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Anhydrous K_2CO_3 as Catalyst for the synthesis of Chalcones under Microwave Irradiation

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Abstract:

A simple method for the synthesis of chalcones is reported using Anhydrous K_2CO_3 under micro wave assisted solid phase, solvent free method. The structures of the synthesized compounds were performed by IR, mass spectroscopy and elemental analysis.

Keywords: *Chalcone, Claisen-Schmidt condensation, Anhydrous K_2CO_3 IR, Mass and Elemental spectral analysis.*

Introduction:

The chalcones are α, β unsaturated ketones containing the reactive keto ethylene group –CO– CH=CH-. Presence of α, β unsaturated carbonyl system in chalcone makes it biologically active. Most of the chalcones are highly biologically active with a number of pharmacological and medicinal applications.[2] Chalcones have been used as anti AIDS agents,[2] cytotoxic agents with antiangiogenic activity[4] antimalarials[5], anti-inflammatory[6] and anti-tumor agents.[7] Keeping in view the advantages of microwave heating and the usage of chalcones as natural biocides, in the present investigation we have carried out the synthesis of some substituted o-hydroxy chalcones by claisen-schimdt condensation. This reaction is generally carried out in presence of base like NaOH or KOH which are harmful, toxic and polluting. Therefore in the present investigation we have used anhydrous K_2CO_3 as the condensing agent which is cheap, non-toxic and easy to use. Further more the reaction can be easily carried out under solvent free condition under microwave irradiation so as to minimize the pollution.

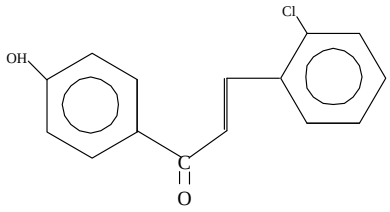
Variously substituted o-hdroxy acetophenones were condensed with aromatic aldehydes in presence of anhy. K_2CO_3 to afford the desired chalcones in 85-90% yields under microwave irradiations. The reaction was completed within 3-5 minutes.

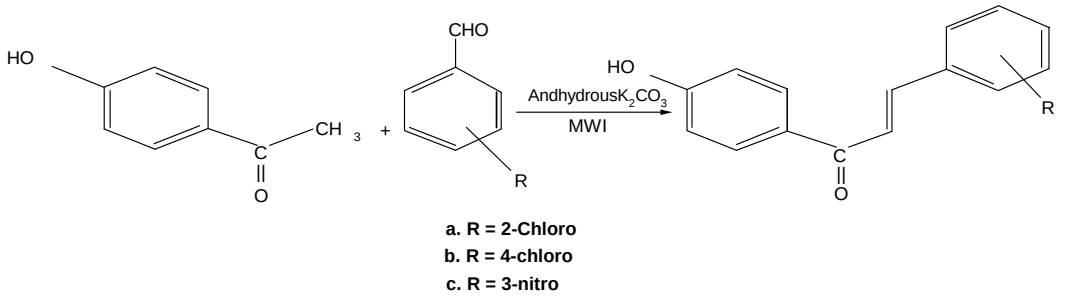
Materials and Methods:

All the products were synthesized and characterized by their spectral analysis. Chemicals, 4-hydroxy acetophenone, 2-chloro benzaldehyde, 4-chloro benzaldehyde, 3-nitro benzaldehydes 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 clasien- Schmidt condensation[21] using anhydrous K_2CO_3 as catalyst under microwave irradiation. 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).

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

4-Hydroxy acetophenone (1gram), 2-chlorobenzaldehye (2ml) and anhydrous K_2CO_3 were thoroughly mixed to form a thick paste. The past was air dried and the residual mass was subjected to microwave irradiation for 3-5minutes.





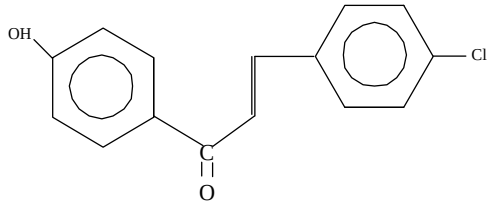
a. R = 2-Chloro
b. R = 4-chloro
c. R = 3-nitro

Scheme 1: Synthetic diagram of 4-hydroxy substituted chalcones

After completion of reaction the contents were dissolved in ethanol. Inorganic material was filtered off and filtrate after concentration in vaccuo was left overnight to get analytical sample of the chalcones in 80-90% yields utilized to synthesize further chalcone.

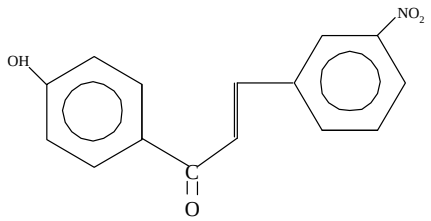
2) Synthesis of 3-(4-chloro phenyl)-1-(4-hydroxyphenyl) prop-2-en-1-one

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 anhydrous. K_2CO_3 (5ml) 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 Discussions:

Clasiew-Schmidt condensation is a versatile method for the preparation of α, β-unsaturated carbonyl compounds (Chalcones). The reaction is generally carried in presence of aqueous alkali. The concentration of the alkali generally lies between 10-60%. Other condensing agents which have been used for this reaction include alkali metal oxides, magnesium tert-butoxide. Potassium carbon compounds (KC_8), boric anhydride, organo-cadmium compounds, and lithium iodide which are quite expensive and require a lot of precautions during their use. In the present investigation we have carried out the condensation of 4-hydroxy acetophenone and aromatic aldehydes in presence of anhydrous potassium carbonate. In comparison to above mentioned condensing agents it is non toxic, non expensive and easy to use reagent. Further more its use in presence on microwave irradiation makes the process eco-friendly and economic and makes a new path in green chemical transformation. In comparison to the conventional method and reagents the yields obtained are higher and cleaner products are obtained.

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.

Table 1: Physicochemical characterization data for synthesized compounds

Compound Number	Molecular formula	Molecular weight	Yield (%)	M.P (°C)	Elemental analysis			MWI
					C	H	N	Reaction Time (Min)
1	$C_{15}H_{11}ClO_2$	259	80	182	69.58 (69.56)	4.23 (4.28)	-	1.5
2	$C_{15}H_{11}ClO_2$	259	82	185	69.71 (69.56)	4.35 (4.28)	-	2.0
3	$C_{15}H_{11}NO_4$	269	83	187	66.85 (66.97)	4.14 (4.28)	5.28 (5.20)	2.0

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) *v* 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) *v* 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) *v* 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}

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.

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 182°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 185°C, 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 187°C, is uncorrected.

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