International Journal of Science and Research (IJSR)
ISSN (Online): 2319-7064
Index Copernicus Value (2013): 6.14 | Impact Factor (2013): 4.438

Green Synthesis and Characterization of Some Substituted Dihydroxychalcones

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Abstract: ***The present communication, reports a series of novel chalcones synthesized from 2,4-dihydroxy / 2,5-dihydroxy acetophenone with substituted benzaldehydes by Claisen-Schimidt base catalyzed condensation and characterized by T.L.C. studies, elemental analysis, conductivity measurements, I.R. spectral studies. The aim of this study is to procure various dihydroxychalcone derivatives in the hope of obtaining new agents, which might show biological activities with improved potency.***

Keywords: Substituted dihydroxychalcone, Microwave irradiation, Claisen-Schmidt condensation, Elemental analysis, IR spectra

1. Introduction

Chalcone represent an important group of natural and synthetic compounds with numerous biological activities. Chemically they are open chain flavonoides of 1, 3-diphenyl-2-propene-1-one skeleton in which two aromatic rings are linked by a three carbon α, β-unsaturated carbonyl system.Screening of the literature reveals that depending on the substitution pattern on the two aromatic rings, a wide range of pharmacological activities have been identified for various chalcones.These include antimicrobial[1-4], anti-inflammatory[5], anti-ulcer [6], anti-cancer[7], anti-oxidant and /or free radical scavenger activity[8-9],anti-convulsant [10-11], anti-fungal [12], analgesic[13], anti-feedent [14], anti-malarial[15], anti-HIV activity[16], anti-histaminic [17],anti-viral [18],and anti-hyperglycemic [19]etc.

A search through the literature reveals that the synthesis and the biochemical potentialities of hydroxychalcones derived from hydroxy acetophenone and substituted aromatic aldehyde generated a high interest among the chemist.Structure of chalcone is flexible which easily allows for structural modification. Skillful structural manipulation of chalcone framework may yet narrow its range of biological activity and enhance its potency for a targeted pharmacological activity. Chalcones are very readily synthesized,and various substitution patterns can beattempted on the two aromatic rings to give a largenumber of potential analogues. Compounds with electron releasing groups such as methoxy and hydroxylshowed better antibacterial activity than the others not having such groups. Compounds having pharmacophores such as chloro, dichloro and fluoro groups have exhibited more antifungal activity on the fungi.Chalcone derivatives with these substituents showing greater antimicrobial activity. [20].

In recent years, microwave irradiation (MWI) and ultrasound irradiation [21]assisted solid support-solvent free organic synthesis have attracted attention as non-conventional technique for rapid organicsynthesis. MWI makes the process eco-friendly, economic with excellent yield and makes a new path ingreen chemicaltransformation [22].In present paper, we report microwave induced synthesis (green synthesis) of substituted dihydroxy chalconein hope of developing some new compounds which might be used as intermediates in synthesis of various classes of bioactive compounds.

2. Material and Methods

All reagents (AR grade) used were purchased from sigma-aldrich co. and HiMedia Laboratories Ltd. Mumbai, India, and used without further purification. Elemental analysis was performed on a Perkin-Elmer 240 CHNElemental analyzer. The IR spectra were recorded on Perkin-Elmer Spectrum Version 10.4.3 (KBr). The progress of reaction and purity of the compounds was checked byTLC on silica gel G plates using benzene:ethyl acetate (9:1) solvent system.

3. Experimental Procedure

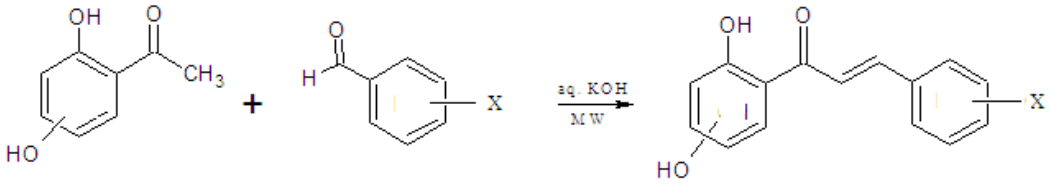
Chalcones were synthesized by Claisen-Schmidt condensation of equimolar (.002M) quantity of 2', 4'-dihydroxy acetophenone and substituted benzaldehyde with 3 ml of alcohol in a conical flask. To this, few drops of aqueous KOH (.003M) solution was added slowly and mixed. The flask was covered with funnel and then taken in a domestic microwave oven. The mixture was irradiated under 180W for 120 to 150 seconds. The progress and completion of reaction was monitor by TLC. The mixture was cooled, washed with ice cold water and neutralized by ice-cold HCl (10%, 6ml). The chalcone precipitate out as solid. The separated solid was filtered and washed with ice cold water till the washing was neutral to litmus. Recrystalized the compound with ethanol and dried at room temperature. The compounds were obtained in yield ranging from 70% to 95%. The physicochemical data and spectroscopic data for the synthesized compounds are given in Table (1-2).

