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Research Article

SYNTHESIS AND ANTIBACTERIAL ACTIVITIES OF SOME CHALCONES AND 1,5-BENZTHIAZEPINE

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ABSTRACT

Some new 1,5-benzthiazepine derivatives have been prepared by the condensation of chalcones with 2-amino thiophenol in methanol and acetic acid. All these derivatives have been screened for antibacterial activities and characterized by spectral studies.

INTRODUCTION

Chalcone is an aromatic α, β-unsaturated ketone that forms the central core for a variety of important biological compounds. Chalcones can be synthesized by condensation of Aryl ketones with aromatic aldehyde[1,2]. Many Literature reviews reveals that the chalcones and its heterocyclic derivatives shows antibacterial acticity[3-6]. 1,5-Benzothiazepines have anti-fungal, anti-bacterial,[7] antifeedant[8], analgesic[9], anti convulsant[10], anti-HIV[11], and squalene synthetase inhibitory activity[12]. 1, 5-Benzothiazepine skeleton is considered as an important moiety in synthetic and pharmaceutical chemistry. We report the reaction of 2-hydroxy-5-methyl-4,6-dibromoacetophenone with various substituted aromatic aldehydes to produced corresponding 2'-hydroxy-5'-methyl-4',6'-dibromo chalcones[1-10]. Which on treatment with 2-aminothiophenol give the corresponding derivatives of 1,5-benzthiazepine[11-20]. The constitution of all compounds synthesized was established by elemental analysis, IR and H^1 NMR spectral study. Compounds were also evaluated for anti bacterial activities.

MATERIAL AND METHODS

The identification and characterization of synthesized compounds were carried out by the following procedure to determine that all the prepared compounds were of different chemical nature than the respective parent compounds. The melting points were determined for the synthesized compounds were taken in open capillary tubes and are uncorrected. IR spectra in KBr were recorded on Perkin-Elmer-377 spectrophotometer and H^1 NMR spectra were recorded on Varian NMR spectrophotometer. All compounds gave satisfactory elemental analysis.

Synthesis of 2'-hydroxy-5' -methyl-4', 6'-dibromo chalcones [1-10]

A mixture of 2-hydroxy-5methyl-4,6-dibromoacetophenone (0.01 mole) and aryl aldehyde (0.01 mole) in ethanol (30 ml) was stirred and to it excess of 40% potassium hydroxide (25 ml) solution was added. The mixture was kept overnight at room temperature. The colour of the reaction mixture was change from yellow to orange. The content was then poured over crushed ice and acidified with hydrochloric acid (1:1). The solid separated was filtered, washed with distilled water, dried and crystallized from ethanol, yield 60-70%.

Preparation of 2,3-dihydro-4(2'-hydroxy-5'-methyl-4',6'-dibromophen-1'-yl)-2-substitutedphenyl-1,5-benzthiazepine[11-20]

2'-hydroxy-5'-methyl-4', 6'-dibromo chalcone [1-10] (0.01 mol) and 2-aminothiophenol (0.01mol) in anhydrous methanol (100ml) and glacial acetic acid (10 ml), was refluxed on water-bath at 60-70°C for 2 hours. Then reaction mixture was cooled. The separated product was filtered and crystallized from ethanol (99%), yield 50-60%.

