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## Synthesis, Characterization and Biological activity of some novel chalcone compounds and their Cobalt Nickel complexes having benzyloxydibromo resacetophenone moiety

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### ABSTRACT

*1-(3,5-dibromo-2,4-dihydroxyphenyl)ethanone (HDBA) and chalcones were prepared by the coupling of benzyle bromide and 3,5-dibromo resacetophenone then condensation with aromatic aldehyde(3a-j) then covert them to its metal complexes. The newly synthesized compounds were evaluated for their characterization, their physical properties and their biological activity.*

**Keywords:** -dibromo-benzyloxy-resacetophenone, ketone aldehyde condensation, chalcones, phenone derivates antimicrobial activity, chalcone metal complexes, cobalt complexes, nickel complexes.

### 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, chalcones are colour compounds. These compounds exhibit high reactivity due to  $\alpha:\beta$ -unsaturated un saturation present in the compounds. Chalcone is also known as 1,3-disubstituted-2-propene-1-ones. Kostanekci and Tambor<sup>1</sup> 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], ploretin[4], 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.

### Antimicrobial activity

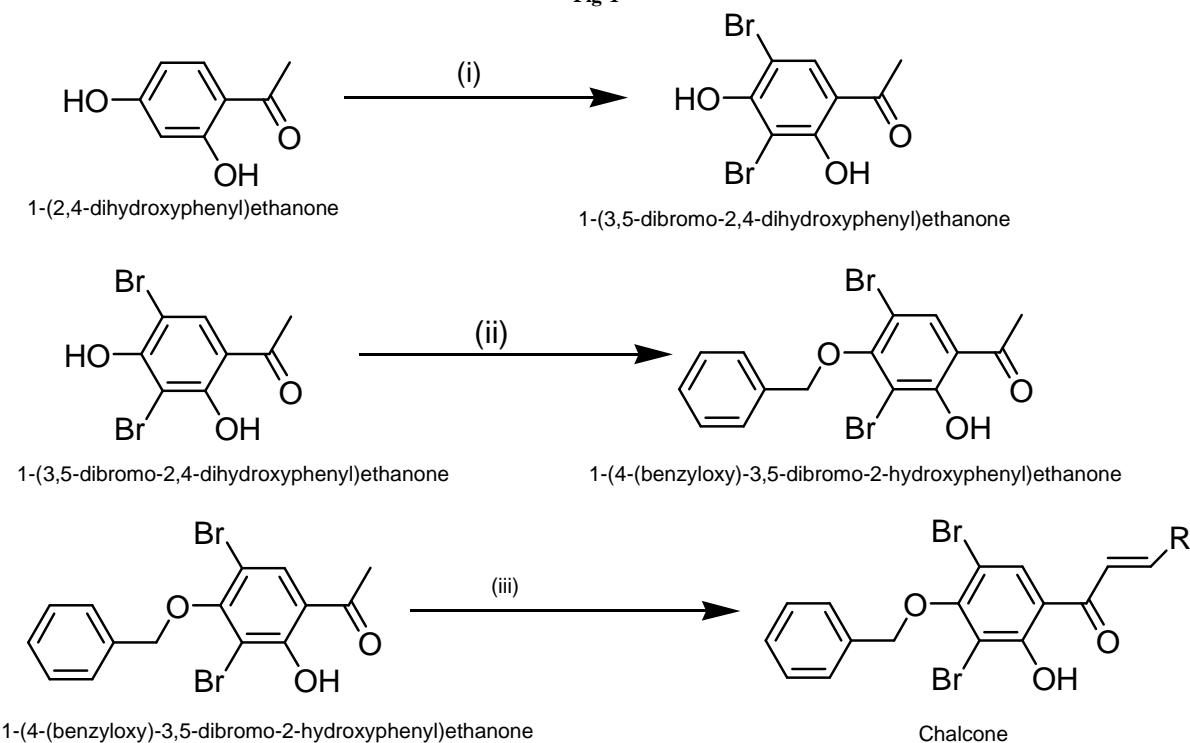
During the present century, chalcones and their derivatives are found to be much in use. Thus, some chalcones exhibited therapeutic properties eg. antiulcer activity, hypotensive activity etc. Antibiotic activity[10-11] have been shown by some chalcones due to presence of an enone function. It has been observed that the bacteriostatic or bactericidal properties get increased with the introduction of substituents like a nitro, bromo group at the  $\alpha$ -position or a bromo or hydroxyl group at the  $\beta$ - position[10]. Some substituted chalcones and their derivatives possess biological properties eg. the growth of microbes[12], tubercle bacilli[13-14], malarial parasites[15], intestinal worms[16] etc. They also inhibit growth of several enzymes[17] and fungi[18-19]. Hypotensive property is associated with some chalcones[20]. Few substituted chalcones were tested for ability to protect adrenaline from destruction[21].

