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Synthesis, Characterization and antimicrobial activity of Novel chalcones Derivatives having benzyloxymonoiodo raceacetophenone moiety

S. S. Sipai, Dr. S. S. Shah

Department of Chemistry, Shri U. P. Arts, Smt. M. G. Panchal Science & Shri V. L. Shah Commerce College, Pilvai, India

ABSTRACT

Chalcones derivatives has wide applications in Pharmaceutical and medicinal chemistry. 1-(4-benzyloxy-2-hydroxy-3-iodophenyl)-3-(substitutedphenyl)prop-2-en-1-one Compounds (B_{1-10}) were synthesized by coupling with aromatic substituted aldehyde. All the synthesized compounds were characterized by IR, ^1H NMR. The synthesized compounds were screened for antimicrobial activity.

Keywords: Chalcones, Phenone Derivatives Antibacterial Activity and Antifungal Activity

I. INTRODUCTION

The presence of chalcones is one main structural components inVarious naturally occurring biologically active compounds chalcones, analogs of 1,3-diaryl pro-2-ene-1-one form a wide class of compounds containing two aromatic rings bound with vinyl ketone fragment. It is well known that largely natural or synthetic chalcones are highly active withextensive pharmaceutical and medicinal application. Chalcones are found to be effective asanalgesic [1], antimalerial [2], antiviral [3], antibacterial [4], antifungal [5], antimitotic [6],cytotoxic [7], antifeedant [8], anti-inflammatory [9], antileishamanial [10], antitumor [11],anticancer [12], antimicrobial [13], antinociceptive [14], insecticidal [15] and antiinvasive [16]activities

II. MATERIAL AND METHODS

Melting points were determined in open capillary tubes and are uncorrected. The IR spectra were recorded in KBr pellets on a Buker spectrometer and 1 H NMR spectra in CDCl3 on Hitachi R-1500, 60 MHz spectrometer using TMS as an internal standard. All chemicals used were of laboratory grade. Preparation of 1–(4-benzyloxy-2-hydroxy-3-iodo phenyl) ethanone (BHIE) and chalcone is as given below.

Synthesis of 1–(4–benzyloxy-2-hydroxy phenyl) ethanone

General procedure:

1–(2, 4 dihydroxyphenyl) ethanone (0.10 mol), Benzyl bromide (0.1 mol) and Potassium carbonate (0.1 mol) were taken in 100ml of Acetone. Reaction mixture was shake for 7 hrs at reflux 50-60°C temperature. Reaction mixture was cooled to room temperature and quenched with 100ml cold water. The final product 1– (2-hydroxy–4-benzyloxyphenyl) ethanone was passed through pass through a filter and rinsed with water. Prepared product was recrystallized by ethanol.

Synthesis of 1–(4–benzyloxy-2-hydroxy–3-iodo phenyl) ethanone: (A)

1– (2-hydroxy–4-benzyloxyphenyl) ethanone (0.1 mol) was taken in 100ml of ethanol. Iodination method[7] has been used. Iodine granules (0.1 mol) and 300ml were taken in 250ml R.B.F and stirred them till 15

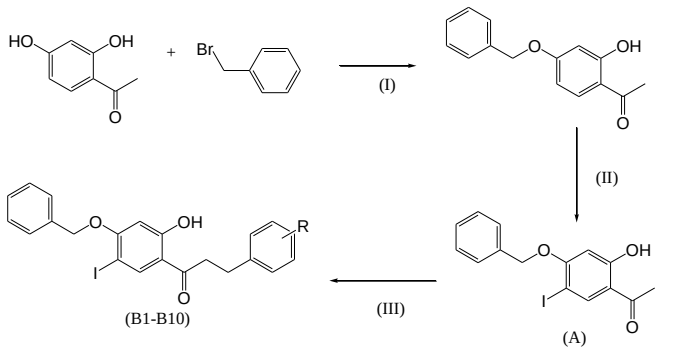
minutes. Iodic acid (0.1 mol) dilute in to 4ml of dist.water in a small beaker. Slowly add this iodic acid solution in to the reaction mixture and stirred them continuously for 30 minutes at 35 – 40 C, the reaction was monitored by TLC. Pour it in to ice. Excess iodine was removed by adding fresh saturated sodium bisulphite solution. Formed material 1–(2-hydroxy–4–benzyloxy-3-iodo phenyl) ethanone was passed through filter out and washes them two to three times with distilled water. Synthesized material was recrystallized in ethanol.

