International Journal of Scientific Research in Chemistry (IJSRCH) | Online ISSN: 2456-8457
© 2018 IJSRCH | Volume 3 | Issue 4

Synthesis, Characterization and antimicrobial activity of some Novel chalcones Derivatives having 1-(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl) ethanone moiety

S. S. Sipai, S. S. Shah, Deep 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-5-chloro-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, ^{13}C NMR ,^{1}H NMR, and Mass spectra. The synthesized compounds were screened for antimicrobial activity.

Keywords : Chalcones, Phenone Derivatives, Antibacterial Activity and Antifungal Activity

I. INTRODUCTION

Chalcones are an important class of compounds which are good intermediates for the synthesis of various heterocyclic compounds like flavones, flavanones, flavanols, aurones, isoxazolines, anthocynins, pyrazolines, pyrimidines, quinoxalines, benzalcoumaranones. The biological and industrial applications of chalcones are also found significant. Due to the presence of chromophor –CO-CH=CH- and other auxochromes. Kostanekci and Tambor[1] gave them the name "Chalcones." Chalcones are characterized by their possession of a structure in which two aromatic ring I and II are linked by an aliphatic three-carbon chain. The chalcones have been found to be useful in providing structure of natural products like cynamaclurin[2], sakuranetin[3], ploretin4, hemlocktanin[5], homoriodictyo[6],etc. Keeping in view of biological importance of this group and their close relationship to flavones, flavanones, flavanols and dihidroflavonals, chalcones have been investigated since long time. It has been of great interest in their study as intermediates for substances of therapeutic importance[7]. Schraufstatter and Deutsch[8] and Calcinari[9] reported antibacterial properties of some chalcones, and have concluded that the bacteriostatic activity is due to their unsaturation.

II. MATERIAL AND METHODS

Melting point of the synthesized compounds was determined in electro thermal apparatus using fused capillary tubes. Monitoring of the reaction and the purity of the compounds was checked by thin-layer chromatography using silica gel G plates of 0.5 mm thickness as stationary phase in combination of n-hexane: ethyl acetate in different ratios as mobile phase.

The Infrared (IR) spectra of the synthesized compounds were recorded on a Fourier Transform IR spectrophotometer (model Shimadzu 8700) in the range of 4000-400 cm-1 using KBr pellets and value of λmax are reported in cm-1 and the spectra were interpreted. Proton Nuclear Magnetic Resonance (1NMR) and (13CNMR) Nuclear Magnetic Resonance

spectra were recorded on Bruker Avance II 400 NMR spectrometer using CDCl3. Chemical shift (δ) are reported in parts per million downfield from internal reference, Tetramethylsilane (TMS) and the spectra were interpreted.

III. EXPERIMENTAL

Synthesis of 1–(4–benzyloxy-2-hydroxy phenyl) ethenone 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-5-chloro-2-hydroxyphenyl) ethanone (BCHE)

1– (4-benzyloxy-2-hydroxyphenyl) ethanone (BHE) (0.1 mole) was taken in 100ml of ethanol and NCS (N-Chlorosuccinimide) was taken in minimum amount of DMF. Reaction material was swirled for 5 – 6 hrs at normal temperature. Reaction material was quenched with 200ml cold water. Formed material 1–(4–benzyloxy-5-chloro-2-hydroxyphenyl) ethanone (BCHE) was passed through filter and rinsed with distilled water. Synthesized material was crystallized in ethan-1-ol.

