International Journal of CURRENT PHARMACEUTICAL RESEARCH
ISSN- 0975-1491 Vol 2, Issue 4, 2010

Research Article

SYNTHESIS AND CHARACTERIZATION OF 4- HYDROXY CHALCONES BY ALDOL CONDENSATION USING $SOCl_2$/EtOH

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Received: 27 Jun 2010, Revised and Accepted: 24 July 2010

ABSTRACT

A novel method for the synthesis of 1,3-diaryl-2-propene-1-ones via Aldol condensation is introduced using $SOCl_2$/EtOH as a catalyst. The HCl is generated in situ by the reaction of $SOCl_2$ with absolute ethanol. The structures of the synthesized compounds were confirmed by IR, mass spectroscopy and elemental analysis.

Keywords: Chalcone, Aldol condensation condensation, IR, Mass and elemental spectral analysis

INTRODUCTION

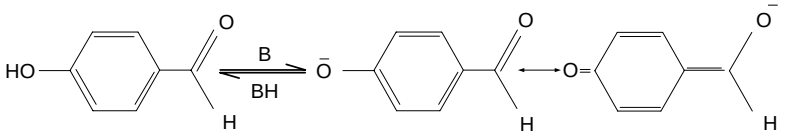
Chalcones are products of condensation of simple or substituted aromatic with simple or substituted acetophenones in presence of alkali. Chalcones constitute an important group of natural products and some of them possess a wide range of biological activities such as antimicrobial anticancer, antitubercular , antiviral etc Chalcones represent an important class of natural compounds with a variety of biological activities[1]. Recent studies on biological evaluation of Chalcones revealed some to be antibacterial, antifungal, anticancer, anti-inflammatory, antitubercular, anti hyperglycemic[2], and antimalarial agents[3]. Chalcones are very common in natural products chemistry[4]. Some derivatives are used as sweeteners, drugs, and sunscreen agents[4]. Chalcones are also well-known intermediates in the synthesis of various heterocyclic compounds[5-6].

Literature reveals that the present work deals with the reaction of 4-hydroxy acetophenone with different aromatic aldehydes to form chalcones (1-3), and the success of all the various synthesized compound were assigned on for basis of elemental analysis, IR and mass spectral data.

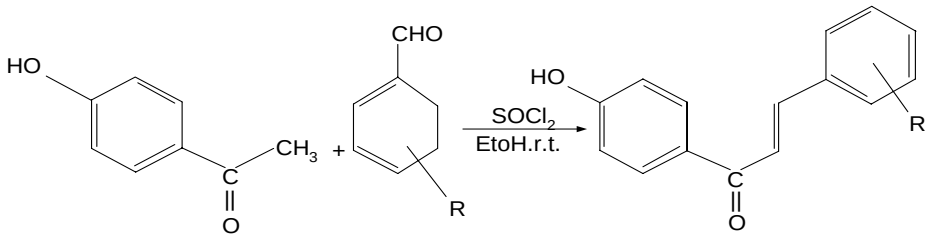
According to the literature data the presence of hydroxy substituents in the aromatic aldehyde hinders the basic catalyze aldol reaction[7]. The reaction behind that is the fact that the basic catalysts decrease the activity of the aldyhyde component because of delocalization of the anion, which is illustrated below in Scheme 1. It is necessary to use protective group for the preparation of the hydroxy chalcones under basic conditions[8]. By using $SOCl_2$ as a convenient alternative to the gaseous HCl in the aldol condensation.

MATERIALS AND METHODS

All the products were synthesized and characterized by their spectral analysis. Chemicals, 4-hydroxy acetophenone, 2-chloro benzaldehyde, 4-chloro benzaldehyde, 3-nitrobenzaldehyde were purchased from S.D. fine Chemicals (India). Melting points were determined in an open capillary tube and or uncorrected. IR spectra were recorded in KBr on a JASCO FT/IR-5300 The mass spectra were recorded on SHIMADZU – LCMS 2010 Spectrometer Elemental analysis was carried out on a FLASH EA 1112 SERIES CHN REPORT THERMO FINNIGAN. Chalcones were synthesized by aldol condensation using $SOCl_2$/ EtOH. The chemicals and solvents used were of laboratory grade and were purified completion of the reaction was monitored by thin layer chromatography on precoated sheets of silica gel-G (Merck, Germany) using iodine vapour for detection. The synthetic pathway is presented in Scheme 1 and physicochemical data and spectroscopic data for the synthesized compounds are given Table (1-3).



