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Green Synthesis of Chalcones under microwave Irradiation

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Abstract: Green chemistry is also called sustainable chemistry, Microwave assisted organic synthesis which is an important tool for heating in the organic synthetic reaction[1,2]. Microwaves act as high frequency electric fields and will generally heat any material containing mobile electric charges, such as polar molecules in a solvent or conducting ions in a solid[3]. This technique offers simple, clean, fast, efficient, and economic method for the synthesis of a large number of organicmolecules. The chalcones and their derivatives are important intermediates in the synthesis of heterocyclic compounds which are of physiological importance. They possess a broad spectrum of biological activities viz antibacterial, antifungal, antitubercular, antitumor, antibetic[17].
Keywords: Microwave irradiation, Chalcone, Synthetic methods.

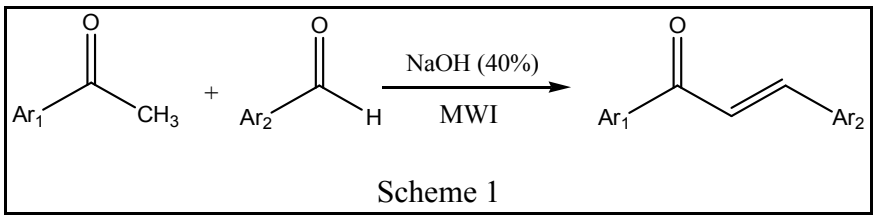
Introduction

Microwave chemistry is the study of chemical reactions under the influence of microwave radiation. The promotion of microwave assisted to speed up chemical reactions in the laboratories[13]. Microwave heating has been shown to dramatically reduce reaction times, increase product yields and enhance product purities by reducing unwanted side reactions compared to conventional heating methods. In resent year microwave assisted in the synthesis of Chalcones[16]. Chalcones are natural biocides and are well known intermediates in the synthesis of heterocyclic compounds exhibiting various biological activities[6]. Many methods are available for synthesis of chalcones, The simplest method involves Claisen-Schmidt condensation of equimolar of simple or substituted aromatic aldehyde with simple or substituted acetophenone in base medium[19].

Experiments

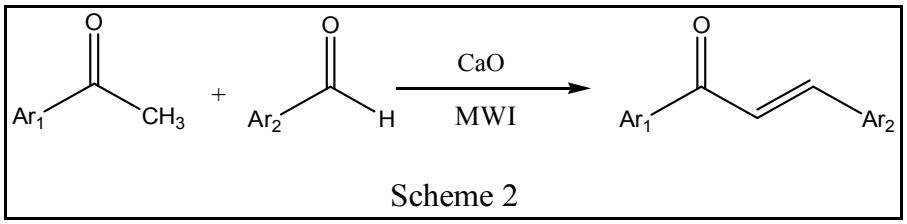
General Procedures for the Synthesis of Chalcones :

1. An equimolar mixture of methyl ketone and aromatic aldehydes dissolved in minimum amount of rectified spirit and NaOH (40%) were placed in a conical flask. The conical flask was covered with a funnel and then the flask was taken in a domestic microwave oven. The reaction mixture was irradiated under 160-320 watt microwave irradiation for 60-120 sec. The progress of the reaction was monitored by TLC after every 30 sec. The reaction mixture was cooled and the obtained solid was recrystallized from suitable solvent[15]. (Scheme 1) .



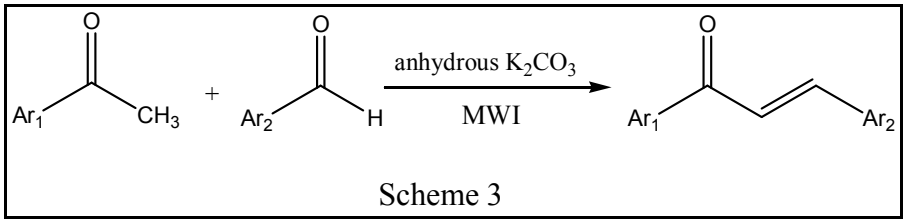
Scheme 1

2. Substituted acetophenone (5mmol) and Substituted benzaldehyde was dissolved in 5mL ethanol. This solution was poured on calcium oxide and well swirled. The solvent was removed under reduced pressure using a rotator evaporator. Resulting free flowing powder was taken in a 25mL beaker and irradiated in microwave oven at 400W for 15 minutes. The progress of the reaction was monitored by TLC. After completion of the reaction, the reaction mixture was cooled, added to 20mL ice cold water and acidified with Conc. HCl, solid was precipitated, filter on suction pump, wash water and dried it. A pure sample was obtained by recrystallization[16].(Scheme 2).



Scheme 2

3. The reaction was carried out in domestic microwave (600 W, 2450 MHz.).Substituted 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 vacum was left overnight to get analytical sample of the chalcones in 80-90% yields[17,18].(Scheme 3).



