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Synthesis and Characterization of Novel Cinnamamide Deriverties and Their Antimicrobial Activities

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Abstract: *Cinnamamides and its derivatives possess wide range of biological activities, and thus novel cinnamamide derivatives were synthesized by using heterocyclic moiety. All the compounds were characterized by on the basis of analytical data, IR, NMR spectral studies. Biological screening of the synthesized compounds is most important task which decides the practical utility of compounds in various fields such as medicinal and pharmaceuticals.The synthesized compounds antimicrobial activities were also evaluated. The preliminary results showed that all the title compounds had certain antimicrobial activities against S. aureus, Bacillus, and E.coli. at a different concentration.*

Key Words**:** *Aldehydes, Antimicrobial activities, Cinnamamides, Heterocyclic moiety, synthesis.*

I. Introduction

Cinnamamides and its derivatives possess wide range of activities in several fields. Such as medicinal, pharmaceuticals, agricultural, biological[1] and many other activities. In medicinal field several Cinnamamides and their derivatives are reported to show central nervous system depressant, anticonvulsant [2], muscle relaxant, antiallergic, antineoplastic, antitumor, anesthetic, analgesic and anti-infective activities [3-7], etc. In the agrochemical field, insecticidal, their avian repellent, herbicidal activities, and several excellent cinnamamide fungicides [8-9] for example dimethomorph, fluormorph and pyrimorph, have been successfully developed.

Biological screening of the synthesized compounds is most important task which decides the practical utility of compounds in various fields such as medicinal, pharmaceuticals etc. Inspire of wide range of their applications and very less attention is paid towards the synthesis of cinnamamides derivatives containing heterocyclic moiety. Literature survey and biological activities' of cinnamamides have motivated to undertake the synthesized novel cinnamamides entitled "(2E)-1-(4-methylpeperazin-1-yl)-3-subsituted phenylprop-2-en-1-one Cinnamamides. The present study is related to the synthesis of some novel cinnamamides containing heterocyclic moiety. Synthesized compounds were characterized by elemental analysis and IR, NMR spectral studies. The preliminary results showed that all the title compounds had certain antimicrobial activities against S. aureus, Bacillus, and E.coli. at a different concentration.

II. Result And Discussion

All synthesized novel cinnamamides compounds contained heterocyclic moiety in the form of N-methyl Piperazine. The Witting reaction is an important method for the synthesis of alkenes. By using this method novel cinnamamides containing heterocyclic moiety entitled (2E)-1-(4-methylpeperazin-1-yl)-3-substituted phenylprop-2-en-1-one Cinnamamides are synthesized from different aromatic aldehydes and Witting reagents having good yields. The yields of synthesized compounds were ranging from 54 to 78%. All synthesized compounds were characterized on the basis of melting point, elemental analysis, Rf value, IR spectra and 1HNMR spectral analysis. Preliminary results showed that all the title compounds had certain antimicrobial activities against S. aureus, Bacillus, and E.coli. at a different concentration.

III. Experimental

3.1 Synthesis of Wittig reagent - N-methyl piperazine chloracetamide were synthesized by using equimolar solution of chloroacetylchloride and N-methyl piperazine in chloroform at 0^{o}C with continuous stirring in fuming chamber. When this reaction mixture gives the salt by adding its solution in benzene to the stirred solution of triphenylphosphine and reaction mixture was refluxed for 4-6 hrs. The solid products obtained were filtered and air dried. Thus for Purification obtained salt was dissolved in 100 ml water then 90 ml of dry benzene, add 1-2 drops of phenolphthalein indicator and add NaOH solution in it till pink colour persist this was indicates that the neutralization of present acid from reagent. Then benzene layer was separated and washed with water and concentrated to one third volume. Finally the product scratched with n-Hexane to obtain solid witting reagent.

