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RESEARCH ARTICLE

Studies on Chalcones: Part-1≠. By green process and evaluation of biological activity

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ABSTRACT

Chalcones were prepared by condensation (Claisen Schmidt) reaction of Acetophenone, o-Hydroxy-acetophenone with Benzaldehyde, employing conventional and microwave assisted-solvent free method. This reaction was carried out with use of fresh anhydrous Na_2CO_3 as catalyst by avoiding hazardous bases like NaOH and KOH for the condensation process which is ecofriendly and covers Green chemistry aspects. Their biological Activities were also evaluated.

Keywords: Microwaves, anhydrous Na_2CO_3, Green chemistry, ecofriendly method, Chalcone and Claisen Schmidt condensation.

INTRODUCTION

From the survey of the literature, it is marked that varied newer methods have been tried to study the chemical reactions of different type. Environmentally, the development of technology is approaching towards environmental and ecofriendly methods. The usage of microwave energy to accelerate the organic reactions is of increasing advantageous over conventional method. The time required for synthesis of molecules requires a short time by microwave oven, easy efficient work up and cleaner products are the major advantages of heat saving. Further, solvent free condition, reactions can be carried out, as the solvents are toxic, requires more safety and expensive also. Hence, utilization of microwave energy serves the part of green chemistry in the synthesis.

≠ **Partly presented orally (OP-34) at National Conference at DNCVP College, Jalgaon on 20th June 2015.**

The chalcones are aromatic compound in which two aromatic rings are linked by a three carbon α,β-unsaturated carbonyl(-CO-CH=CH-) system. IUPAC name of chalcone is 1, 3-diphenyl-prop-2-en-1-one, in this, it possesses conjugate double bonds and a delocalized electron on both benzene ring [1]. Chalcones and compounds with the backbone of chalcone possesses *an* unsaturated carbonyl group are one of the important biocides and versatile synthons for different reactions and most of the chalcones are biologically active and been used as antimicrobial[2], anti-inflammatory[3], anti-ulcer[4], antimalerial[5] and anti-tubercular[6]. Keeping in view the advantages of microwave heating and its biological activities we targeted the present investigation, for synthesis of some substituted o-hydroxy chalcones by claisen-schimdt condensation using the microwave reaction. Chalcones are condensed products of substituted aromatic with simple or various substituted acetophenone in the presence of alkali such as KOH or NaOH [7, 8] which are harmful, toxic and pollution causing. We have used the condensing agent which is cheap i.e.; *sodium carbonate (Na_2CO_3)*, which is non-toxic and easy to handle.

EXPERIMENTAL

All the chemicals (Acetophenone, o-Hydroxy-acetophenone, Benzaldehyde, n-Hexane and Ethylacetate) and reagent(Na_2CO_3) used were (of synthesis grade) purchased from the SIGMA-ALDRICH and are used as supplied without further purification. The progress of the reaction and purity of the chalcone-**I** and **II** was checked by TLC on silica gel glass plates. The reaction was carried out in domestic microwave oven (Microwave oven, Power = 100). The melting points reported are uncorrected and were taken in open capillaries. The UV-Vis spectra were

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recorded in absolute alcohol, on Shimadzu (UV-1800). The FTIR spectra were recorded by using KBr pellets on a FTIR Spectrophotometer (Shimadzu, 4000-400 cm^{-1}).

SYNTHESIS OF CHALCONES I and II:

Acetophenone (0.02 mol) and benzaldehyde (0.018 mol) and 0.15 moles of anhydrous Na_2CO_3 were mixed forming a thick paste, air dry it and the formed mass was subjected to microwave irradiation for 4 - 6 min. All the reaction contents were dissolved in solvent alcohol. The inorganic solids were filtered off and filtrate is then concentrated under vaccum. It was left overnight to get the chalcone-**I**. Similarly chalcone-**II** was synthesized using o-Hydroxy-acetophenone instead of acetophenone.

Antibacterial activity:

The in vitro Antimicrobial activity of synthesized chalcone-**I** and **II** [dissolved in dimethylsulfoxide (DMSO)] were determined by disc diffusion and the zone of inhibition (ZOI) method. [8b]. Chloramphenicol was used as positive control for bacteria. All human pathogenic bacteria viz *Staphylococcus aureus* was procured from National Chemical Laboratory, Pune. The agar medium was purchased from Hi-media Laboratories Ltd., Mumbai, India. Preparation of nutrient broth, subculture, base layer medium, agar medium and peptone water was done as per the standard procedure. Discs measuring 6.25 mm in diameter were punched from Whatman No.1 filter paper. Stock solutions of synthesized compounds diluted in dimethyl sulphoxide (1% DMSO) to give final concentration of 500 µg/ml and 1000 µg/ml. A reference standard for was made by dissolving accurately weighed quantity of chloramphenicol [500 and 1000 µg/disc(0.5 mm), respectively] in sterile distilled water, separately. Then, the paper discs impregnated with the solution of the compound tested were placed on the surface of the media inoculated with the microorganism. The plates were incubated at 37ºC for 24 h. All the experiments were carried out in duplicate. Simultaneously, controls were maintained DMSO (by employing 0.1 mL) which did not reveal any inhibition. The zone of inhibition produced by each compound was measured in mm. The distinct differences in the antibacterial properties of these compounds further justify the purpose of this study. The importance of this work lies in the possibility that the new compound might be more efficacious drugs against bacteria and a more thorough investigation regarding the structure–activity relationships, toxicity and in their biological effects could be helpful in designing more potent antibacterial agents for therapeutic use.

