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# SYNTHESIS OF SOME NOVEL BIOACTIVE POLYHYDROXY CHALCONES

**Nana V. Shitole, Suraj B. Ade and Subhash M. Lonkar***

P.G. Department of Chemistry, MSP Mandal's Shri Shivaji College, Parbhani-431 401, M.S.India.

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***Correspondence for Author**
**Dr. Subhash M. Lonkar**
P.G. Department of Chemistry, MSP Mandal's Shri Shivaji College, Parbhani-431 401, M.S.India.

## ABSTRACT

A series of novel 1-(2'-hydroxy substituted phenyl)-3-(-2'-hydroxy substituted phenyl)-2-propen-1-ones were synthesized by Claisen Schmidt condensation of -2'-hydroxy substituted acetophenone and 2' -hydroxy substituted arylaldehyde in alkaline medium KOH and ethanol. An excellent yield 75-80 % were obtained in bulb oven at 55-60$^0$C after 14-16 hrs. The compounds were found to have antimicrobial activity.

**KEYWORDS***:* Polyhydroxychalcones, 2'-hydroxy acetophenone, 2'-hydroxy arylaldehyde, antimicrobial activity.

## INTRODUCTION

The Claisen–Schmidt (CS) condensation between acetophenone and benzaldehyde derivatives are valuable C–C bond-forming reaction to produce chalcones. Chalcones belong to the flavonoid families which are synthesized in factories to preserve the health of plants against infections and parasites.[1] Chalcones having one or more hydroxyl groups attached to ring A or B is called polyhydroxy chalcones. Chalcones are widely distributed in nature. They are well known for their biological and medicinal properties[2] antimicrobial[3], antifungal[4] anti-inflammatory[5], insecticidal[6], analgesic[7], antimalerial[8], antitumor[9], anticancer[10] and as nutritional antioxidant in cancer and heart disease[11]. An attention has been focused due to such wide range of pharamaceutical and antimicrobial properties of chalcones thought to be worthwile synthesized some new polyhydroxy chalcones have enhanced biological activity. Various reagents have been introduced as the methodology was developed during last few decades, such as $Cp_2ZrH_2$ [12], $Cp_2TiPh_2$ [13], $RuCl_3$ [14], $SmI_3$ [15], $TiCl_3(CF_3SO_3)$ [16], $La^{3+}$-immobilized organic solid [17], KF-$Al_2O_3$ [18], $Mg(HSO_4)_2$ [19],

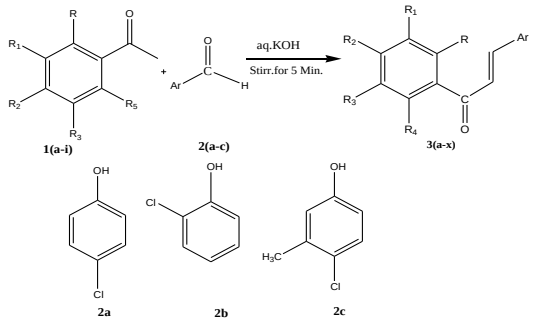
$BF_3 \cdot OEt_2$ [20], $InCl_3$ [21], TMSCl/NaI [22], silica chloride [23], silica-supported phosphorus pentoxide (P2O5/SiO2) or silicaphosphinoxide (silphox, [POCl3-n(SiO2)n]) as heterogeneous reagents [24], 1-methyl-3(2 (sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride[25] sodium-modified-hydroxyapatite (Na-HAP)[26]. The application of these methods suffer from some disadvantages such as the use of costly or less easily available reagents, harsh reaction conditions, long reaction times, poor yields, and the use of toxic solvents. Therefore, despite a number of procedure, an efficient, practical and facile method for syntheses of these polyhydroxy chalcones are desired.

## EXPERIMENTAL

All aldehydes were obtained from freshly opened container and used without further purification, where melting point was determined in open capillary tubes and was uncorrected The purity of compounds was checked routinely by TLC (0.5 mm thickness) using silica gel –G-coated. At plates (Merck) and spots were visualized by exposing the dry plates in iodine vapours, IR spectra (υ max in $cm^{-1}$) were scanned on FTIR Perkin Elmer model RXI spectrometer.[1] $H_{NMR}$ spectra were recorded in $CDCl_3$ on Gemini 200 $MH_Z$ instrument using TMS as on internal standard. The mass spectra (GCMS) were recorded on Cintra-15-GCMS-Spectrometer.The elemental analysis (Halogens) was performed on Carlo-Erba-1108 elemental analyser. General procedure for Synthesis 1-(-2'-hydroxy-3-iodo-5-methylphenyl)-3-(-2'-hydroxy-5'-chloro -6'-methylphenyl)-2-propen-1-one.

