



## SYNTHESIS OF FLAVONES BY MICROWAVE METHOD

## Chemistry

R. S. Nirwan

Department of Chemistry, MSG College (University of Pune), Malegaon Camp 423 105, India

## ABSTRACT

Chalcones are synthesized by a base catalyzed Claisen Schmidt condensation reaction and then treated with dimethyl sulphoxide in presence of iodine to get flavones by conventional method and microwave irradiation. The structures of these compounds were confirmed by IR, UV, <sup>1</sup>H NMR, Mass and elemental analysis.

## KEYWORDS

Chalcones, Microwave, Flavones, IR, <sup>1</sup>H NMR.

## INTRODUCTION.

The chemistry of chalcones has generated intensive scientific studies throughout the world. Especially interest has been focused on the synthesis and biodynamic activities of chalcones. The name "Chalcones" was given by Kostanecki and Tambor [1]. These compounds are also known as benzalacetophenone or benzylidene acetophenone. In chalcones, two aromatic rings are linked by an aliphatic three carbon chain. Chalcone bears a very good synthon so that variety of novel heterocycles with good pharmaceutical profile can be designed. These are unsaturated coloured ketone containing the reactive keto-ethylenic group -CO-CH=CH-.

The flavones are naturally occurring heterocyclic compounds belonging to the flavonoid group. These are accumulating in almost any part of plant, from the roots to the flower petal, and used as dyes for wool. Much attention has been paid to the synthesis of heterocyclic compounds like flavones and other heterocycles mainly due to their potential pharmaceutical activities [2-9]. Flavonoids are plant compounds found in fruits, vegetables, olive oil, tea, and red wine, serving as prominent secondary metabolites in dietary components. Flavonoids are plant compounds found in fruits, vegetables, olive oil, tea, and red wine, serving as prominent secondary metabolites in dietary components, tea, and red wine [10-16].

Microwave synthesis has advantages over conventional because it has rapid rate of reaction, cleaner reaction and good yield is obtained. By conventional method more amount of solvent are required and it creates water pollution. Hence an attempt has been made to synthesize flavones by microwave irradiation. In this paper we report synthesis of flavones from chalcones by conventional methods and microwave irradiation (Green synthesis).

## Experimental details:

Melting points were taken in open capillary tubes and are uncorrected. All the synthesized compounds were purified recrystallisation and column chromatography on Merck silica gel (60-120 mesh) using suitable solvents. Completion of the reactions was monitored using E-Merck silica gel TLC plates. IR spectra were recorded on a Perkin Elmer Model- spectrometer on KBr pellets. UV was recorded on Labindia/UV 3000 UV/ Vis Spectrophotometer. <sup>1</sup>H NMR spectra were recorded on Nuclear Magnetic Resonance Spectrometer (300 MHz) Varian Mercury 300 using CDCl<sub>3</sub> as a solvent and tetramethylsilane (TMS) as an internal standard. Elemental analyses were carried out on a CHNS (O) analyzer (Model- Flash EA 1112series). Mass spectra (MS) were recorded on Gas Chromatograph - Mass Spectrometer (GC-MS), Model: GCD- HP1800A.

## General procedure for the synthesis of Flavones by conventional method:

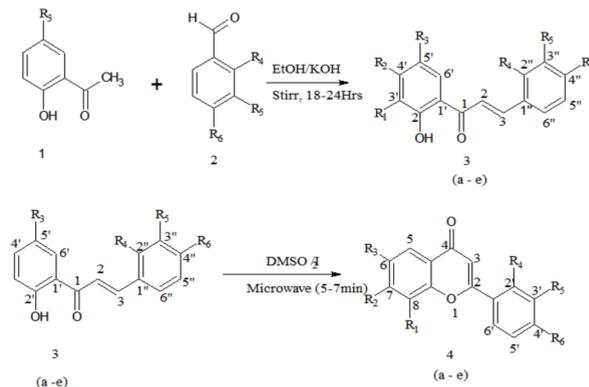
All chemicals needed for the synthesis were obtained from commercial source (AR grade with purity > 99%) and used without further purification. Aqueous KOH, 0.02 mol) was added to the suspension of 4-chloro 2-hydroxyacetophenone (2.0504 g, 0.01 mol) and *p*-chlorobenzaldehyde (0.01 mol) in ethanol. The reaction mixture was stirred at room temperature for overnight. The reaction mixture was poured into ice cold water and acidified with HCl (1M). The solid obtained was filtered (chalcone 3(a-e)) and crystallized from ethanol. Chalcone (0.002 mol) was dissolved in DMSO (20 ml) and a crystal of

iodine was added to it. The mixture was refluxed 30-45 min. The solid obtained after dilution with excess of water was filtered, washed with aqueous 20% sodium thiosulphate till the product become colorless. Further, the product was purified by column chromatography using hexane: ethyl acetate (80:20 v/v) as an eluent.