In heterocyclic derivatives of chalcones, isoxazolines have shown good antimicrobial, antitubercular[22], antiviral[23] and antifungal activities[24]. Pyrazolines are important nitrogen containing heterocycles possessing diverse biological activity[25-27]. Some pyrazoline derivatives have shown considerable promise as chemotherapeutic agents.

Doshiet al[28] reported some cyanopyridines as a potential antitubercular agents. Pyrimidine derivatives occupy a unique position as leiodynamic agents. Both are essential components of nucleic acid and also as therapeutic agents[29-30]. Some chalcone derivatives have been reported as anti inflammatory or antiallergic agents[31]. Furthermore, they found that chalcones with a 3,4-dihydroxy cinnamoyl structure strongly inhibited lipid peroxidation in cat liver microsomes[32]. The 3,4-dihydroxy chalcones are rapidly and extensively metabolized the systematic administration. These finding suggest that the chalcones may be promising non-toxic topical anti-inflammatory agents.

### MATERIALS AND METHODS

**Fig-1**



**Scheme 1** Reagents and conditions: **i**)Bromination, S/Ethanol; **ii**) K<sub>2</sub>CO<sub>3</sub> & benzylebromide/S: Ethanol; **iii**) Aldehyde & 50-60% NaOH in S/Ethanol; **(a)** R=Benzaldehyde; **(b)** R=4-Bromobenzaldehyde; **(c)** R=4-Chlorobenzaldehyde; **(d)** R=Anisaldehyde; **(e)** R=3-Chlorobenzaldehyde; **(f)** R=2-Chlorobenzaldehyde; **(g)** R=4-(N,N-dimethylamino)benzaldehyde; **(h)** R=3-Bromobenzaldehyde; **(i)** R=2-Hydroxy benzaldehyde; **(j)** R=3-hydroxy benzaldehyde;

Melting points were determined in open capillary tubes and are uncorrected. The IR spectra were recorded in KBr pellets on a Beker spectrometer and 1 H NMR spectra in CDCl<sub>3</sub> on Hitachi R-1500, 60 MHz spectrometer using TMS as an internal standard. All chemicals used were of laboratory grade.

Preparation of 1-(3,5-dibromo-2,4-dihydroxyphenyl)ethanone (HDBA) and chalcone is as given in fig 1.

#### **Synthesis of 1-(3,5-dibromo-2,4-dihydroxyphenyle) ethanone (HDE) (i)**

Take 1-(2,4-dihydroxyphenyl)ethanone(0.10 mole) in ethanol(20%) Than make Br<sub>2</sub> solution in the chloroform. Cool the reaction mixture up to <5°C than add drop wise previously prepared Br<sub>2</sub> solution with vigorous stirring. After completion of addition allow to stay the reaction mixture for 3 hour under stirring. After the completion of the reaction (By TLC), allow the reaction mass to concentrate (by evaporation), after some time the crystalline material of HDE observed. Cool the mass filter it and wash with cold solution of ethanol.