**Experimental Procedure:** 2'-hydroxy-3-iodo-5-methylacetophenone (0.01mole) and 2'-hydroxy-5-chloro-6-methyl arylaldehyde (0.01mole) was dissolved in ethanol (20ml),under stirring add dropwise aqueous KOH (25 %, 15ml) was stirred for 5min at room temperature and kept 14-16 hours in a bulb oven at 55-60 $^0$C. The reaction mixture was poured over crushed ice and acidified with dil.HCl. The separated solid was filtered and recrystallised from glacial acetic acid to give 1-(-2'-hydroxy-3-iodo-5-methylphenyl)-3-(-2'-hydroxy-5'-chloro -6'-methylphenyl)-2-propen-1-one

| Sr.no. | R | $R_1$ | $R_2$ | $R_3$ | $R_4$ |
|---|---|---|---|---|---|
| 1 | OH | H | OH | H | H |
| 2 | OH | I | OH | I | H |
| 3 | OH | Br | OH | Br | H |
| 4 | H | I | OH | I | H |
| 5 | OH | I | H | Cl | H |
| 6 | OH | I | H | $CH_3$ | H |
| 7 | OH | I | $CH_3$ | Cl | H |
| 8 | H | $CH_3$ | OH | I | H |




**Scheme; Synthesis of polyhydroxy chalcones**

**Table 2: Antimicrobial activity data of pyrazoline derivatives 2(a-e) and 3(a-e)**

| Entry | Zone of inhibition in mm | | % of germination after 12hrs. | |
|---|---|---|---|---|
| | Bacteria | | Fungi | |
| | *S.aureus* | *E.coli* | *H.torulosum* | *A.niger* |
| 3a | 14 | - | - | 10 |
| 3b | -- | 08 | 10 | 10 |
| 3c | 08 | 12 | 12 | 15 |
| 3d | 07 | 12 | 13 | - |
| 3e | 14 | 11 | 10 | 10 |
| 3f | 08 | 13 | 12 | 17 |
| 3u | 08 | 19 | 15 | 14 |
| 3v | 07 | 16 | - | 08 |
| 3w | 15 | 15 | 17 | 12 |
| 3x | 06 | 12 | 14 | 14 |
| Control | 00 | 00 | 00 | 00 |
| Tetracycline | 20 | 20 | -- | -- |

-- = Not show zone inhibition

**Table 2: Physical data of the synthesized compounds[a]**

| Entry | Mol. Formula | Yield[b] (%) | M.P. ($^0$C) | Halogen analysis % Found(Calcu.) |
|---|---|---|---|---|
| | | | | X= Cl, Br, I |
| **3a** | $C_{15}H_{11}O_4Cl$ | 75 | 110-112 | 12.70(12.20) |
| **3b** | $C_{15}H_9O_4ClI_2$ | 70 | 165-167 | 53.82(53.33) |
| **3c** | $C_{15}H_9O_4ClBr_2$ | 65 | 159-160 | 43.94(43.53) |
| **3d** | $C_{15}H_9O_3ClI_2$ | 71 | 164-166 | 54.21(54.94) |
| **3e** | $C_{15}H_9O_3Cl_2I$ | 69 | 185-187 | 45.61(45.97) |
| **3f** | $C_{16}H_{12}O_3ClI$ | 67 | 183-185 | 39.75(39.16) |
| **3g** | $C_{16}H_{11}O_3Cl_2I$ | 61 | 161-163 | 44.96(44.05) |
| **3h** | $C_{16}H_{12}O_3ClI$ | 71 | 196-198 | 39.68(39.16) |
| **3i** | $C_{15}H_{11}O_4Cl$ | 68 | 123-125 | 12.55(12.20) |
| **3j** | $C_{15}H_9O_4ClI_2$ | 70 | 175-177 | 53.81(53.33) |
| **3k** | $C_{15}H_9O_4ClBr_2$ | 65 | 152-154 | 43.11(43.53) |
| **3l** | $C_{15}H_9O_3ClI_2$ | 64 | 158-160 | 55.00(54.94) |
| **3m** | $C_{15}H_9O_3Cl_2I$ | 68 | 176-178 | 45.23(45.97) |