This experimental procedure was followed to prepare other analogs of this series 4(a-e) and presented in scheme 1

## General procedure for the synthesis of Flavones by Microwave method:

The chalcone 0.01 mol was suspended in (DMSO, 2 ml) and to this solution a crystal of iodine was added. The mixture was subjected to microwave irradiation for five minutes. The mixture was diluted with excess water and extracted with dichloromethane (DCM). The organic layer was washed with aqueous 20% sodium thiosulphate till the product become colorless. Further, the product was purified by column chromatography using hexane: ethyl acetate (80:20 v/v) as an eluent. The synthesized flavones listed in Table I.



Scheme I

Table-I

Flav ones	R1	R2	R3	R4	R6	Yield by conventional method (%)	Yield by MW (%)	M.P. (OC)
a	H	H	H	H	OCH3	68	82	180-182
b	H	H	Cl	H	Cl	67	80	220
c	H	H	Br	H	Cl	69	84	230
d	H	H	Cl	H	OCH3	60	78	179-181
e	H	H	Br	H	OCH3	61	82	185-187

## Spectral data of flavones:

4a: 2-(4-methoxyphenyl)-4H-chromen-4-one:  
IR (KBr): 3067 (Ar-CH), 1645 (C=O), 1512, 1607 (C=C), 1314, 1373 (C-O), 1263 (C-OCH<sub>3</sub>); UV λ<sub>max</sub> (MeOH), nm (absorbance): 316 (2.320); LC-MS: M.W. (253), 253 (M+); <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 3.91 (s, 3H, OCH<sub>3</sub>), 6.76 (s, 1H, 3-H), 8.25 (dd, 1H, 5-H), 7.44 (t, 1H, 6-H), 7.73 (dt, 1H, 7-H), 7.53 (d, 1H, 8-H), 7.89 (d, 2H, 2' & 6'-H), 7.02 (d, 2H, 3' & 5'-H).

4b: 6-chloro-2-(4-chlorophenyl)-4H-chromen-4-one:

IR (KBr): 3071 (Ar-CH), 1643 (C=O), 1600, 1562 (C=C), 1354 (C-O), 712 (C-Cl); UV  $\lambda_{\text{max}}$  (MeOH), nm (absorbance): 299 (0.558);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  6.80 (s, 1H, 3-H), 8.19 (d, 1H, 5-H), 7.87 (d, 1H, 7-H), 7.83 (d, 1H, 8-H), 7.26–7.67 (m, 4H, 2' & 6'-H and 3' & 5'-H).

4c: 6-bromo-2-(4-chlorophenyl)-4H-chromen-4-one:

IR (KBr): 3069 (Ar-CH), 1640 (C=O), 1602, 1562, 1435 (C=C), 718 (C-Cl), 642 (C-Br); UV  $\lambda_{\text{max}}$  (MeOH), nm (absorbance): 299 (0.566);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  6.81 (s, 1H, 3-H), 8.35 (d, 1H, 5-H), 7.86 (d, 1H, 7-H), 7.50 (d, 1H, 8-H), 7.65 (d, 2H, 2' & 6'-H), 7.54 (d, 2H, 3' & 5'-H).

4d: 6-chloro-2-(4-methoxyphenyl)-4H-chromen-4-one:

IR (KBr): 3073 (Ar-CH), 1645 (C=O), 1605, 1566 (C=C), 1314 (C-O), 1186 (C-O), 734 (C-Cl); UV  $\lambda_{\text{max}}$  (MeOH), nm (absorbance): 320 (2.015);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  3.90 (s, 3H,  $\text{OCH}_3$ ), 6.75 (s, 1H, 3-H), 8.19 (d, 1H, 5-H), 7.08 (dd, 1H, 7-H), 7.51 (d, 1H, 8-H), 7.62 (dd, 2H, 2' & 6'-H), 7.04 (dd, 2H, 3' & 5'-H).

4e: 6-bromo-2-(4-methoxyphenyl)-4H-chromen-4-one:

IR (KBr): 3075, (Ar-CH), 1635 (C=O), 1605, 1566, 1512 (C=C), 1273, 1255, 1188 (C-O), 656 (C-Br); UV  $\lambda_{\text{max}}$  (MeOH), nm (absorbance): 327 (0.513);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta$  3.91 (s, 3H,  $\text{OCH}_3$ ), 6.96 (s, 1H, 3-H), 8.37 (s, 1H, 5-H), 7.92 (d, 1H, 7-H), 7.90 (d, 1H, 8-H), 7.49 (d, 1H, 2'-H), 7.79 (d, 1H, 6'-H), 7.05 (d, 2H, 3' & 5'-H).

### CONCLUSION:

An efficient general microwave method has been used to synthesize flavones. The rate of reaction is high and completes in shorter time. Good yield is obtained by microwave irradiation than conventional method. By microwave irradiation less amount of DMSO and other solvents are required hence creates less pollution.

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