3.2 Synthesis of (2E)-1-(4-methylpeperazin-1-yl)-3-Substituted phenylprop-2-en-1-one Cinnamamides –

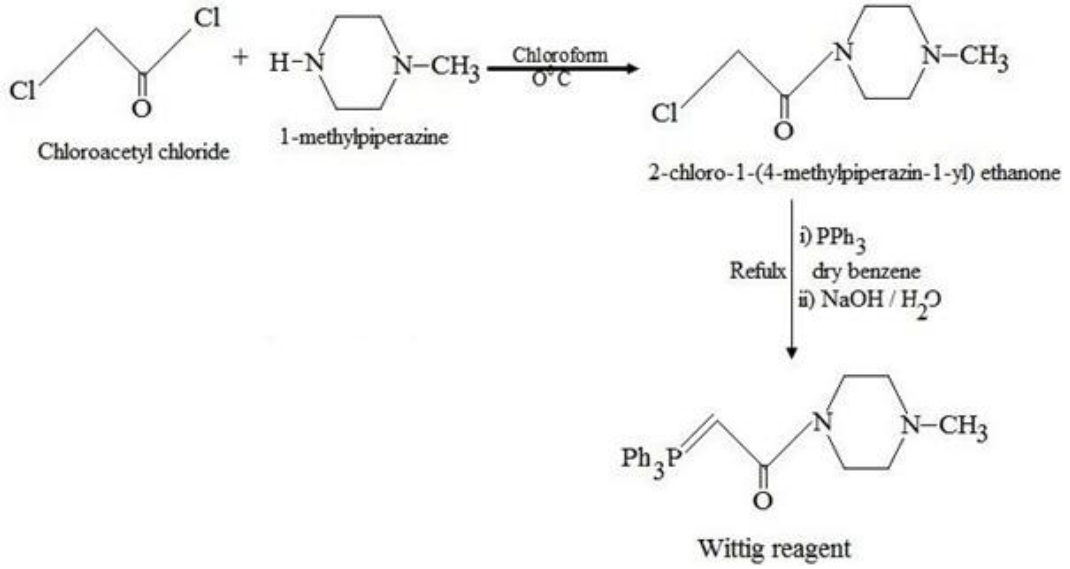
Equimolar solution of Witting reagent and various aromatic aldehydes in water and refluxed for 4 to 6 hrs. The progress of reaction was monitored by thin layer chromatography. Melting points were taken by open capillary method. The elemental analysis was calculated for carbon, hydrogen, nitrogen and chlorine. IR spectra were recorded with TMS as internal standard using CDCl3. All Synthesized compounds were purified by column chromatography. All chemicals used were of analytical grade.

IV. Antimicrobial Activities

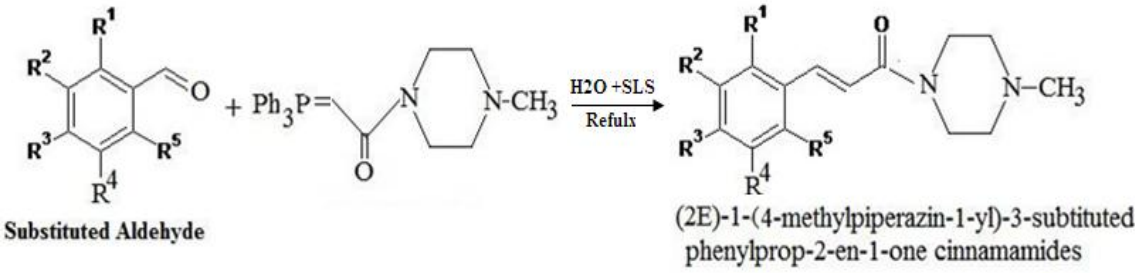
Antimicrobial activities of newly synthesized compounds were carried out by using cup plate agar diffusion method at 01 mg / ml, 0.5 mg/ml, and 0.25 mg/ml in DMSO against using antibiotics ciprofloxacin. Plates were incubated 24hrs at 37^0C and zone of inhibition were measured in mm. Result have been incorporated in table, all synthesized compound were found to be moderately active against bacteria.