| | | | | |
|---|---|---|---|---|
| **3n** | $C_{16}H_{12}O_3ClI$ | 61 | 170-172 | 39.10(39.66) |
| **3o** | $C_{16}H_{11}O_3Cl_2I$ | 69 | 157-159 | 44.92(44.05) |
| **3p** | $C_{16}H_{12}O_3ClI$ | 64 | 187-189 | 39.00(39.66) |
| **3q** | $C_{16}H_{13}O_4Cl$ | 70 | 115-117 | 11.97(11.63) |
| **3r** | $C_{16}H_{11}O_4ClI_2$ | 65 | 171-173 | 51.43(51.91) |
| **3s** | $C_{16}H_{11}O_4ClBr_2$ | 69 | 187-189 | 42.00(42.22) |
| **3t** | $C_{16}H_{11}O_3ClI_2$ | 61 | 170-172 | 53.15(53.52) |
| **3u** | $C_{16}H_{11}O_3Cl_2I$ | 67 | 181-183 | 44.91(44.05) |
| **3v** | $C_{17}H_{14}O_3ClI$ | 61 | 192-194 | 37.52(37.88) |
| **3w** | $C_{17}H_{13}O_3Cl_2I$ | 70 | 155-157 | 42.31(42.71) |
| **3x** | $C_{17}H_{14}O_3ClI$ | 63 | 120-122 | 37.21(37.88) |

**Spectral data**

**3a.** $^1$H NMR ($CDCl_3$) δ ppm : 6.99 (d, 1H), 7.54 (d, 1H), 6.82-7.63 (m, 6H, Ar-H),7.03 (s, 1H, Ar-OH), 7.14 (s, 1H, Ar-OH), 8.13 (s, 1H, Ar-OH).IR max cm$^{-1}$: 3212 -OH, 3034 C=C-H, 1695 >C=O, 1603, 1507, 1487-C=C-.MS.(m/z): 290($M^+$).

**3b.** $^1$H NMR ($CDCl_3$) δ ppm: 7.02 (d, 1H), 7.63 (d, 1H), 7.22-7.94 (m, 4H, Ar-H), 6.05 (s, 1H, Ar-OH), 8.14 (s, 2H, Ar-OH).IR max cm$^{-1}$: 3244 -OH, 3029 - C=C-H, 1692 >C=O, 1593, 1512, 1476-C=C-.MS.(m/z): 542($M^+$).

**3c.** $^1$H NMR ($CDCl_3$) δ ppm: 7.23 (d, 1H), 7.76 (d, 1H), 7.09-7.84 (m, 4H, Ar-H), 6.23 (s, 1H, Ar-OH), 8.82 (s, 2H, Ar-OH).IR max cm$^{-1}$: 3289 -OH, 3015 - C=C-H, 1691 >C=O, 1535, 1500, 1443-C=C-.MS.(m/z): 546($M^+$).

**3d.** $^1$H NMR ($CDCl_3$) δ ppm: 7.01 (d, 1H), 7.79 (d, 1H), 7.14-8,01 (m, 5H, Ar-H), 6.76 (s, 1H, Ar-OH), 8.01 (s, 1H, Ar-OH).IR max cm$^{-1}$: 3276 -OH, 3018 - C=C-H, 1687 >C=O, 1587, 1518, 1423-C=C-.MS.(m/z): 526($M^+$).

**3e.** $^1$H NMR ($CDCl_3$) δ ppm: 7.12 (d, 1H), 7.72 (d, 1H), 7.09-7.91 (m, 5H, Ar-H), 7.76 (s, 1H, Ar-OH), 8.21 (s, 1H, Ar-OH).IR max cm$^{-1}$: 3289 -OH, 3056 - C=C-H, 1696 >C=O, 1589, 1551, 1445-C=C-.MS.(m/z): 434($M^+$).

**3f.** $^1$H NMR ($CDCl_3$) δ ppm: 2.64 (s, 3H, -$CH_3$), 7.07 (d, 1H), 7.52 (d, 1H), 7.01-7.71 (m, 5H, Ar-H), 7.56 (s, 1H, Ar-OH), 8.11 (s, 1H, Ar-OH).IR max cm$^{-1}$: 3277 -OH, 3021 - C=C-H, 1671 >C=O, 1595, 1536, 1412-C=C-.MS.(m/z): 414($M^+$).

