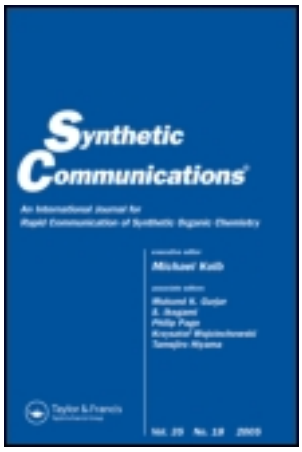

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MICROWAVE ASSISTED KNOEVENAGEL CONDENSATION: A FACILE METHOD FOR THE SYNTHESIS OF CHALCONES *

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MICROWAVE ASSISTED KNOEVENAGEL CONDENSATION: A FACILE METHOD FOR THE SYNTHESIS OF CHALCONES*

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ABSTRACT

Microwave irradiation of aromatic aldehydes with acetophenones or malonic acid in presence of anhydrous zinc chloride was resulted exclusively chalcones and cinnamic acids respectively in high yields.

The basic skeleton of chalcones is widely figured in natural products[1] and are known to have multi pronged activity. Some of them are useful as drugs and agrochemicals.[2–4] Recently much attention has been paid on the synthesis of chalcones mainly from acetophenones and aromatic aldehydes. Several catalysts such as basic alumina,[5] Al_2O_3–$AlPO_4$,[6] P_2O_5–piperidine,[7] ultrasonic rays using C-200[8] and Lewis acid[9,10] have been used for Knoevenagel condensation reaction. In addition, media such as strong alkali[11] or quarternary ammonium salts[12] have also been employed.

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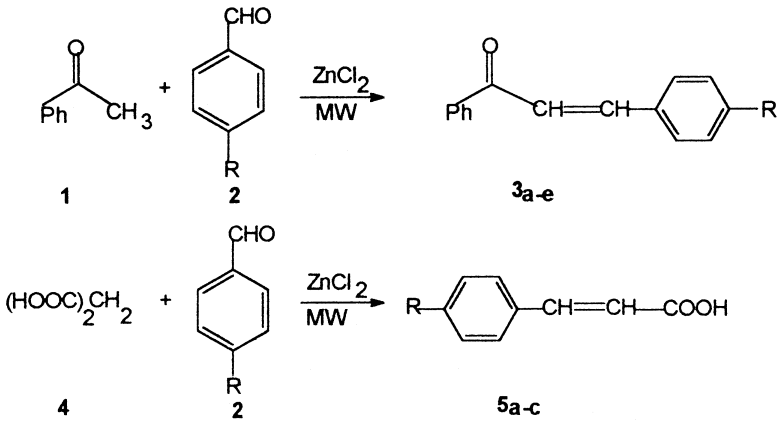
Further studies on the efficient synthesis of chalcones is of current interest because of their wide range of applications. Thus we wish to report here for the first time a simple and facile approach to chalcones and cinnamic acids.

The methodology involves the reaction of aromatic aldehydes with various acetophenones or malonic acid independently in presence of catalytic amount of zinc chloride under thermal as well as microwave conditions. The reaction under thermal condition is found to be sluggish and took longer time with yields in the range 45–50%. However in the microwave conditions the rate of reaction is fast as a result the reaction times are shorter and yields are high with easy isolation of products. In case of malonic acid reaction, it is assumed to be initially the formation of benzylidene product followed by decarboxylation. The details of schematic diagram cited below in Scheme.



Scheme.

All the compounds have been characterised based on spectral data and were comparable to the literature reports. The number of compounds synthesised have been tabulated in Table.

EXPERIMENTAL

Melting points were determined in open glass capillaries on a Mettler FP51 melting point apparatus and are uncorrected. IR spectra were recorded on FT-IR Schimadzu Perkin-Elmer 1310 infrared spectrophotometer.

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Table. Synthesis of Chalcones Under Microwave Conditions

Product	Reac. Time (min)	R	Yield (%)	M.p. (°C)	Lit. m.p. (°C)
3a	5	H	85	54	55[13]
3b	3	Cl	82	115	112[14]
3c	3	OCH_3	90	75	76[15]
3d	4	CH_3	87	94	96[15]
3e	5	NO_2	85	163	165[16]
5a	4	H	77	133	132.5[17]
5b	3	Cl	75	247	241[18]
5c	3	CH_3	79	195	198[19]

^{1}H NMR spectra were recorded on Varian Gemini (200 MHz) spectrometer and TMS was used as internal standard. Mass spectra were recorded on a VG-micromass 7070H instrument at 70 eV.

Synthesis of Chalcones: General Procedure

1. Thermal conditions: A mixture of aromatic aldehyde (0.01 mole) active methylene compound (0.01 mole) and anhydrous zinc chloride (0.001 mole) was heated at 100°C for 6 h. The reaction mixture was cooled and treated with aqueous alcohol (20 mL). The separated solid was filtered, washed with *n*-hexane and dried. The product was recrystallised by benzene–hexane to give chalcones.

2. Microwave conditions: A mixture of aromatic aldehyde (0.01 mole), active methylene compound (0.01 mole) and zinc chloride (0.001 mole) was taken in ACE tube, flushed with argon and tightly capped. The mixture is subjected to microwave heating for 5 min in a domestic oven (600 W, BPL BMO 700 T) and then it is allowed to reach to room temperature. The reaction mixture was treated with aqueous ethanol (20 mL) and the separated solid was filtered, washed with *n*-hexane and dried. The solid was recrystallised by benzene–hexane.

CONCLUSION

The new synthetic methodology developed has universal applications in the synthesis of a number of chalcones.

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