V. Schemes

5.1 Synthesis of witting reagent-



5.2 Synthesis of Cinnamamides derivatives of heterocyclic moiety-



VI. Tables

Table No. 1 Antimicrobial activities of the compounds (in mm)

Compounds	S. aureus			Bacillus			E.coli		
	1Mg/ml	0.5Mg/ml	0.25Mg/ml	1Mg/ml	0.5Mg/ml	0.25Mg/ml	1Mg/ml	0.5Mg/ml	0.25Mg/ml
Ia	14	13	11	12	10	09	13	11	10
Ib	15	14	12	14	13	12	18	17	15
Ic	18	17	14	13	12	10	15	13	12
Id	15	14	12	21	18	16	19	17	16
Ie	12	10	09	18	17	16	12	11	10
If	17	16	15	31	28	25	16	15	12
Ig	23	19	12	15	12	13	16	14	10
Ih	24	22	21	18	16	17	18	15	16
Ii	20	18	14	24	21	19	30	28	24
Ij	24	22	21	19	15	12	29	26	22

Table No.2-Substituted aromatic aldehydes used in the synthesis of Cinnamamides

Sr. No.	Substituent → / Compounds ↓	R1	R2	R3	R4	R5
1	Ia	H	H	H	H	H
2	Ib	H	H	OMe	H	H
3	Ic	H	OMe	OMe	H	H
4	Id	H	OMe	OMe	OMe	H
5	Ie	H	-O-CH_2-O-		H	H
6	If	NO_2	H	H	H	H
7	Ig	H	H	Cl	H	H
8	Ih	H	H	NO_2	H	H
9	Ii	H	H	$N(Me)_2$	H	H
10	Ij	H	H	OH	H	H

Table No.3- Characteristics data for synthesized Cinnamamides

Sr. No.	Compounds	Molecular Formula	Molecular Wt.	Yield %	M.P.^{0}C
1	Ia	$C_{14}H_{18}ON_2$	230	76	69
2	Ib	$C_{15}H_{20}O_2N_2$	260	62	165
3	Ic	$C_{16}H_{22}O_3N_2$	290	54	205
4	Id	$C_{17}H_{24}O_4N_2$	320	78	172
5	Ie	$C_{15}H_{18}O_3N_3$	288	66	62
6	If	$C_{14}H_{17}O_3N_2$	261	58	102
7	Ig	$C_{14}H_{17}ON_2Cl$	264.5	78	79
8	Ih	$C_{14}H_{17}O_3N_3$	275	70	218
9	Ii	$C_{16}H_{23}ON_2$	259	56	200
10	Ij	$C_{14}H_{18}O_2N_2$	246	64	82

Table No.4-Elemental analysis of synthesized compounds

Compounds	Molecular Formula	% C	% H	% O	% N	% Cl
Ia	$C_{14}H_{18}ON_2$	73.06 (73.04)	7.88 (7.83)	7.00 (6.96)	12.19 (12.17)	---
Ib	$C_{15}H_{20}O_2N_2$	69.26 (69.23)	7.72 (7.69)	12.36 (12.31)	10.82 (10.77)	---
Ic	$C_{16}H_{22}O_3N_2$	66.26 (66.21)	7.64 (7.59)	16.62 (16.55)	9.70 (9.66)	---
Id	$C_{17}H_{24}O_4N_2$	63.80 (63.75)	7.54 (7.50)	20.06 (20.00)	8.80 (8.75)	---
Ie	$C_{15}H_{18}O_3N_3$	62.58 (62.50)	6.30 (6.25)	16.71 (16.66)	14.62 (14.58)	---
If	$C_{14}H_{17}O_3N_2$	64.42 (64.36)	6.53 (6.51)	18.42 (18.39)	10.75 (10.72)	---
Ig	$C_{14}H_{17}ON_2Cl$	64.02 (63.51)	6.48 (6.42)	6.10 (6.05)	10.56 (10.58)	13.40 (13.42)
Ih	$C_{14}H_{17}O_3N_3$	62.12 (61.10)	6.21 (6.18)	17.46 (17.45)	15.28 (15.27)	---
Ii	$C_{16}H_{23}ON_2$	74.16 (74.13)	8.90 (8.88)	6.20 (6.17)	10.84 (10.81)	---
Ij	$C_{14}H_{18}O_2N_2$	68.31 (68.29)	7.32 (7.31)	13.03 (13.00)	11.40 (11.38)	---

In bracket calculated percentages of element.