**3g.** $^1$H NMR ($CDCl_3$) δ ppm: 2.51 (s, 3H, -$CH_3$), 6.99 (d, 1H), 7.63 (d, 1H), 7.131-7.52 (m, 4H, Ar-H), 7.87 (s, 1H, Ar-OH), 8.13 (s, 1H, Ar-OH).IR max cm$^{-1}$: 3289 -OH, 3067 - C=C-H, 1697 >C=O, 1598, 1574, 1464-C=C-.MS.(m/z): 448($M^+$).

**3h.** $^1$H NMR ($CDCl_3$) δ ppm: 2.54 (s, 3H, -$CH_3$), 7.08 (d, 1H), 7.23 (d, 1H), 7.13-7.52 (m, 5H, Ar-H), 7.76 (s, 1H, Ar-OH), 8.09 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3265 -OH, 3023 - C=C-H, 1689 >C=O, 1543, 1523, 1468-C=C-.MS.(m/z): 414($M^+$).

**3i.** $^1$H NMR ($CDCl_3$) δ ppm: 7.11 (d, 1H), 7.41 (d, 1H), 7.11-7.61 (m, 6H, Ar-H), 7.76 (s, 1H, Ar-OH), 8.09 (s, 2H, Ar-OH).IR max $cm^{-1}$: 3244 -OH, 3012 - C=C-H, 1687 >C=O, 1587, 1567, 1454-C=C-.MS.(m/z): 290($M^+$).

**3j**. $^1$H NMR ($CDCl_3$) δ ppm: 7.19 (d, 1H), 7.48 (d, 1H), 7.21-7.71 (m, 4H, Ar-H), 7.23 (s, 1H, Ar-OH), 8.43 (s, 2H, Ar-OH).IR max $cm^{-1}$: 3242 -OH, 3042 - C=C-H, 1689 >C=O, 1588, 1556, 1457-C=C-.MS.(m/z): 542($M^+$).

**3k.** $^1$H NMR ($CDCl_3$) δ ppm: 7.11 (d, 1H), 7.64 (d, 1H), 7.26-7.71 (m, 4H, Ar-H), 7.42 (s, 1H, Ar-OH), 8.12 (s, 2H, Ar-OH).IR max $cm^{-1}$: 3242 -OH, 3042 - C=C-H, 1689 >C=O, 1588, 1556, 1457-C=C-.MS.(m/z): 448($M^+$).

**3l.** $^1$H NMR ($CDCl_3$) δ ppm: 7.15 (d, 1H), 7.71 (d, 1H), 7.06-7.62 (m, 5H, Ar-H), 7.32 (s, 1H, Ar-OH), 7.91 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3222 -OH, 3032 - C=C-H, 1689 >C=O, 1578, 1566, 1455-C=C-.MS.(m/z): 526($M^+$).

**3m.** $^1$H NMR ($CDCl_3$) δ ppm : 7.05 (d, 1H), 7.74 (d, 1H), 6.92-7.93 (m, 5H, Ar-H), 9.03 (s, 1H, Ar-OH), 8.13 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3257 -OH, 3021 C=C-H, 1689 =C=O, 1587, 1500, 1480-C=C-.MS.(m/z): 434($M^+$).

**3n**. $^1$H NMR ($CDCl_3$) δ ppm: 2.32 ( s, 3H –$CH_3$), 7.11 (d, 1H), 7.62 (d, 1H), 7.06-7.71 (m, 5H, Ar-H), 7.33 (s, 1H, Ar-OH), 7.81 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3282 -OH, 3052 - C=C-H, 1686 >C=O, 1577, 1566, 1458-C=C-.MS.(m/z): 414($M^+$).

**3o.** $^1$H NMR ($CDCl_3$) δ ppm: 2.52 ( s, 3H –$CH_3$), 7.19 (d, 1H), 7.53 (d, 1H), 7.01-7.75 (m, 4H, Ar-H), 7.23 (s, 1H, Ar-OH), 7.61 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3272 -OH, 3042 - C=C-H, 1685 >C=O, 1579, 1562, 1453-C=C-.MS.(m/z): 448($M^+$).