Synthesis of 1-(4-benzyloxy-2-hydroxy-3-iodo phenyl)-3-(substituted phenyl) prop-2-en-1-one from 1–(4–benzyloxy-2-hydroxy–3-iodo phenyl) ethanone :(B_1-B_{10})

General procedure :

1-(4-benzyloxy-2-hydroxy-3-iodophenyl) ethanone (0.01 mol) and substituted aromatic aldehydes (0.01 mol) were dissolved in ethanol (25 ml) was added 10% sodium hydroxide solution, (25 ml) was added slowly and the mixture stirred for 4 hrs, the reaction monitored by TLC. Then it was poured into 400 ml of water with constant starring and neutralized with 10% hydrochloric acid solution and left overnight in refrigerator. The precipitate obtained was filtrated, washed and recrystallized from ethanol.

REACTION SCHEME



R= H, 2-Cl, 3-Cl, 4-Cl, 2-OH, 3-OH, 4-OH, 2-OCH_3, 3-OCH_3, 4-OCH_3

1-(4-benzyloxy-2-hydroxy-3-iodophenyl) ethanone (A)

Mass;368.17 ; IR(KBr cm-1): 2870(C-H str. vib.) 3032(-Aromatic C-H),1573,1489, (C=C str.Vib.),879(-C – H o.o.p multi sub. benzene),1280, 1080(C-O-C str.vib), 3634(O-H str.vib), 1620(-C=O str.vib),501(C-I str.vib),;1H NMR 6.44 – 7.77 (s,7H,of the Ar-H) ,13.5 (s,1H, Ar-OH), 5.16 (2H,s, -CH2-O-), 2.5 (3H,s, O=CCH3),;Yield 64.30%;

1-(4-Benzyloxy-2-hydroxy- 3-iodophenyl)-3-phenylprop-2-en-1-one [B1]:

Mass;456.27 IR(KBr cm-1): 3063(-Aromatic C-H),1573, 1489,(C=C str. Vib.),817(-C – H o.o.pmulti sub. benzene),1273, 1072(C-O-C str.vib), 3634(O-H str.vib), 1627(-C=O str.vib), 501(C-I str.vib) ,972(CH=CH bending),;1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-), Yield 57.23%;

1-(4-benzyloxy -2-hydroxy-3-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B2]:

Mass; 490.72 IR(KBr cm-1): 3032(Aromatic C-H),1573, 1489,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1280, 1041(C-O-C str.vib), 3201(O-H str.vib), 1627(-C=O str.vib), 786(C-Cl str.vib) 578(C-Istr.vib),972(CH=CH bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH2-O-),; Yield 59.91%;

1-(4-benzyloxy- 2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B3]:

Mass;472.27 IR(KBr cm-1): 3050(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1273, 1080(C-O-C str.vib), 3201(O-H str.vib), 1627(-C=O str.vib), 501(C-I str.vib),964(CH=CHbending), 1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 64.32%;

1-(4-benzyloxy- 2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B4]:

Mass:486.3; IR(KBr cm-1): 3063(Aromatic C-H),1558, 1404,(C=C str. Vib.),825(-C – H o.o.p multisub. benzene),1280, 1072(C-O-C str.vib), 3194(O-H str.vib), 1627(-C=O str.vib), 540(C-I) str.vib),972(CH=CH)bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 62.81%;

1-(4-benzyloxy -2-hydroxy-3-iodophenyl)-3-(2-chlorophenyl) prop-2-en-1-one [B5]:

Mass; 490.72 IR(KBr cm-1): 3063(Aromatic C-H),1573, 1450,(C=C str. Vib.),864(-C – H o.o.p multisub. benzene),1226, 1049(C-O-C str.vib), 3649(O-H str.vib), 1627(-C=O str.vib), 732(C-Cl str.vib) 509(C-Istr.vib),972(CH=CH bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH2-O-),; Yield 58.50%;

1-(4-benzyloxy- 2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B6]:

Mass;472.27 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1219, 1080(C-O-C str.vib), 3518(O-H str.vib), 1620(-C=O str.vib), 501(C-I str.vib),941(CH=CHbending), 1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 59.37%;

1-(4-benzyloxy- 2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B7]:

Mass:486.3; IR(KBr cm-1): 3063(Aromatic C-H),1573, 1489,(C=C str. Vib.),856(-C – H o.o.p multisub. benzene),1280, 1072(C-O-C str.vib), 3194(O-H str.vib), 1627(-C=O str.vib), 509(C-I) str.vib),972(CH=CH)bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 53.17%;

1-(4-benzyloxy -2-hydroxy-3-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B8]:

Mass; 490.72 IR(KBr cm-1): 3032(Aromatic C-H),15723, 1492,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1273, 1041(C-O-C str.vib), 3439(O-H str.vib), 1620(-C=O str.vib), 786(C-Cl str.vib) 501(C-Istr.vib),972(CH=CH bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.49 (m, 2H, -CH=CH-), 5.16(d,2H, -CH2-O-),; Yield 58.23%;

1-(4-benzyloxy- 2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B9]:

Mass;472.27 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),879(-C – H o.o.p multisub. benzene),1280, 1080(C-O-C str.vib), 3510(O-H str.vib), 1620(-C=O str.vib), 501(C-I str.vib),966(CH=CHbending), 1H NMR:8.0 -7.7 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 59.71%;

1-(4-benzyloxy- 2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B10]:

Mass:486.3; IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),815(-C – H o.o.p multisub. benzene),1280, 1041(C-O-C str.vib), 3510(O-H str.vib), 1627(-C=O str.vib), 501(C-I) ,648 (C-I) str.vib),966(CH=CH)bending), 1H NMR:8.0 -7.2 (m,12H, of the Ar-H) ,6.44-6.52 (m, 2H, -CH=CH-), 5.17 (d,2H, -CH2-O-),; Yield 54.33%;

Antibacterial Activity

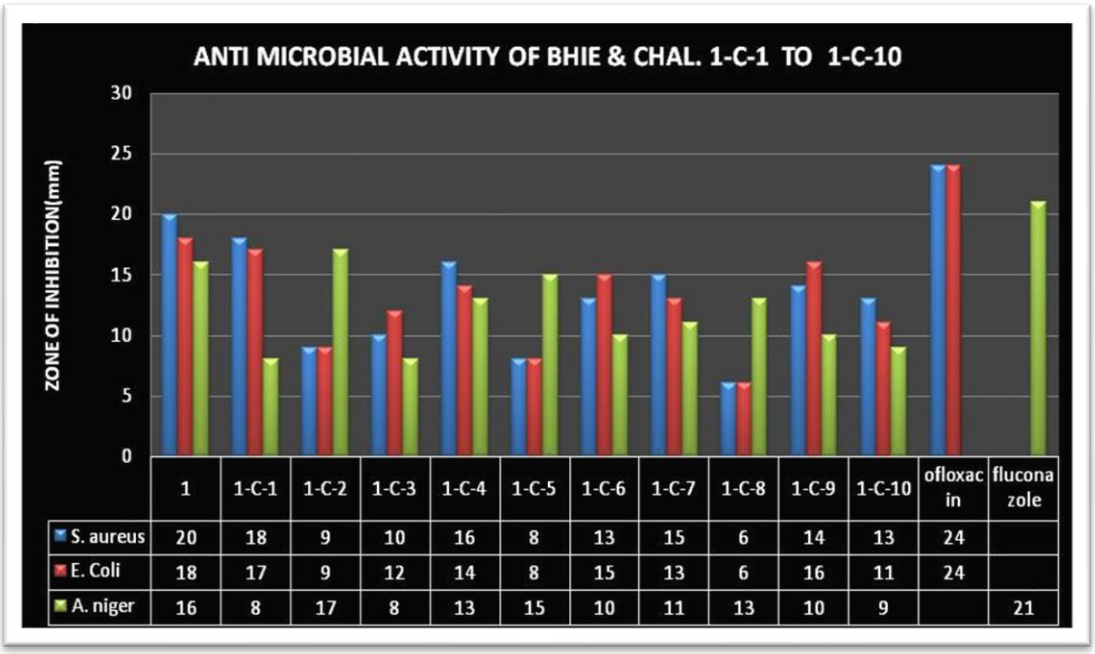
The purified products were screened for their antibacterial activity by using cup-plate agar diffusion method. Thenutrient agar broth prepared by the usual method, was inoculated aseptically with 0.5 ml of 24 hrs old subculture of *Staphylococcus aureus* and *Escherichia coli* in separate conical flasks at 400-500C and mixed well by gentle shaking.About 25 ml of the contents of the flask were poured and evenly spread in petridish (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 filledwith 0.1 ml (1 mg/ml) solution of sample in Acetone.