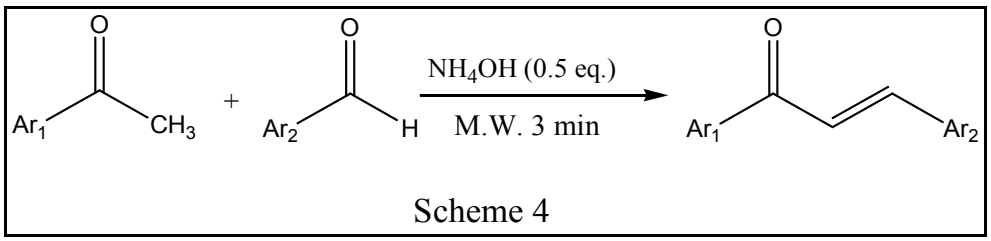
Scheme 3

4. A mixture of acetophenone derivative (0.01mol), substituted aldehyde (0.01mol) and $ZnCl_2$ (0.001mol) was taken in ACE tube, flushed with argon and tightly capped. The mixture was subjected to microwave heating for 3-5 min. in a domestic oven and allowed to reach the room temperature. The reaction mixture was treated with aq. ethanol (20 ml) and the separated solid was filtered, washed with n-hexane and dried. The solid was recrystallized .

5. To a solution of acetophenone (0.01 mole) and substituted aromatic aldehyde (0.01mole) in ethanol (5 mL) taken in a borosil flask (100 mL), inorganic catalysts (5-7 g) was added. The reaction mixture was mixed and the adsorbed material was dried, in air and irradiated inside the microwave oven . Progress of the reaction was monitored by TLC. After the completion of the reaction, the product was cooled, purified and recrystallized[19].

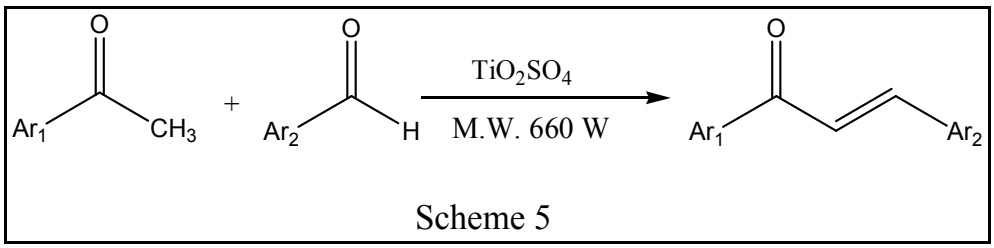
6. The acetophenone (0.001mol), suitable aldehyde (0.0011mol), finely powdered base NaOH/KOH/$Ba(OH)_2$ and few drops of dry methanol were mixed thoroughly in an open corning glass wide-mouth test tube. The amount of monoacidic bases i.e. NaOH, KOH used in the synthesis were 0.002mol while that of diacidic base S-200 $Ba(OH)_2$ was 0.001mol.This mixture was irradiated under MW in a domestic microwave oven for 25 seconds. Then, the reaction mixture was cooled, poured over crushed ice and was neutralized with dil. HCl. The crude product then filtered which was recrystallized[20].

7. Ketone (1mmol), aldehyde (1mmol) neutral alumina (2 g), and ammonium chloride (0.5mmol) were mixed thoroughly with the help of mortar pastle and the mixture was taken in a Pyrex beaker (10 mL). The solid mixture was irradiated under a microwave (480 W, 3 min). After completion of reaction indicated by TLC, the reaction mixture was cooled to room temperature, poured in ethanol, and filtered. After evaporation of the solvents corresponding condensation products were recovered in crystalline forms[14]. (Scheme 4).



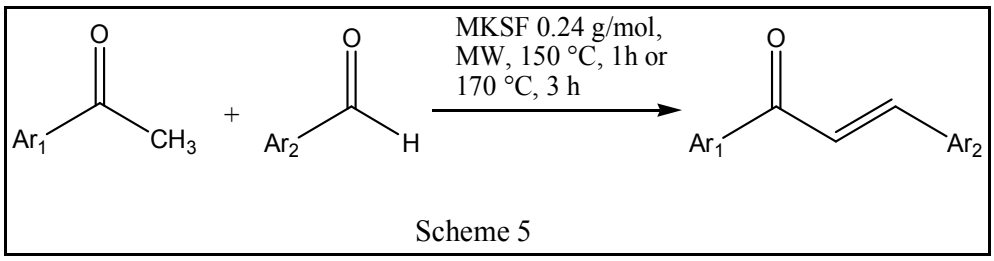
Scheme 4

8. Appropriate quantities of substituted acetophenones (2mmol) and aldehyde (2 mmol) and 0.15 g of sulfated titania were taken in a 50 mL beaker and closed with the lid. This mixture was subjected to microwave irradiation for 2-4 minutes at 650 W. After completion of the reaction, dichloromethane (20 mL) was added, followed by simple filtration. The filtrate was concentrated and the obtained solid was purified by recrystallization[9].(Scheme 5) .



Scheme 5

9. A mixture of the suitable aldehyde (1.0 mmol), acetophenone (1.0 mmol) and clay catalyst (240 mg) was warmed at 150 °C in a sealed tube under microwave irradiation, for 1 h. In the reactions involving solid starting materials, they were thoroughly mixed by grinding in a mortar before irradiation. The reaction mixture was diluted with hot ethanol (20 mL), the catalyst was filtered off, the solvent was evaporated and the residue was purified by crystallization[20].(Scheme 5).



Scheme 5

Conclusion

Microwave assisted organic synthesis which is an important tool for green chemistry .Microwave irradiation synthesis is not only use the least time, but also to increase the yield. Microwave irradiation is becoming an increasingly popular method of heating Microwave assisted organic synthesis has emerged as a new “lead” in organic synthesis which makes the chemistry to go green.

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