#### **Synthesis of 1-(4-Benzyl-3,5-dibromo-2-hydroxyphenyle) ethanone (HDBA) (ii) :**

1-(3,5-dibromo-2,4-dihydroxyphenyle) ethanone (HDE) (0.10 mole), Benzylebromide (0.12 mole) and Potassium carbonate (0.15 mole) were taken in 500ml of Acetone. The reaction mixture was stirred for 6-8 hours at reflux temperature. The reaction mass was cooled to 15 – 20°C temperature and quenched with approx 700ml water. The resulting product 1-(4-Benzyl-3,5-dibromo-2-hydroxyphenyle) ethanone (HDBA) was filtered and washed with water. The obtained product was recrystallized in ethanol.

#### **Preparation of chalcone (3a-j)**

Chalcones can be prepared by the most convenient method available in literature involves the Claisen-Schmidt condensation of equimolar quantities of a substituted acetophenone with substituted benzaldehydes in the presence of aqueous alkali. In this reaction the concentration of alkali used is ranged between 10 to 60%.<sup>17,18,23,32,41</sup>

Following the above procedure, the attempts have been made to prepare chalcones from HDBA using various aromatic aldehydes. On the basis of the results, it was found that chalcones could easily be prepared from HDBA using aromatic aldehydes at room temperature after several hours.

In some cases it was needed to establish the procedure for preparing chalcones from HDBA using some aldehydes, different reaction conditions were applied. From the results, it was found that the chalcones could be prepared from HDBA using some aldehydes by refluxing the reaction mixture at 75 – 80°C temperature for 3 hours.

#### **1-(4-Benzyl-3,5-dibromo-2-hydroxyphenyle) ethanone (HDBA) (3)**

m.p 158- 160°C; Mass; 400.06 ; IR(KBr cm-1): 2980(C-H str. vib.) 3067(-Aromatic C-H),1571, 1469,(C=C str.Vib.),879(-C – H o.m.m.p multi sub. benzene),1240, 1031(C-O-C str.vib), 3428(O-H str.vib), 1622(-C=O str.vib), 554(C-Br str.vib),; 1H NMR 7.30 – 7.8 (s,6H,of the Ar-H ),12.8 (s,1H, Ar-OH), 5.02 (2H,s, -CH<sub>2</sub>-O-), 2.5 (3H,s, O=CCH<sub>3</sub>); Yield 58%;

#### **1-(2-hydroxy-3,5-dibromo-4-Benzyl-2-hydroxyphenyl)-3- phenylprop-2-en-1-one [3a]:**

m.p 141-145°C; Mass; 488.17 ; IR(KBr cm-1): 2869(C-H str. vib.) 3053(-Aromatic C-H),1569, 1460,(C=C str.Vib.),871(-C – H o.m.m.p multi sub. benzene),1238, 1029(C-O-C str.vib), 3480(O-H str.vib), 1633(-C=O str.vib), 571(C-Br str.vib),; Yield 60.33%;

#### **1-(2-hydroxy-3,5-dibromo-4-benzyl-2-hydroxyphenyl)-3-(4-bromophenyl) prop-2-en-1-one [3b]:**

m.p 179-181°C; Mass; 567.06 ; IR(KBr cm-1): 2848(C-H str. vib.) 3041(-Aromatic C-H),1574, 1432,(C=C str.Vib.),873(-C – H o.m.m.p multi sub. benzene),1240, 1021(C-O-C str.vib), 3464(O-H str.vib), 1626(-C=O str.vib), 613(C-Br str.vib),; Yield 59.13%;

#### **1-(2-hydroxy-3,5-dibromo-4-benzyl-2-hydroxyphenyl)-3-(4-chlorophenyl) prop-2-en-1-one [3c]:**

m.p 167-169°C; Mass; 522.61 ; IR(KBr cm-1): 2833(C-H str. vib.) 3030(Aromatic C-H),1565, 1440,(C=C str.Vib.),854(-C – H o.m.m.p multi sub. benzene),1238, 1013(C-O-C str.vib), 3447(O-H str.vib), 1636(-C=O str.vib), 611(C-Br str.vib),; Yield 61.42%;











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