

ECOFRIENDLY MICROWAVE ASSISTED SYNTHESIS OF SOME CHALCONES

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ABSTRACT

Claisen –Schmidt condensation has been carried out for the synthesis of some o-hydroxy chalcones using microwave assisted solid phase , solvent free method . Instead of normal bases like NaOH, KOH the condensation has been carried out in presence of anhydrous K₂CO₃ as catalyst which makes the process eco-friendly, economic and easy and becomes a part of e-chemistry.

Keywords: *Microwaves, Chalcones, Claisen –Schmidt condensation.*

INTRODUCTION

Over the years various innovative methods have been devised to speed up the chemical reactions. In these environmentally conscious days the development of technology is directed towards environmentally sound and ecofriendly methods. The usage of microwave energy to accelerate the organic reactions is of increasing interest and offers several advantages over conventional heating techniques .¹ Synthesis of the molecules which normally requires a long time can be achieved conveniently and rapidly in microwave oven. Less reaction time , easy work up and cleaner products are the major advantages of microwave heating . Further more the reactions can be carried out under solvent free conditions which holds a strategic position as the solvents are often very toxic , expensive, problematic to use . Solvent free condition is especially suitable for microwave activation.Thus the use of microwave energy for the synthesis of organic compounds forms a part of green chemistry .

Chalcones having an α , β unsaturated carbonyl group are one of the important biocides and versatile synthons for various chemical transformations. Most of the chalcones are highly biologically active with a number of pharmacological and meadical applications.² Chalcones have been used as anti AIDS agents³, cytotoxic agents with antiangiogenic activity⁴ ,antimalarials⁵ ,anti-inflammatory⁶ and anti-tumor agents⁷. 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-schmidt 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 K₂CO₃ 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 .

Variouly substituted o-hydroxy acetophenones were condensed with aromatic aldehydes in presence of anhy.K₂CO₃ to afford the desired chalcones in 85-90 %yields under microwave irradiations. The reaction was completed within 3-5-minutes.

EXPERIMENTAL

The melting points reported are uncorrected and were taken in open capillaries.The IR spectra were recorded on Perkin–Elmer spectrophotometer using KBr(v cm-1). ¹H-NMR were recorded on Bruker DRX-100 spectrometer (chemical shift δ ppm) and mass spectra (FAB) were taken on Jeol-SX-300 mass spectrometer using m-nitro benzyl alcohol as the matrix. The purity of the products and progress of the reaction was checked by tlc on silica gel plates. The reaction was carried out in domestic microwave oven (Samsung M1630N , output 600watts, frequency 2450 MHz.).

O-Hydroxy acetophenone(0.01mol), substituted benzaldehyde (0.01mol) and anhydrous K_2CO_3 were thoroughly mixed to form a thick paste. The paste was air dried and the residual mass was subjected to microwave irradiation for 3-5minutes. After completion of reaction the contents were dissolved in ethanol . Inorganic material was filtered off and filtrate after concentration in vacuo was left overnight to get analytical sample of the chalcones in 80-90% yields.

RESULTS AND DISCUSSION

Claisen-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 alkali⁸⁻¹⁴. The concentration of the alkali generally lies between 10-60%. Other condensing agents which have been used for this reaction include alkali metal oxides,¹⁵ magnesium tert.-butoxide¹⁶, potassium carbon compounds(KC_8)¹⁷, boric anhydride¹⁸, organo- cadmium compounds¹⁹, and lithium iodide²⁰ which are quite expensive and require a lot of precautions during their use. In the present investigation we have carried out the condensation of o-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. Furthermore 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.

The identity of the products obtained was confirmed on the basis of their elemental analysis spectral data, mmp's and co-tlc with authentic sample The IR spectra of these compounds gave prominent peaks at 1640-1630 cm^{-1} , (C=O), 3100—3000 cm^{-1} (C-H stret.), 1000-950 cm^{-1} (C-H out of plane deform), 1300-1310 cm^{-1} (CH=CH in plane deform) and 3400-3500 cm^{-1} (OH stret.). ¹H-NMR spectra of chalcones gave double doublet for vinylic protons at δ 7.6-7.7, and a multiplet for aromatic protons at δ 7.8-8.0 . The mass spectra of these compounds gave molecular ion peaks corresponding to their molecular masses.

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Table: Physical Data of Chalcones

Compd.	M.P. °C	Reaction time		% yield	
		Conven. (hrs)	MWI (min)	conven.	MWI
2-OH chalcone	89	8.0	1.5	67	86
2-OH-4-OMe chalcone	92	7.0	2.0	70	82
2-OH-3,4-diOMe chalcone	112	8.0	2.0	70	82
2-OH-3,4,5-triOMe chalcone	142	9.0	2.5	71	83
2-OH-4-Cl chalcone	152	6.0	3.0	72	88
2-OH-4-NMe ₂ chalcone	160	8.0	3.5	68	83
2,4- diOH chalcone	135	7.0	2.0	70	83

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