Synthesis of 1–(4–benzyloxy-5-chloro-2-hydroxy–3-iodophenyl) ethanone (BCHIE)

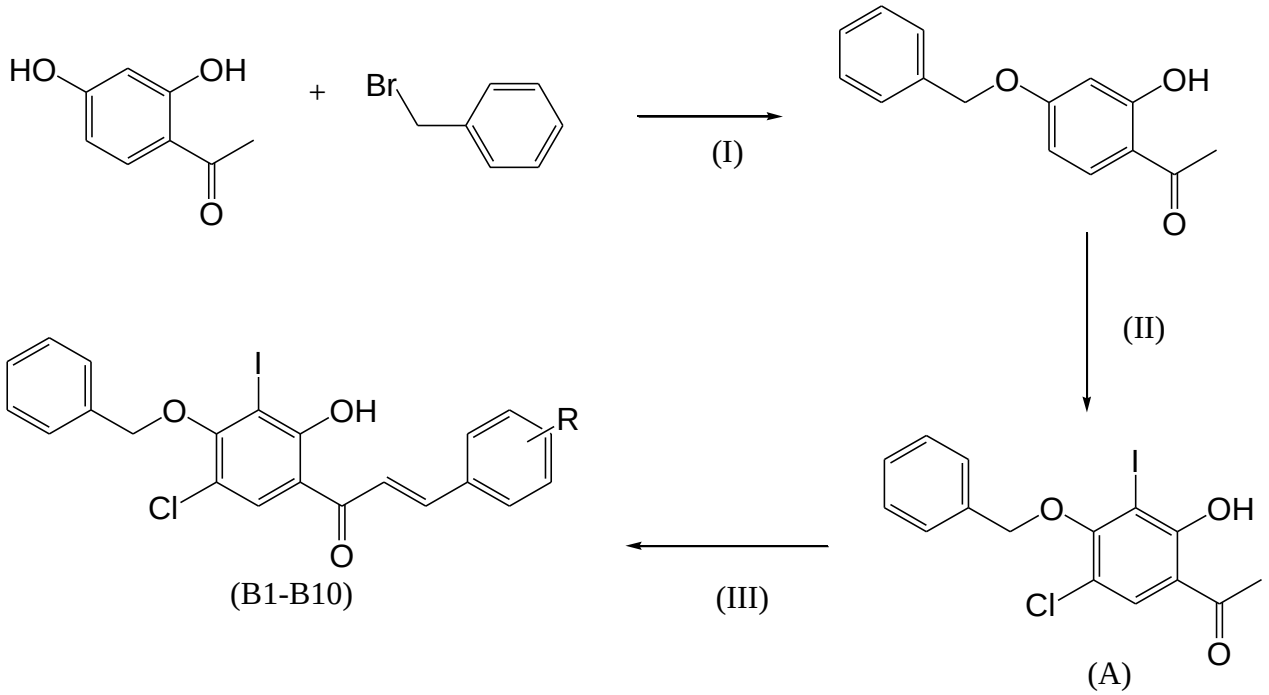
To prepare 1–(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl)ethanone (BCHIE)iodination method has been used 2.42 gm (0.01 mole), 2.54 gm(0.01 mol) iodine granules and 30 ml ethyl alcohol were taken in 250 ml beaker and shake till 5 to 10 minutes. 1.76 gm (0.01 mol) iodic acid dilute in to 4 ml of dist. Water in a small beaker. Slowly add this iodic acid solution in to the mixture of BCHE and iodine in etanol and stire them continuosly for 30 min. at 35° – 40o C. pour in to ice. Excess iodine was removed by adding sat. Sodium bisulfite solution. Filter out precipitates and wash them two to three times with dist. Water. Recrystallized from ethyl alcohol.

Synthesis of 1-(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl)-3-(substituted phenyl) prop-2-en-1-one from 1–(4–benzyloxy-5-chloro-2-hydroxy–3-iodo phenyl) ethanone :(B_1-B_{10})

General procedure:

1-(4-benzyloxy-5-chloro-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-5-chloro-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), (C-I)563,740(C-Cl 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%;

1-(4-Benzyloxy-5-chloro-2-hydroxy- 3-iodophenyl)-3- phenylprop-2-en-1-one [B1]:

Mass;455.9 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), 3432(O-H str.vib), 1627(-C=O str.vib), 563(C-I)732(C-Clstr.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 54%;

1-(4-benzyloxy -5-chloro-2-hydroxy-3-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B2]:

Mass; 490.4 IR(KBr cm-1): 3069(Aromatic C-H),1560, 1490,(C=C str. Vib.),857(-C – H o.o.p multisub. benzene),1230, 1052(C-O-C str.vib), 3649(O-H str.vib), 1627(-C=O str.vib), 740(C-Cl str.vib) 563(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 56%;

1-(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B3]:

Mass;471.9 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1223, 1078(C-O-C str.vib), 3425(O-H str.vib), 1649(-C=O str.vib), 740(C-Cl str.vib) 563(C-I str.vib),975(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 58%;

1-(4-benzyloxy-5-chloro-2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B4]:

Mass:485.9; 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), 740(C-Cl str.vib)563(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 61%;

1-(4-benzyloxy -5-chloro-2-hydroxy-3-iodophenyl)-3-(2-chlorophenyl) prop-2-en-1-one [B5]:

Mass; 490.4 IR(KBr cm-1): 3063(Aromatic C-H),1589, 1473,(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), 740(C-Cl str.vib) 563(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 56%;

1-(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B6]:

Mass;471.9 IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1220, 1080(C-O-C str.vib), 3518(O-H str.vib), 1620(-C=O str.vib), 740(C-Cl str.vib) 563(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 58%;

1-(4-benzyloxy-5-chloro-2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B7]:

Mass:485.9; 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), 740(C-Cl str.vib) 563(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 61%;

1-(4-benzyloxy -5-chloro-2-hydroxy-3-iodophenyl)-3-(4-chlorophenyl) prop-2-en-1-one [B8]:

Mass; 490.4 IR(KBr cm-1): 3032(Aromatic C-H),1589, 1481,(C=C str. Vib.),817(-C – H o.o.p multisub. benzene),1273, 1049(C-O-C str.vib), 3634(O-H str.vib), 1620(-C=O str.vib), 740(C-Cl str.vib) 563(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 56%;

1-(4-benzyloxy- 5-chloro-2-hydroxy-3-iodophenyl)-3-(3-hydroxyphenyl) prop-2-en-1-one [B9]:

Mass;471.9 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), 740(C-Cl str.vib) 501(C-I str.vib),956(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 58%;

1-(4-benzyloxy-5-chloro-2-hydroxy- 3-iodophenyl)-3-(4-methoxyphenyl) prop-2-en-1-one [B10]:

Mass:485.9; IR(KBr cm-1): 3032(Aromatic C-H),1489, 1404,(C=C str. Vib.),848(-C – H o.o.p multisub. benzene),1273, 1041(C-O-C str.vib), 3634(O-H str.vib), 1627(-C=O str.vib), 740(C-I) ,563 (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 61%;