**3p.** $^1$H NMR ($CDCl_3$) δ ppm: 2.54 (s, 3H –$CH_3$), 7.10 (d, 1H), 7.54 (d, 1H), 7.01-7.75 (m, 5H, Ar-H), 7.12 (s, 1H, Ar-OH), 7.51 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3256 -OH, 3032 - C=C-H, 1685 >C=O, 1567, 1537, 1423-C=C-.MS.(m/z): 430($M^+$).

**3q.** $^1$H NMR ($CDCl_3$) δ ppm: 2.54 (s, 3H –$CH_3$), 7.21 (d, 1H), 7.63 (d, 1H), 7.08-7.75 (m, 5H, Ar-H), 7.12 (s, 1H, Ar-OH), 7.51 (s, 1H, Ar-OH), 8.04 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3246 -OH, 3032 - C=C-H, 1693 >C=O, 1568, 1533, 1423-C=C-.MS.(m/z): 305($M^+$).

**3r**. $^1$H NMR ($CDCl_3$) δ ppm: 2.50 (s, 3H –$CH_3$), 7.20 (d, 1H), 7.61 (d, 1H), 7.07-7.70 (m, 3H, Ar-H), 7.12 (s, 1H, Ar-OH), 7.51 (s, 1H, Ar-OH), 7.14 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3246 -OH, 3022 - C=C-H, 1690 >C=O, 1568, 1533, 1423-C=C-.MS.(m/z): 556($M^+$).

**3s**. $^1$H NMR ($CDCl_3$) δ ppm: 2.48 (s, 3H –$CH_3$), 7.15 (d, 1H), 7.66 (d, 1H), 7.07-7.67 (m, 3H, Ar-H), 7.10 (s, 1H, Ar-OH), 7.57 (s, 1H, Ar-OH), 7.18 (s, 1H, Ar-OH).IR max $cm^{-1}$: 3240 -OH, 3072 - C=C-H, 1691 >C=O, 1568, 1533, 1423-C=C-.MS.(m/z): 462$M^+$).

**3t**. $^1$H NMR ($CDCl_3$) δ ppm: 2.48 (s, 3H –$CH_3$), 7.07 (d, 1H), 7.71 (d, 1H), 7.12-7.77 (m, 4H, Ar-H), 7.09 (s, 1H, Ar-OH), 7.51 (s, 1H, Ar-OH). IR max $cm^{-1}$: 3240 -OH, 3072 - C=C-H, 1691 >C=O, 1568, 1533, 1423-C=C-.MS.(m/z): 540$M^+$).

**3u**. $^1$H NMR ($CDCl_3$) δ ppm: 2.51 (s, 3H –$CH_3$), 7.11 (d, 1H), 7.61 (d, 1H), 7.07-7.77 (m, 4H, Ar-H), 7.19 (s, 1H, Ar-OH), 7.41 (s, 1H, Ar-OH). IR max $cm^{-1}$: 3271 -OH, 3042 - C=C-H, 1691 >C=O, 1566, 1543, 1422-C=C-.MS.(m/z): 550$M^+$).

**3v**. $^1$H NMR ($CDCl_3$) δ ppm: 2.49 (s, 3H –$CH_3$), 2.52 (s, 3H –$CH_3$), 7.09 (d, 1H), 7.51 (d, 1H), 7.01-7.67 (m, 4H, Ar-H), 7.12 (s, 1H, Ar-OH), 7.91 (s, 1H, Ar-OH). IR max $cm^{-1}$: 3281 -OH, 3051 - C=C-H, 1690 >C=O, 1568, 1543, 1422-C=C-.MS.(m/z): 428$M^+$).

**3w.** $^1$H NMR ($CDCl_3$) δ ppm: 2.44 (s, 3H –$CH_3$), 2.50 (s, 3H –$CH_3$), 7.02 (d, 1H), 7.57 (d, 1H), 7.11-7.81 (m, 3H, Ar-H), 7.11 (s, 1H, Ar-OH), 7.61 (s, 1H, Ar-OH). IR max $cm^{-1}$: 3287 -OH, 3058 - C=C-H, 1691 >C=O, 1567, 1533, 1412-C=C-.MS.(m/z): 462$M^+$).