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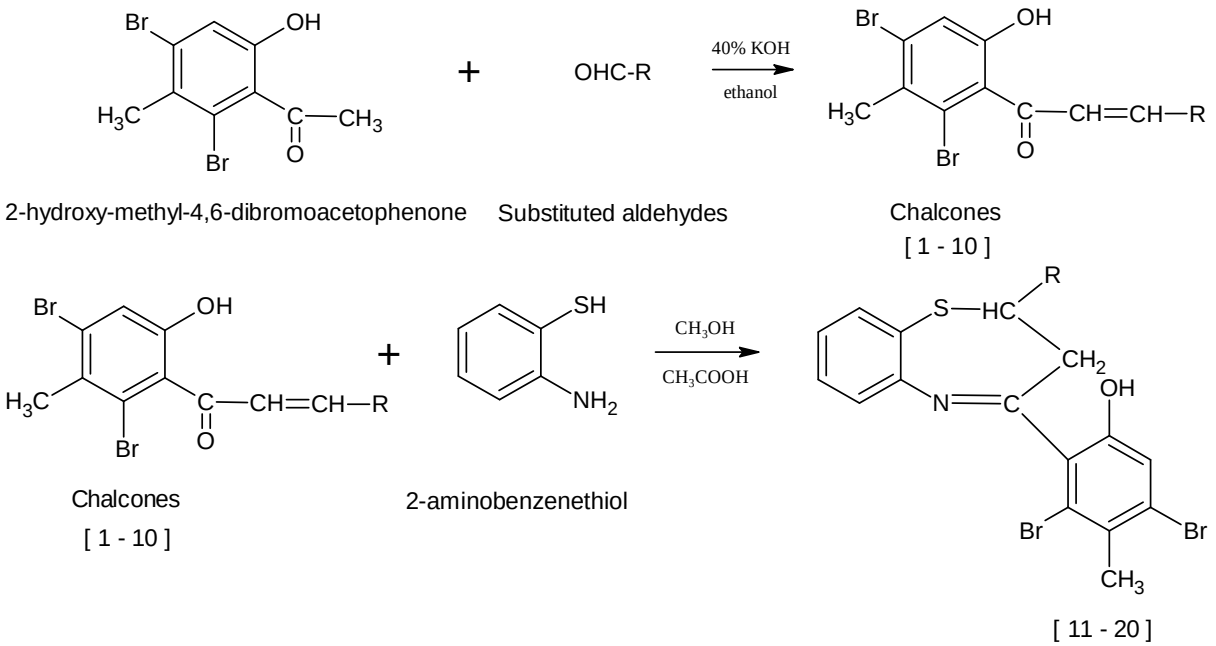
Reaction Scheme



Where R = 4-chlorophenyl, 4-hydroxyphenyl, Phenyl, 2,4-dichlorophenyl, 3-phenoxyphenyl, 2,6-dichlorophenyl, 3-nitrophenyl, 3,4,5-trimethoxyphenyl, 4-methoxyphenyl, 4-N,N-dimethylaminophenyl.

Scheme

Table 1 Characterization Table of 2'-hydroxy-5'-methyl-4',6'-dibromo chalcones[1-10] and 2,3-dihydro-4(2'-hydroxy-5'-methyl-4',6'-dibromophen-1'-yl)-2-substitutedphenyl-1,5-benzthiazepine[21-30]

Compd. No.	R	Molecular formula	(M. wt.)	Yield (%)	M.P. ^{0}C.
1	4-chlorophenyl	$C_{16}H_{11}O_2Br_2Cl$	430.522	62.36	124
2	4-hydroxyphenyl	$C_{16}H_{12}O_3Br_2$	412.077	68.60	118
3	Phenyl	$C_{16}H_{12}O_2Br_2$	396.078	60.38	130
4	2,4-dichlorophenyl	$C_{16}H_{10}O_2Cl_2Br_2$	464.967	57.55	142
5	3-phenoxyphenyl	$C_{22}H_{16}O_3Br_2$	488.175	50.59	112
6	2,6-dichlorophenyl	$C_{16}H_{10}O_2Br_2$	394.062	57.76	162
7	3-nitrophenyl	$C_{16}H_{11}O_4NBr_2$	441.075	59.04	156
8	3,4,5-trimethoxyphenyl	$C_{19}H_{18}O_5Br_2$	486.156	62.50	120
9	4-methoxyphenyl	$C_{17}H_{14}O_3Br_2$	426.104	60.36	108
10	4-N,N-dimethylaminophenyl	$C_{18}H_{17}O_2NBr_2$	439.146	63.89	128
11	4-chlorophenyl	$C_{22}H_{17}ONSBr_2Cl$	540.702	49.73	109
12	4-hydroxyphenyl	$C_{22}H_{18}O_2NSBr_2$	520.264	50.36	117
13	Phenyl	$C_{22}H_{18}ONSBr_2$	504.265	51.11	126
14	2,4-dichlorophenyl	$C_{22}H_{16}ONSBr_2Cl_2$	573.154	39.33	134
15	3-phenoxyphenyl	$C_{28}H_{21}O_2NSBr_2$	595.354	55.09	98
16	2,6-dichlorophenyl	$C_{22}H_{16}ONSBr_2Cl_2$	573.154	44.29	104
17	3-nitrophenyl	$C_{22}H_{17}O_3N_2SBr_2$	549.262	47.84	142
18	3,4,5-trimethoxyphenyl	$C_{25}H_{23}O_4NSBr_2$	593.335	47.28	125
19	4-methoxyphenyl	$C_{23}H_{20}O_2NSBr_2$	534.291	48.05	136
20	4-N,N-dimethylaminophenyl	$C_{24}H_{23}ON_2SBr_2$	547.333	55.95	128

Table-2 ^{1}H NMR spectral data table of 2'-hydroxy-5'-methyl-4-methoxy-4',6'-dibromo chalcone(Compound no. 9).