RESULTS AND DISCUSSION

The Claisen-Schmidt condensation is an important C-C bond formation for the synthesis of Chalcones, viz. [1,3-Diphenyl-prop-2-en-1-one]. It is generally carried out by the use of strong bases such as NaOH or KOH in polar solvents (MeOH or DMF). Synthesis of chalcone is a single step method. The yields of the synthesized compounds were found to be significant. The structure of the synthesized compounds was confirmed by TLC, UV-Vis and FTIR analysis. The progress of the reaction is monitored by TLC using the mobile phase ethyl acetate and n-hexane (4:6) solvent system on silica gel plates, the spots on the plates were identified by iodine chamber and U.V lamp used as visualizing agents. The reaction gave a single spot indicating the completion of reaction and the results (R_f values) obtained are depicted in **Table-1**. The Physical constant of the synthesized chalcones are in concurrence with the literature reports.

The results of UV-Vis spectra are depicted in **Fig.-1** and **Fig.-2** for the respective chalcone-**I** and –**II**, indicates presence of the hydrogen bonding and hence there are only two peaks as compare to Chalcone-**II** as there are four peaks. The synthesized and studied compounds give the characteristic FTIR peak and the obtained results are as depicted in **Table-1**. This data proved that the presence of particular functional group in the synthesized compound.

Table-1: Physical properties and Spectral data(UV-Vis and FTIR) of Chalcones, I and II synthesized by conventional method.

Sr. No.	R_f Value	Colour of Product	m.p.	UV-Vis λ_{max}.in nm	FTIR absorption valves, in cm^{-1}
I	0.73	Cream	56–57°C[9]	208.5 240.0 288.0 308.0	1662 $\nu_{-C=O}$; 1590 $\nu_{->C=C<}$ (olefinic); 1572, 1463 $\nu_{->C=C<}$ (aromatic); 2351 ν_{-C-H};
II	0.84	White	88-90°C	207.5 251.0	1669 $\nu_{-C=O}$; 1585 $\nu_{->C=C<}$ (olefinic); 1569, 1448 $\nu_{->C=C<}$ (aromatic); 2355 ν_{-C-H}; 3085 ν_{-O-H}

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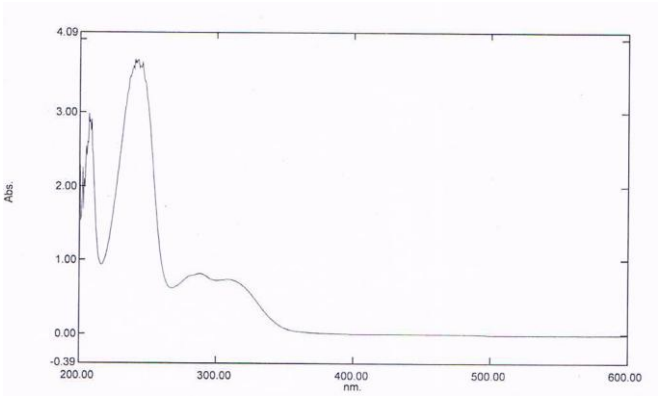


Fig.-1: UV-Vis Spectra of I i.e (E)-1-(2-Hydroxyphenyl)-1-phenylprop-2-en-3-one

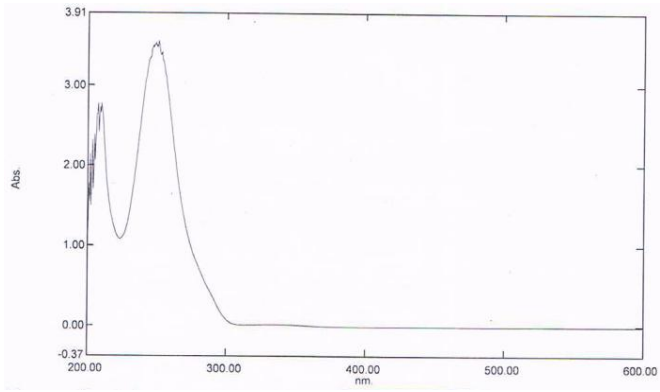
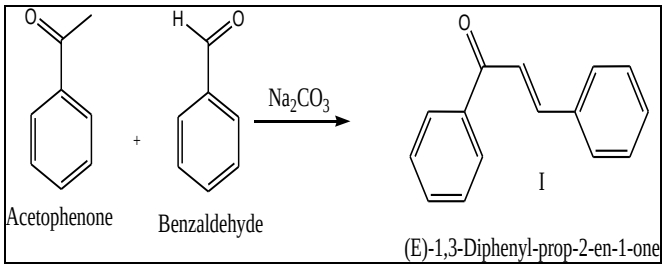