**3x.** $^1$H NMR ($CDCl_3$) δ ppm: 2.54 (s, 3H –$CH_3$), 2.51 (s, 3H –$CH_3$), 7.07 (d, 1H), 7.77 (d, 1H), 7.11-7.71 (m, 3H, Ar-H), 7.01 (s, 1H, Ar-OH), 7.69 (s, 1H, Ar-OH). IR max $cm^{-1}$: 3288 -OH, 3058 - C=C-H, 1681 >C=O, 1557, 1533, 1419-C=C-.MS.(m/z): 428$M^+$).

## RESULTS AND DISCUSSION

The chemistry of hydroxyl chalcones has generated an intensive scientific interest due to their biological and industrial applications. An eminent feature of hydroxyl chalcone is that, they serve as the starting material for the synthesis of different classes of flavonoids, viz.

flavanones, flavones, flavanol and also versatile intermediate for the preparation of various heterocycles viz. pyrazolines, phenylpyrazolines, chalconimines and pyrimidines.

In the present content the polyhydroxy chalcones were synthesized by employing Claisen-Schmidt condensation. The method has been implemented due to the merits. It has moderate reaction conditions excellent yield of the product and workup, isolation is easier. The equimolar quantities of 2'-hydroxy substituted acetophenone and 2'-hydroxy aryl aldehyde was dissolved in ethanol, 25% of KOH was added in portion at room temperature. The reaction flask was loosely corcked and kept at $60^0$C in a bulb oven for 14-16 hours. The contents of the flask were acidified with HCl and poured over crushed ice. The solid filtered and washed with cold water and recrystallied from acetic acid.

To study the generality of this process, variety of examples were illustrated for the synthesis of polyhydroxy chalcones and results are summarized in Table 1. The reaction is compatible for various substituents such as $CH_3$, OH, Cl, Br and I. The formation of desired product was confirmed by $^1$H NMR, IR and mass spectroscopic analysis technique. Also the melting points were recorded and compared with the corresponding literature data.

It is evident that the Claisen-Schmidt condensation method is excellent yield with enhanced antimicrobial activity.

**Antimicrobial Activity**

The information regarding the various species of bacterial used to carry out screening is given below

**(i) *Escherichia coil (E. coil)***

*Escherichia coil* are gram –negative bacteria they occur in lower portion of the intestine and urinary tract. They causes urinary tract infection, some strains can cause gastro-enteritis.

**(ii) *Staphylococcus aureus (S. aureus)***

*S. aureus* are the gram positive non motile cocci arranged in groups. They are parasites in the skin an mucous membranes occurring of human and animals.

*S. aureus* produces many toxins that contribute to the bacetrium's pathogenicity by increasing its ability toinvade the both or damage tissus. *S. aureus* is the again of toxic shock syndrome a severe infection causing high fever and vomiting and sometimes death. It also produces an entertoxin that cause vomiting and nausea when ingested and is one of the most common of food poising.

For establishment of antimicrobial activity of the synthesized compounds, we utilized the reported disk diffusion method. The experiment was performed at a concentration of 150 ppm, we checked the activity of these molecules against different strains of bacteria and fungi as mentioned in Table 2. 10%DMSO was used as solvent control.

**Antifungal activity**

In the present investigation some of the new synthesized pyrazolines were assessed for their antifungal activity against fungi like *Aspergillus nigar (A.niger), Helminthosporium torulosum (H.toulosum).* The assessment of activity of spore germination method in petridish was fallowed. Spore suspension was prepared from seven days old PDA (Potato dextrose agar) slant cultures. Spore suspensions were placed in small's petridishes. Solutions of different synthesized compounds were prepared in 90:10(V/V) water ethanol and the concentration of compounds (150ppm) was adjusted in spore suspension. Petridishes were placed for incubation period of 12hours under moist chambers. Aqueous ethanol (90:10 v/v) served as control. Percentage germination with effect of these compounds after a period of 12 hours was recorded by observing Petridis directly under microscope.

The obtained data of activity of all these tested compounds is shown in Table 2.

## CONCLUSION

In conclusion, we have described a general and highly efficient procedure for the preparation of polyhydroxy chalcones derivatives. The remarkable advantage of this protocol is mild reaction conditions, excellent yields of product, operational and experimental simplicity. We believe that, this methodology will be a valuable addition to the existing methods for the synthesis of polyhydroxy chalcones. These polyhydroxy chalcones were shown good antimicrobial activity and also used as starting material for synthesis of various heterocyclic compounds

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