



A FACILE MICROWAVE ASSISTED SYNTHESIS OF FLAVONES FROM 2-HYDROXY ACETOPHENONES AND AROMATIC ALDEHYDES

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Abstract

A series of heterocyclic Flavones have been synthesized with biologically active Chalcones. These chalcones were prepared by the condensation of substituted 2'-Hydroxy acetophenones and Pyridine / Pyrrole 2-carboxaldehyde, further the solution of chalcones in DMSO and few crystals of iodine was irradiated in a microwave oven for few minutes produces Flavones. It is an easy way to get high yield of product without intermediacy of O-arylated intermediates. The structure of the synthesized compounds have been characterized by elemental analysis, IR, ¹H NMR, UV-Vis spectroscopy

Keywords: Heterocyclic Chalcones, Substituted Flavones, Elemental analysis, Spectral analysis

Introduction

Flavones exhibit diverse biological activities like antiviral¹, antihepatotoxic², anti inflammatory³ and retardness of lipoxygenase⁴ depending upon their pattern of oxygenation. several Flavones are known to be agents that reduce the induction of cancer⁵ in animals by functioning as MFO (mixed function oxygenase) systems. The compounds Flavoxate (a smooth muscle relaxant)⁶ and dimeflin⁷ (an effective CNS brain-stem stimulant) based on this skeleton are used in medicine.

Flavonoids form a class of benzo-γ-pyrone derivatives. They are polyphenolic compounds which constitute one of the most numerous and ubiquitous groups of plant metabolites. Flavonoids are generally present as glycosylated conjugates in fruits, vegetables and other plant products consumed in a normal diet⁸. Flavones, flavonones, flavanols, anthocyanidins and catechins are the immediate family members of flavonoids. Flavones are a group of oxygen containing heterocycle that contain the 2-phenyl benzopyrone. Among the naturally

occurring oxygen heterocycles Flavones (2-phenyl-4H-1-benzopyran-4-ones) are important and abundant group of Flavonoids. These are substances endowed with a wide number of pharmacological activities⁹⁻¹⁶. Flavones have been synthesised by various methods, among these various methods, the oxidative cyclisation of 2- hydroxy chalcones is more important. These various methods are of limited application as in some of these procedures, the yields are not satisfactory, while some procedures gave a mixture of products containing flavone, flavanone and aurone. so we adopted the oxidative cyclisation of 2- hydroxy chalcones, this method avoids the intermediacy of O-arylated intermediates, and provides the desired products in high yield without contamination from undesired side products.

Objectives:

This method has been implemented for the synthesis of flavones due to following merits

1. This reaction is fairly quick
2. Gives excellent yield of the product

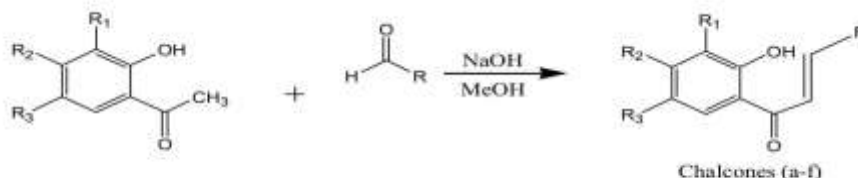
- Products formed without intermediacy of O-arylated Intermediates

- Workup and isolation is easier

Synthesis of Chalcones:

A mixture of substituted acetophenone (0.01 mol), aromatic carboxaldehyde (pyrrole/pyridine 2-carboxaldehyde (0.01 mol) and NaOH (0.02 mol) were dissolved in methanol. The reaction mixture was

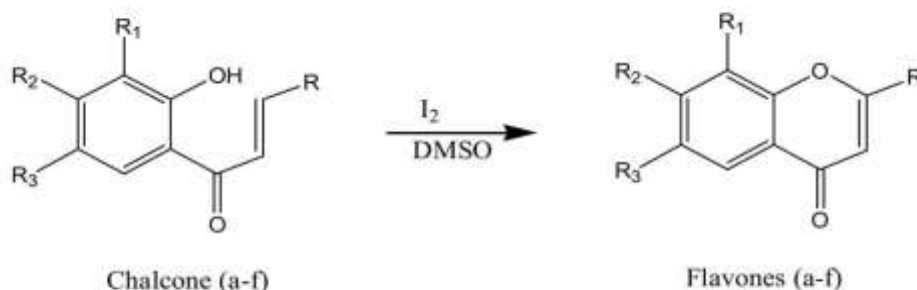
kept at room temperature for 24 hours. The progress of the reaction was monitored by TLC. After completion of the reaction the reaction mixture was poured in ice cold water and then acidified by dil. HCl. The solid obtained was filtered washed with cold water and recrystallize from ethanol.