Scheme 1: Anion delocalization of the aldehydic component

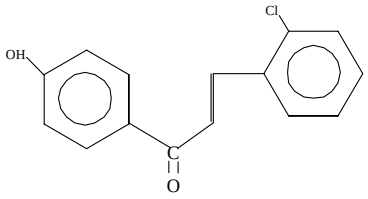


a. R = 2-Chloro
b. R = 4-chloro
c. R = 3-nitro

Scheme 2: Synthetic diagram of 4-hydroxy substituted chalcones

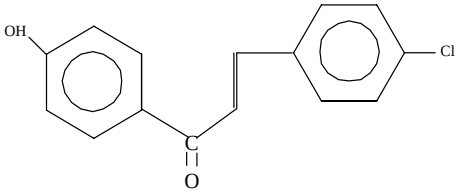
1) Synthesis of 3-(2-chlorophenyl)-1-(-4-hydroxyphenyl) prop-2-en-1-one

To a stirred mixed of 4-hydroxy acetophenone (0.01 mol) and 2-chloro benzaldehyde (0.01 mol) in absolute ethanol (5 ml) and thionyl chloride (0.05ml) dropwise and continue stirring for two hour at room temperature. Allow to stand reaction mixture for 12 hours Precipitate the reaction mixture by addition of water. Filter the product, wash with cold ethanol and allowed to afford.



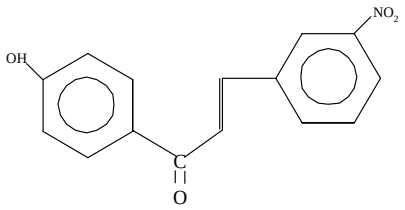
2) Synthesis of 3-(4-chloro phenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one

Reaction with 4-hydroxy acetophenone (1gm) and 4-chlorobenzaldehyde (1.1 gm); 3-(4-chlorophenyl)-1-(4-hydroxyphenyl)prop-2-en-1-one was obtained by the above described procedure.



3. Synthesis of 1-(4-hydroxyphenyl)-3-(3-nitro phenyl) prop-2-en-1-one

A mixture of 4-hydroxy acetophenone (1.0 gm) in absolute ethanol (5ml), added thionyl chloride (0.05ml) and 3-nitro benzaldehyde (1.1 gm), 1-(4-dihydroxyphenyl)-3-(3-nitro phenyl) prop-2-en-1-one was obtained by the above described procedure.



RESULTS AND DISCUSSION

This paper reports a simple and effective method for the synthesis of chalocnes by an acid catalyzed aldol reaction we used $SOCl_2$ as a convenient alternative to the gaseous HCl in eh eldol condensation. The HCl is generated in situ by the reaction of $SOCl_2$ with absolute ethanol. Chalcones are obtained in good to good to excellent yields. Our purpose was to synthesize a series of chalcones, starting from benzaldehyde and acetophenone or their substituted derivatives using $SOCl_2$/ Et OH as a catalyst.

Synthesis of chalcone is a single step method. The synthesized chalcone derivatives were undergone physicochemical characterization and the obtained results are given in Table.2. The yields of the synthesized compounds were found to be significant. The structure of the synthesized compounds was confirmed by IR, Mass and elemental analysis. Elemental analysis showed that the percentage of the nitrogen, hydrogen and carbon was found experimentally is equivalent to the calculated values in all compounds.

All the compounds give the characteristic IR peak that proved that the presence of particular functional group (Table 2) and mass spectroscopy helps to find the molecular weight of the synthesized compounds (Table 3). The Chalcone derivatives showed that the molecular ion peak that equivalent to the molecular weight of proposed compound. Hence m/z value confirms the molecular weight of the respective synthesized compound.

Table 1: Physicochemical characterization data for synthesized compounds

Compound number	Molecular formula	Molecular weight	Yield (%)	M.P (°C)	Elemental analysis		
					C	H	N
1	$C_{15}H_{11}ClO_2$	259	80	180	69.58 (69.56)	4.23 (4.28)	-
2	$C_{15}H_{11}ClO_2$	259	82	190	69.71 (69.56)	4.35 (4.28)	-
3	$C_{15}H_{11}NO_4$	269	83	182	66.85 (66.97)	4.14 (4.28)	5.28 (5.20)