Antifungal Activity

A niger was employed for testing antifungal activity by cup-plate agar diffusion method. The culture was maintainedon Sub rouse dextrose agar slants. Sterilized Sub rouse dextrose agar medium was inoculated with 72 hrs old 0.5 mlsuspension of fungal spores in a separate flask. About 25 ml of the inoculated medium was evenly spreaded in asterilized petridish and allowed to set for 2 hrs. The cups (8 mm in diameter) were punched in petridish and loaded with 0.1 ml (2 mg/ml) of solution of sample in Acetone. The plates were incubated at 20 –250C for 72 hrs. After thecompletion of incubation period, the zones of inhibition growth is in the form of diameter in mm was measured.Along the test solution in each petridish one cup was filled up with solvent which acts as control. The zones ofinhibition.

Microbial activity of 1–(4–benzyloxy-2-Hydroxy-3-iodo phenyl) ethanone chalcones from Aromatic Aldehydes



	1	1-C-1	1-C-2	1-C-3	1-C-4	1-C-5	1-C-6	1-C-7	1-C-8	1-C-9	1-C-10	ofloxacin	fluconazole
S. aureus	20	18	9	10	16	8	13	15	6	14	13	24	
E. Coli	18	17	9	12	14	8	15	13	6	16	11	24	
A. niger	16	8	17	8	13	15	10	11	13	10	9		21

III. RESULTS AND DISCUSSION

In the present work, some novel chalcones of 1– (4–benzyloxyphenyl-2-hydroxy-3-iodophenyl) ethanone (BHIE) from ten aromatic substituted aldehydes have been prepared. During the preparation work, it was found that most of the chalcones using aromatic aldehydes could be easily prepared by most convenient claisen-schmidt condensation method. The chalcones could be easily prepared at room temperature after 72 hours. It was found that the chalcones derived from aromatic aldehydes were stable and can be easily converted to its heterocyclic compound. To establish a new synthetic process for chalcones, different reaction conditions were applied. From the results, it was found that the chalcones could be prepared from BHIE using aromatic substituted aldehyde by shaking the reaction mixture at normal temperature for 4 hours. Thus, a new developed synthetic process has been applied to prepare chalcones from BHIE using aromatic substituted aldehydes in the present work.

To check the applicability of the prepared compounds, they were screened for their antibacterial and antifungalactivity by using cup-plate diffusion method. The antibacterial activity of each compound was compared withstandard drug viz. Ofloxacin and

antifungal activity was compared with standard drug viz. Fluconazole. The zone ofinhibition was measured in millimeter. From the results, it may be generalized that the antibacterial activity ongram-positive and gram-negative bacteria of chalcones. Most of all compounds show moderate and poor antibacterial activity. The antifungal activity of each compound was found poor with compared to standard drug.

IV. CONCLUSION

We have prepared new chalcones containing benzyloxy moiety in their structured excepting enhanced bioactivity. None of the compounds have shown good antimicrobial activity compared to slandered drugs.

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