Synthesis of Flavones:

A solution of substituted 1,3-diaryl 2-propene-1-ones (0.01 mol) containing Iodine (1-2 crystal) was refluxed for 2-3 hours. The reaction mixture was cooled at room temperature and poured onto crushed ice with constant stirring. To the

cold reaction mixture, an aqueous sodium thiosulphate solution (20%) was added until the solution was colourless followed by ice cold water (5 ml). A solid gets separated out and after collection and recrystallization it from dilute alcohol, got the flavones.



Sr.No.	Product	Substituents			
		R ₁	R ₂	R ₃	R
1.	Ia	H	H	Cl	
2.	Ib	Br	H	Cl	
3.	Ic	I	H	Cl	
4.	Id	H	H	Cl	
5.	Ie	Br	H	Cl	
6.	If	I	H	Cl	

Table no.1 analytical data of compounds

Sr. No.	Product	Mol. Formula	Yield (%)	M.P. (°C)	Elemental Analysis	
					Calcd. / (Found)	Halogen
1.	Ia	C ₁₂ H ₉ O ₂ NCl	92	98	13.65 (13.42)	
2.	Ib	C ₁₂ H ₉ O ₂ NClBr	90	210	34.12 (34.07)	
3.	Ic	C ₁₂ H ₉ O ₂ NClI	89	218	42.15 (42.10)	
4.	Id	C ₁₃ H ₉ O ₂ NCl	91	68	14.31 (14.02)	—
5.	Ie	C ₁₃ H ₉ O ₂ NClBr	85	84	35.37 (35.33)	—
6.	If	C ₁₃ H ₉ O ₂ NClI	88	96	43.50 (43.46)	

Spectral data of the selected compounds

Compound Ia:

IR (KBr) : 2924 due to -OH, 1647 due to >C=O, 1583 due to >C=C<, 1245- 1242 due to C-O-C cm⁻¹

¹H NMR (DMSO-d₆) : Shows absence of singlet at 12.52 δppm

M.S (m/z) : 259 [M⁺ ion] [%rel. intensity]

Compound Ib:

IR (KBr) : 3028, due to -OH, 1658, due to >C=O, 1537, due to >C=C<, 1224, due to C-O-C cm⁻¹, 1168, 964, 800, 727 cm⁻¹

¹H NMR (DMSO-d₆): δ 7.31-8.65 (m, 5H, Ar-H), shows absence of singlet at 11.92 δppm

M.S (m/z): 338 [M⁺ ion],[% rel. intensity]

Compound Ic:

IR (KBr): 3047 due to -OH, 1656 due to >C=O, 1533 due to >C=C<, 1161 due to C-O-C cm⁻¹, 966, 889, 700 cm⁻¹.

¹H NMR (DMSO-d₆) : δ 6.28-8.20 (m, 5H, Ar-H), shows absence of singlet at 12.86 δppm

M.S (m/z): 385 [M⁺ ion],[% rel. intensity]

Compound Id:

IR (KBr): 3066, due to -OH, 1635, due to >C=O, 1593 due to >C=C<, 1232 due to C-O-C cm⁻¹, 968, 869 cm⁻¹.

¹H NMR (DMSO-d₆) : δ 7.00–7.80 (m, 5H, Ar-H), shows absence of singlet at 11.70 δ ppm

M.S (m/z): 246 [M⁺ ion] [% rel. intensity]

Conclusion

In summary, we have synthesized some novel hetero Flavones having pyridine/ pyrrole moiety. All the synthesized compounds gave satisfactory spectral and analytical data.

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