Chemical shift	Relative Number of Protones	Assignment
2.30	3	$-CH_3$
3.85	3	$-OCH_3$
6.35	1	-OH
6.40	1	=CH-
7.25	1	-CH=
7.30-7.90	5	Ar-H

Table 3 ^{1}H NMR spectral data table of 2,3-dihydro-4-(2'-hydroxy-5'-methyl-4',6'-dibromophen-1'-yl)-2-(4"-N,N-dimethylaminophenyl)-1,5-benzthiazepine(Compound no. 20).

Chemical shift	Relative Number of Protones	Assignment
2.30	3	$-CH_3$
2.85	2	$-CH_2$ of benzothiazepine ring
3.05	6	$-CH_3$
4.32	1	-CH of benzothiazepine ring
6.35	1	-OH
6.72-8.05	9	Ar-H

^{1}H NMR Spectroscopy

Nuclear magnetic resonance (NMR) spectroscopy is one of the latest physical methods which is use for the structure elucidating of organic compounds. PMR spectra of chalcones and benzothiazepine were recorded on varian spectrophotometer. Spectra were examined in $CDCl_3$ at room temperature using TMS as internal standard.

Infrared spectra

Infrared absorption were recorded using potassium bromide pallets method. The spectra were recorded using "Perkin-Elmer" spectrophotometer. The results are describe in table no. 4 and 5.

Table 4 IR spectra of 2'-hydroxy-5'-methyl-4-hydroxy-4',6'-dibromo chalcone(Compound no. 2).

Position of absorption band (cm^{-1})	Intensity	Band and its mode of vibration	Functional group
610	s	C-Br stretching	Bromo compound
1390	sh	O-H bending	Ar-OH intramolecular
1445	s	C-H bending	-
1585	w	C=C stretching	Alkene group
1640	s	C=O stretching	Ketone
2940,2840	m	C-H stretching	-
3380	sh	O-H stretching	Ar-OH group

S=strong, m=medium, b=broad, w=weak, sh=sharp, v=variable

Table 5 IR spectra of 2,3-dihydro-4-(2'-hydroxy-5'-methyl-4',6'-dibromophen-1'-yl)-2-(4"-N,N-dimethylaminophenyl)-1,5-benzthiazepine(Compound no. 20).

Position of absorption band (cm^{-1})	Intensity	Band and its mode of vibration	Functional group
610	s	C-Br stretching	Bromo compound
870	s	C-S stretching	Thiazepine ring
1310	m	C-N stretching	Compound containing C-N group
1380	sh	O-H bending	Ar-OH intramolecular
1475	m	C-H bending	-
1580	s	C=N stretching	Compound containing C=N group
2870,2980	m	C-H stretching	-
3425	sh	O-H stretching	Ar-OH group

S=strong, m=medium, b=broad, w=weak, sh=sharp, v=variable

Antibacterial activity

The synthesized compounds were screened for their antibacterial activity using *S.aureus, E. coli* by cup plate method using DMF as solvent. All the compounds shows mild activity against both bacteria in comparison with ampicilin and gentamycin. The results are describe in table no. 6.

RESULTS AND DISCUSSION

All the tested compounds have shown antibacterial activity. As compared to the available routine antimicrobial compounds like Ampicilline and Gentamycin, The chalcones derivatives have shown the medium activity and the 1,5-benzthiazepine derivatives have shown weak activity against both organism.

Among the tested compounds no. **6,8,10,15**, and **16** shown the maximum activity against all the compounds towards gram +ve bacteria i.e. S. aureus while rest of the compounds have shown good activity against S. aureus bacteria. The compound no. **6,7,10** and **20** have shown the maximum activity amongst all the compounds towards gram –ve bacteria i.e. E.coli and the compound no. **1,3,4,8,9** and **18** have shown the medium activity against E. coli bacteria while the reast of the compounds have shown weak activity agains E. coli bacteria. The rest of the compounds are found less active against both bacteria.

Table 6

Compound No.	Zone of inhabitation in mm Antibacterial (24 hrs.)	
	S.aureus (+ve)	E.coli (-ve)
1	11	12
2	10	10
3	10	12
4	12	13
5	09	10
6	13	15
7	11	16
8	14	10
9	09	12
10	14	14
11	11	10
12	11	09
13	12	11
14	11	09
15	15	10
16	13	10
17	10	08
18	11	13
19	12	10
20	12	14
Standard Drugs:		
Ampicilin	18	-
Gentamycin	-	21

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