against two Gram positive Bacteria-B.subtilius and S. Aureus and two Gram negative bacteria- E.coli and P.aerogunisona and for antifungal activity against Aspergillus niger using Serial Dilution Method. Ciprofloxacin was used as reference standard for antibacterial activity. Ketoconazole was used as reference standard for antifungal activity.

Serial Dilution method is used to determine Minimum Inhibitory Concentration (MIC) of antimicrobial agent to inhibit the microorganisms. This can be achieved by dilution of agents in either agar or broth medium. In the present study, Broth dilution method was used to determine MIC.

Nutrient Broth medium was used for antibacterial activity and Sabouraud medium for antifungal activity. All the synthesized compounds were diluted into 2,4,8, 16,32µg/ml and DMSO was used as control.

(MIC)

Compounds	MIC (conc. In µg/ml)				
	B. Subtilis	S.Aureus	E.Coli	P.Aeroginosa	A.Niger
A1	8	8	8	8	16
A2	8	8	8	8	4
A3	16	8	8	16	4
A4	16	16	8	8	8
A5	8	16	16	16	4
A6	8	8	16	8	4
A7	8	16	8	8	8
A8	32	32	32	32	16
A9	16	8	8	8	8
A10	8	8	8	8	4
A11	8	16	8	8	16
A12	8	16	16	8	16
A13	16	32	32	16	8
A14	16	8	16	8	4
A15	16	32	16	16	4
A16	32	32	32	32	8
A17	8	8	8	8	4
A18	8	8	8	8	4
A19	8	8	8	16	4
A20	16	16	8	8	8
A21	8	16	16	8	4
A22	8	8	8	8	4
A23	16	16	8	16	4
A24	32	32	32	32	8
Standard	0.2	0.4	0.2	0.2	0.64

5. Result and Discussion

From the above Result, it is found that compounds **A1**(1,3-diphenylprop-2-en-1-one), **A2**(3-(4-chlorophenyl)-1-phenylprop-2-en-1-one), **A10**(3-(4-chlorophenyl)-1-(naphthalen-1-yl)prop-2-en-1-one), **A17**(1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one), **A18**(3-(4-chlorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one) and**A22**(1-(naphthalen-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one) are comparatively more active than all other compounds, for both Gram-Positive and Gram-Negative bacteria. The Chalcone are found to be more active for Gram-Negative bacteria compare to gram-Positive.

For the anti-fungal activity compounds **A3**(3-(4-methoxyphenyl)-1-phenylprop-2-en-1-one), **A5**(3-(2-nitrophenyl)-1-phenylprop-2-en-1-one), **A6**(3-(3-nitrophenyl)-1-phenylprop-2-en-1-one), **A10**(3-(4-chlorophenyl)-1-(naphthalen-1-yl)prop-2-en-1-one), **A14**(1-(naphthalen-1-yl)-3-(3-nitrophenyl)prop-2-en-1-one), **A15**(3-(2,4-dichlorophenyl)-1-(naphthalen-1-yl)prop-2-en-1-one), **A17**(1-(naphthalen-2-yl)-3-phenylprop-2-en-1-one), **A18**(3-(4-chlorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one), **A19**(3-(4-methoxyphenyl)-1-(naphthalen-2-yl)prop-2-en-1-one), **A21**(1-(naphthalen-2-yl)-3-(2-nitrophenyl)prop-2-en-1-one), **A22**(1-(naphthalen-2-yl)-3-(3-nitrophenyl)prop-2-en-1-one)&**A23**(3-(2,4-dichlorophenyl)-1-(naphthalen-2-yl)prop-2-en-1-one) are found to be more active than all other compounds. The Chalcone are found to be more active as antifungal than antibacterial.

6. Conclusion and Future Scope

Various Chalcone derivatives are synthesized by Claisen-Schmidt Condensation Reaction and evaluated for their antimicrobial activity. The screening of synthesized compounds for antimicrobial activity showed that these compounds have appreciable antimicrobial activity. The results provide insights that will aid the optimization of the Chalcone derivatives for the better activity and may prove helpful for further lead optimization, virtual screening, molecular docking and molecular dynamics studies.

7. Acknowledgement

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