Table 2: IR spectral data of synthesized compounds

Compound number	Compound	IR. Spectral data
1	3-(2-chlorophenyl)-1-(-4-hydroxyphenyl) prop-2-en-1-one	IR (KBr) *ν* cm^{-1} 3261 cm^{-1}(-OH) 1691 cm^{-1} (C=0) 1591 cm^{-1} (C=C)
2	3-(4-chlorophenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one	IR (KBr) *ν* cm^{-1} 2982 cm^{-1}(-OH) 1682 cm^{-1} (C=0) 1591 cm^{-1} (C=C)
3	1-(4-hydroxyphenyl)-3-(3-nitro phenyl) prop-2-en-1-one	IR (KBr) *ν* cm^{-1} 3142 cm^{-1}(-OH) 1651 cm^{-1} (C=0) 1606 cm^{-1} (C=C)

Table 3: Mass spectral data of synthesized compounds

Compound number	Compound	Molecular weight	Mass spectral data
1	3-(2-chlorophenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one	259	259 M^{+2}
2	3-(4-chlorophenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one	259	259 M^{+2}
3	1-(4-hydroxyphenyl)-3-(3-nitro phenyl) prop-2-en-1-one	269	269 M^{+2}

3-(2-chlorophenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one have the molecular formula of $C_{15}H_{11}ClO_2$. The molecular ion peak at 259 (M^{+2}) showed that m/z is equivalent to molecular weight of proposed compound. Hence m/z value confirms the molecular weight of the compound. The IR band at 1691 cm^{-1} suggesting the presence of (C=O) group. The IR band at 1591 cm^{-1} indicates that the presence of (C=C) group. IR band at 3261 cm^{-1} indicates presence of (-OH) group. Melting point of the compound is 180°C which is uncorrected.

The molecular formula of 3-(4-chlorophenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one is $C_{15}H_{11}ClO_2$. The obtained molecular ion peak at 259 M^{+2} showed that m/z is equivalent to molecular weight of proposed compound. Hence m/z value confirms the molecular weight of the compound. The IR band at 1682 cm^{-1} suggesting the presence of (C=O) group. The IR band at 1591 cm^{-1} indicates that the presence of (C=C) group. IR band at 2982 cm^{-1} indicates presence of (-OH) group. Melting point of the compound is 190°C which is uncorrected.

The obtained molecular ion peak of 1-(4-hydroxyphenyl)-3-(3-nitro phenyl) prop-2-en-1-one (molecular formula $C_{15}H_{11}NO_4$) at 269 (M^{+2}) that m/z is equivalent to molecular weight of proposed compound. Hence m/z value confirms the molecular weight of compound. The IR band at 1651cm^{-1} suggesting the presence of (C=O) group. The IR band at 1606 cm^{-1} indicates that the presence of (C=C) group. IR band at 3142 cm^{-1} indicates presence of (-OH) group. Melting point of the compound is 182 °C which is uncorrected.

CONCLUSION

In conclusion, we describe an efficient – protocol for the chalcones can be synthesized in good yields from aromatic aldehydes and ketones using the catalytic system $SOCl_2$/ EtOH. Thus, the present method constitutes a novel synthesis of chalcones with the condition and good yields. The synthesized compounds were characterized by TLC, melting point, IR spectroscopy, elemental analysis and mass spectroscopy. The results obtained form this study confirmed that the product has formed. Henceforth viewing these characteristic properties more compounds can be synthesized and subjected to pharmacological evaluation. These Chalcone derivatives may have variety synthesis and characterization of some new chalcone derivatives.

REFERENCES

1. Nowakowska, Z.; Kedzia, B.; Schroedere,Eur. J. Med. Chem. 2008, 43, 707-713.
2. Narender, T.; Papi Redy, K.A. Tetrahedron Lett. 2007, 48, 3177-3180,
3. Mishra, N.; Arora, P.; Kumar, B.; Mishra, L.C.; Bhattacharya, A.; Awasthi, S.K.; Bhasin, V.K. Eur.J.Med. Chem. 2008, 43, 1530-1535.
4. Li, J.T.; Yang, W.-Z.; Wang, S.-X.; Li, S.-H.; Li, T.-S. Ultrasonics Sonochemistry 2002, 9, 237-239,.
5. Karthikeyan, M.S.; Holla, B.S.; Kumari, N.S Eur. J. Med. Chem. 2007, 42, 30-36.
6. Rateb, N.M., Sulfur Silicon Relat. Elem. 2007, 182, 2393-2407.
7. D.N.Dhar,. JohnWiley & Sons, New York, 1981.
8. M.L.Go, X.Wu, X.L.Liu, Curr.Med.Chem. 12(2005) 483.