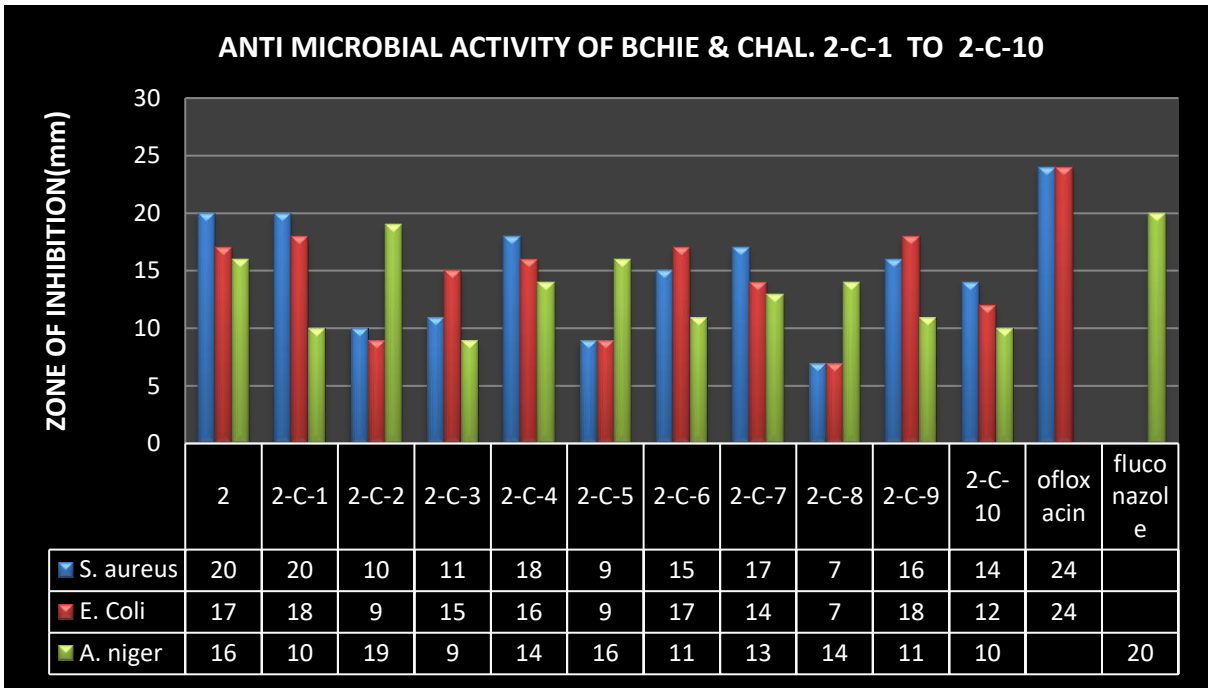
Antibacterial Activity

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 theinoculated 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-5-chloro-2-hydroxy-3-iodophenyl) ethanone and chalcones



	2	2-C-1	2-C-2	2-C-3	2-C-4	2-C-5	2-C-6	2-C-7	2-C-8	2-C-9	2-C-10	oflox acin	fluco nazole
S. aureus	20	20	10	11	18	9	15	17	7	16	14	24	
E. Coli	17	18	9	15	16	9	17	14	7	18	12	24	
A. niger	16	10	19	9	14	16	11	13	14	11	10		20

IV. RESULTS AND DISCUSSION

In the present work, some novel chalcones of 1–(4–benzyloxy-5-chloro-2-hydroxy-3-iodophenyl) ethanone (BCHIE) 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 BCHIE 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 andantifungal 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.

V. 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 standard drugs.

VI. ACKNOWLEDGMENT

We are thankful to Department of Chemistry Shri U.P. Arts, Smt M.G. Panchal Science & Shri V.L. Shah Commerce College Pilvai (India) for providing the necessary facilities for the research work. We are also thankful to the director NFDD complex, Saurashtra Uni. Rajkot for providing the necessary spectral data IR, ^{1}NMR, and ^{13}NMR.

VII. REFERENCES

[1]. Kostaneki S. V. and Tambor.; Chem. Ber.,32,1921 (1899).
[2]. Milter P. and Soha; J. Ind. Chem.Soc., 11,257 (1934).
[3]. Blicke F. F. and Kawall C. E.; J.Am. Chem. Soc., 54,428 (1942).
[4]. Shinoda J., Sato J. and Kawago.; J. Pharm. Soc., Japan, 24, 1459 (1904).
[5]. Russel A.; J.Chem.Soc.,1506 (1934).
[6]. Kurth E. F.; J. Am. Chem. Soc., 68, 697(1946).
[7]. Rohrmann E., jones R.G. and Sonale H. A.; ibid., 66,1856 (1944).
[8]. Schrautstalter E. and Deutsch S.; C.A. 44, 3568 (1904).
[9]. Calcinari R.; C.A. 77, 48007 (1950).

Suggested Citation :

S. S. Sipai, S. S. Shah, Deep S. Shah, "Synthesis, Characterization and antimicrobial activity of Some Novel chalcones Derivatives having 1-(4-benzyloxy-5-chloro-2-hydroxy-3-iodophenyl) Ethanone Moiety", International Journal of Scientific Research in Chemistry (IJSRCH), ISSN : 2456-8457, Volume 3, Issue 4, pp.89-114, September-October.2018
URL : http://ijsrch.com/IJSRCH183413