VII. Spectral Data Studies

Ia=(2E)-1-(4-methylpeperazin-1-yl)-3- phenylprop-2-en-1-one Cinnamamides-
IR (cm^{-1})1656, 1595
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.7(d), (1H), (CH=CHCO), J=15.94 HZ; 6.8(d), (1H), (CH=CHC_6H_5) J=15.94 HZ; 7.2-7.5(m), (5H), (C_6H_5).
Ib- IR (cm^{-1})1685, 1600
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.9(d), (1H), (CH=CHCO), J=15.60 HZ; 7.0(d), (1H), (CH=CHC_6H_5) J=15.60HZ; 3.2(s), (3H), (OMe); 7.2(d), (2H); 7.4(d), (2H).
Ic- IR (cm^{-1})1678, 1644
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.9(d), (1H), (CH=CHCO), J=15.70 HZ; 7.2(d), (1H), (CH=CHC_6H_5) J=15.70HZ; 3.2(s), (6H), (OMe); 6.9-7.1(m), (3H), Ar-H.
Id- IR (cm^{-1})1688, 1636
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.9(d), (1H), (CH=CHCO), J=15.8 HZ; 7.4(d), (1H), (CH=CHC_6H_5) J=15.84HZ; 3.2(s), (9H), (OMe); 6.6(s),(2H), Ar-H.

Ie-IR(cm^{-1}) 1670, 1650
1HNMR-1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.9(d), (1H), (CH=CHCO), J=15.84 HZ; 7.4(d), (1H), (CH=CHC_6H_5) J=15.84HZ; 4.8(s), (4H), (H_2COCH_2); 7.4-7.6(s), (3H), Ar-H.
If- IR (cm^{-1})1664, 1680
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.8(d), (1H), (CH=CHCO), J=15.72 HZ; 7.3(d), (1H), (CH=CHC_6H_5) J=15.72HZ; 7.4-7.5(m), (4H), Ar-H.1H
Ig- IR (cm^{-1})1656, 1580
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.6(d), (1H), (CH=CHCO), J=15.70 HZ; 6.8(d), (1H), (CH=CHC_6H_5) J=15.70HZ; 7.3-7.5(m), (4H).
Ih- IR (cm^{-1})1664, 1680
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.7(d), (1H), (CH=CHCO), J=15.70 HZ; 6.8(d), (1H), (CH=CHC_6H_5) J=15.70HZ; 7.3-7.4(m), (4H).
Ii- IR (cm^{-1})1654, 1546
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.7(d), (1H), (CH=CHCO), J=15.70 HZ; 6.8(d), (1H), (CH=CHC_6H_5) J=15.70HZ; 7.3-7.4(m), (4H); 2.5 (s), (6H), NMe_2.
Ij- IR (cm^{-1})1656, 1680, 2550
1H NMR-(δ)-2.2(s), (3H), NMe; 2.9(t), (4H); 3.5(t), (4H); 6.7(d), (1H), (CH=CHCO), J=15.70 HZ; 6.8(d), (1H), (CH=CHC_6H_5) J=15.70HZ; 7.3-7.4(m), (4 H); 5.6 (s), (1H), OH.

VIII. Conclusion

The given study clearly showed that all new cinnamamides derivatives were synthesized by using heterocyclic moiety N-methyl piperazine and different aromatic aldehyde gives very good yield. All synthesizes compounds were characterized on the basis of elemental analysis, IR spectra and ^{1}HNMR spectral analysis. Synthesized compounds have been tested for antimicrobial activities and it gives very good results with increasing the concentration of synthesized compounds.

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