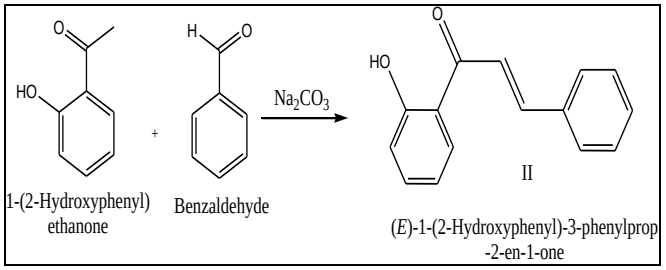
Fig.-2: UV-Vis Spectra of II i.e (E)-1-(Phenyl)-1-phenylprop-2-en-3-one

Based on the results obtained and above discussion the proposed structures of the studied chalcone-**I** and –**II** are as shown below. **Fig.-3** shows the diagram of recrystallization of chalcone-**I**.

Reaction of Acetophenone and Benzaldehyde: Scheme-I



Reaction of 2-Hydroxy-Acetophenone and Benzaldehyde: Scheme-II



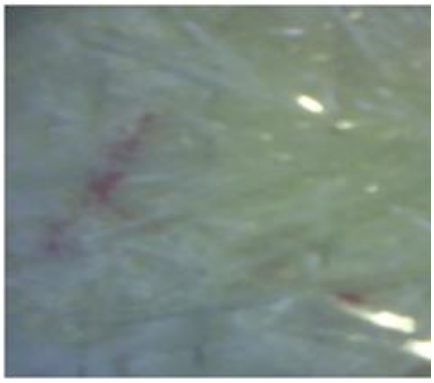


Fig.-3: The crystal form of Chalcone-II after recrystallization.

The study of zone of inhibition of the chalcone-**I** and –**II** gave the results and revealed that the synthesized compound showed different degrees of inhibition against Gram positive bacteria shown in Table 2. The **II** showed excellent activity against *S. aureus* at both concentration i.e. 500 µg/ disc and 1000 µg/disc. The compound **I** have shown good to moderate activity against *S aureus* at concentration i.e. 500µg/disc and 1000 µg/disc. The size of the zone of inhibition is usually related to the level of antimicrobial activity present in the sample or product - a larger zone of inhibition usually means that the antimicrobial is more potent.

Table 2: Antibacterial activity of the synthesized chalcone, positive control, chloramphenicol and negative control, DMSO measured by the Halo Zone Test (in mm).

Compounds	**Antibacterial activity for *Staphylococcus aureus* (% inhibition) in mm**	
	500 µg/disc	1000 µg/disc
I	16.9	21.0
II	23.4	31.8
Chloramphenicol	38.1	48.2
DMSO	-	-

The graphical representation of the antibacterial activity is depicted in **Fig. 4**. indicated the graphical representation of Antibacterial Activity of synthesized chalcones against *S. aureus* after 24 hrs.

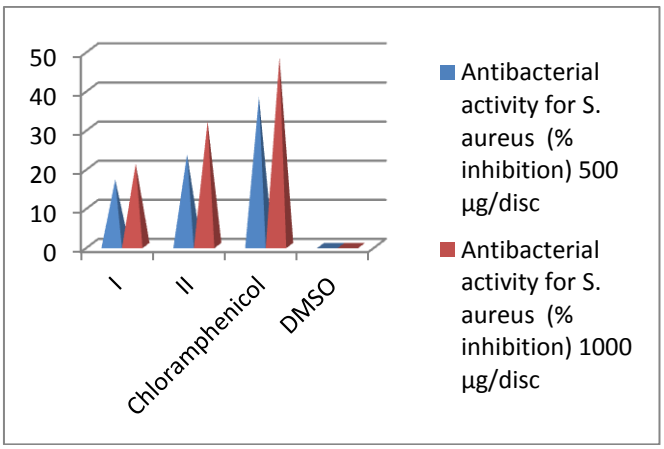


Fig. 4: Antibacterial Activity of synthesized Chalcones against S. aureus after 24 hrs.

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Glimpses of the Activity:

- Compound, **I** and **II**, both are active against *S. aureus* at higher concentration (respective at 500 and 1000 □g/disc).
- Compound, **II** is more active than compound, **I,** at all the studied concentration
- Compounds, **I** and **II** are less active as compare to the standard drug, Chloramphenicol**,** at all the studied concentration.

CONCLUSION

The present study reports the successful synthesis and antifungal activity of Chalcone compounds. These compounds will be useful as synthone or building block by organic researchers in the near future.

The observed antifungal activity of all the compounds have been studied which may be due to the >C=CH-CO- linkage of the chalcone. The results of antifungal activity were compared with that of the standard drug, Chloramphenicol.

Scope: There is a future scope for using these compounds for the organic transformations and screening of these compounds against different microorganism and the data obtained will be useful for the society to study their further studies for Budding Organic chemist and the other Researchers.

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