Table 1: Physicochemical Characterization Data for Synthesized Compound

Comp. No.	Molecular Formula	Substituent (Ring A)	Substituent X (Ring B)	Molar Conductance $ohm^{-1} cm^2$ /mole	Founded (calculated) %	
					C	H
1	$C_{15}H_{12}O_3$	5-OH	-	3.50	75.30(75.00)	5.08(5.00)
2	$C_{15}H_{11}O_3F$	5-OH	2-F	3.75	69.60(69.77)	4.11(4.18)
3	$C_{15}H_{11}O_3F$	5-OH	4-F	3.90	69.68(69.77)	4.45(4.18)
4	$C_{15}H_{11}O_3Cl$	5-OH	2-Cl	4.70	65.48(65.57)	3.96(4.02)
5	$C_{15}H_{11}O_3Cl$	5-OH	4-Cl	4.50	65.55 (65.57)	4.20 (4.02)
6	$C_{15}H_{11}O_3Br$	5-OH	4-Br	4.10	56.45(56.51)	3.60 (3.45)
7	$C_{15}H_{12}O_3$	4-OH	-	4.50	75.30 (75.00)	5.20 (5.07)
8	$C_{15}H_{11}O_3F$	4-OH	2-F	3.70	69.60(69.77)	4.40(4.18)
9	$C_{15}H_{11}O_3F$	4-OH	4-F	3.95	69.76(69.77)	4.59(4.18)
10	$C_{15}H_{11}O_3Cl$	4-OH	2-Cl	4.60	65.56(65.57)	4.06(4.02)
11	$C_{15}H_{11}O_3Cl$	4-OH	4-Cl	3.80	65.60(65.57)	4.40(4.02)
12	$C_{15}H_{11}O_3Br$	4-OH	4-Br	4.20	56.45 (56.51)	3.47(3.45)
13	$C_{15}H_{12}O_4$	4-OH	4-OH	5.90	70.82 (70.31)	4.50 (4.69

Table 2: IR stretching frequencies of the Functional group of the chalcone

Comp. No.	Compound name	(-OH at 2',4'/ 2',5' position)(cm^{-1})	(>C=O) (cm^{-1})	(C=C)(Ali.) (cm^{-1})
1	2',5'-dihydroxychalconeone	3340	1686	1650
2	2-fluoro, 2',5'-dihydroxychalcone	3310	1674	1565
3	4-fluoro, 2',5'-dihydroxychalcone	3350	1678	1595
4	2-chloro, 2,5-dihydroxychalcone	3280	1665	1605
5	4-chloro, 2',5'-dihydroxychalcone	3275	1682	1598
6	4-bromo, 2',5'-dihydroxychalcone	3350	1665	1600
7	2',4'-dihydroxychalcone	3350	1690	1550
8	2-fluoro, 2',4'-dihydroxychalcone	3162	1626	1603
9	4-fluoro, 2',4'-dihydroxychalcone	3294	1681	1589
10	2-chloro, 2',4'-dihydroxychalcone	3275	1672	1630
11	4-chloro, 2',4'-dihydroxychalcone	3292	1603	1518
12	4-bromo, 2',4'-dihydroxychalcone	3300	1690	1592
13	4-hydroxy, 2',4'-dihydroxychalcone	3171	1655	1580



Scheme 1: Synthetic diagram of substituted dihydroxychalcone

4. Result and Discussion

The synthesis of the chalcones was accomplished according to the Claisen-Schmidt condensation of methyl ketones with appropriate aromatic aldehyde under microwave irradiation. The corresponding reactions proceeded smoothly and in good to excellent yields (65-95 %). Synthesis of chalcone is a single step process. The synthesized chalcone derivatives were undergone physicochemical characterization. All the chalcones were confirmed by positive Wilson test [23], red or pink coloration with Conc.H_2SO_4.Elemental analysis showed that the percentage the carbon and hydrogen were foundexperimentally in agreement with the calculated values in all compounds.The low values of molar conductance of all the hydroxy chalcones suggest the non-electrolytic nature of the chalcones (Table 1). All the compounds give the characteristic IR peak that proved the presence of particular functional group. It is quite evident there are two different types of –OH groups present in different molecules of the chalcones. The stretching frequencies of >C=O groups in different chalcone lie between 1700-1600 cm^{-1} and the conjugated carbon-carbon double (aliphatic) bond at 1650-1500 cm^{-1} (Table 2).

5. Conclusion

In this work, we have demonstrated the synthesis of chalcones using microwave irradiation.The advantage ofthis method are high yields, short reaction times, low cost, simple experimental and isolation procedures. The synthesized compounds were characterized by TLC, melting point, elemental analysis and IR spectroscopy.The results confirmed that the product has formed. The data obtained during the study will be certainly useful to go for further research for synthesizing new chalcone derivatives.

6. Acknowledgment

The authors express their a deep sense of gratitude to the Principal, Dr. D. K. Bhasin and Dr. S. P. Mital, Head, Department of Chemistry, D.A.V. (P.G.) College, Dehradun for providing the necessary facilities and constant

encouragement. Authors are also thankful to University Grant Commission, New Delhi for financial support.

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