INTERNATIONAL JOURNAL OF PHARMACEUTICAL SCIENCES
[ISSN: 0975-4725; CODEN(USA): IJPS00]
Journal Homepage: https://www.ijpsjournal.com

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Review Article

Microwave Assisted Organic Synthesis of Non-Heterocyclic Compounds

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ARTICLE INFO
Published: 20 Oct 2025
Keywords:
Microwave heating is a quick, targeted, and environmentally responsible technique
DOI:
10.5281/zenodo.17394299

ABSTRACT

In the microwave-assisted synthesis, has emerged as a powerful technique for accelerating chemical reactions the mechanisms observed are ionic conduction and dipolar polarization. Microwave heating is a quick, targeted, and environmentally responsible technique. Compared to microwave synthesis, the conventional heating process requires more time, complex equipment, which raises costs, and excessive material and solvent use, which pollutes the environment more. It efficiently accelerates various reactions including those forming non-heterocyclic compounds like alkanes and cycloalkanes. Its application in non-heterocyclic synthesis promotes sustainable and high through put organic synthesis process.

INTRODUCTION

Microwave heating was first observed when Percy Spencer, an engineer at Raytheon, accidentally discovered that microwaves from a radar system melted a chocolate bar in his pocket. This led to the invention of the “Radarange” in 1947 and the later development of microwave ovens, which became common in households by the 1970s and 1980s. In 1986, microwave radiation was first reported for use in chemical synthesis. Organic synthesis, a key area of chemical research, has evolved with advancements in environmentally friendly and efficient methods. Modern chemists now use diverse techniques—such as photochemical, electrochemical, sonochemical, microwave, and enzymatic methods—to promote reactions. Among these, microwave irradiation has gained popularity in the past decade for its ability to rapidly and efficiently synthesize various compounds by selectively heating polar molecules.

Microwave assisted

Microwave assisted refers to the use of microwave energy to speed up chemical, biological, or material processes by providing rapid and uniform heating. It is widely used in synthesis, extraction, and material processing to improve efficiency and reduce reaction time.

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Relevant conflicts of interest/financial disclosures: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Types of micro wave assisted

Microwave Assisted Reactions using Solvents

Water, with tunable dielectric properties, can act as a green substitute for organic solvents, easing product isolation and reducing waste. Enhanced Microwave Synthesis (EMS) sustains microwave energy throughout the reaction, giving higher yields and cleaner chemistries than conventional methods. Combining water and microwaves is gaining attention for diverse organic and radical transformations in both solvent- and solid-phase reactions.

Microwave Assisted Reactions under Solvent-Free Conditions

Microwave-assisted solvent-free organic synthesis (MASFOS) is a green, waste-free process avoiding organic solvents while ensuring high selectivity. It includes reactions with phase transfer catalysis, solid supports, or neat reactants, offering cleaner and eco-friendly alternatives for modern chemistry.

Microwave Assisted Reactions using Solid-Liquid Phase

Solid-liquid phase transfer catalysis (PTC) enables efficient anionic reactions under microwaves using quaternary ammonium salts. The electrophile (R–X) acts as both reactant and organic phase, avoiding extra solvents and enhancing reactivity.

Microwave Assisted Reactions on Mineral Supports in Dry Media

Mineral supports absorb microwaves efficiently, giving rapid, uniform heating. This enables faster reactions with less product degradation than conventional heating.

Non-Heterocyclic Nucleus

- Microwave Assisted Heterofunctionalization of Alkenes and Alkynes
- Microwave assisted synthesis and antibacterial activity of chalcones derivatives
- Microwave-assisted catalytic decomposition of methane over activated
- Microwave assisted of carambola shaped Cu O with hierarchical
- Microwave-assisted conversion of ethane to ethylene
- Microwave-induced cracking and reforming of benzene on activated carbon
- Microwave-Assisted Polyamide Synthesis
- Controlling selectivity in catalysis: Selective greener oxidation of cyclohexene Under microwave conditions
- Microwave assisted phase transfer catalysis: An efficient solvent free method for the synthesis of cyclopropane derivatives
- Solid-Liquid Phase Transfer Catalysis and Microwave-Assisted Green Synthesis of Tetra cyclone

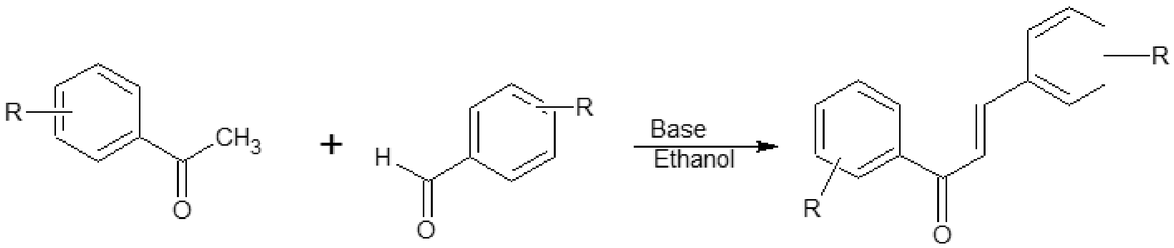
Microwave assisted synthesis and antibacterial activity of chalcones derivatives

Experimental method

- Equimolar amounts (0.01 mol each) of substituted acetophenone and substituted benzaldehyde were dissolved in ethanol in a conical flask.
- A 10% aqueous KOH solution was added dropwise with continuous stirring.
- For the microwave method, the reaction mixture was irradiated at 480 W for 15–30 seconds.
- For the conventional method, the mixture was stirred and left at room temperature for 24 hours.

- Melting points were measured in open capillaries, reactions monitored by TLC, and IR spectra recorded on an FTIR-8400 using KBr pellets.
- ^{1}H NMR spectra were obtained on a DZIRE400 spectrometer in acetone with TMS as internal standard; all reagents were of laboratory grade.

Scheme: Reaction scheme of chalcones; {R': 4-Cl, 4-Br, R: H, 4-N(CH3)2, C4H4O}.



Time taken and %yield for compounds

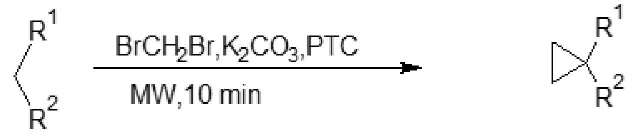
Table 2: Time taken and 1% yield for compounds

Compound code	Conventional method		Microwave method	
	Time(h)	yield (%)	Time(sec)	Yield (%)
C1	8	77	15	95
C2	7	72	18	90
C3	7	45	15	80
C4	7	80	20	96
C5	8	64	25	94
C6	8	49	15	87

Microwave assisted phase transfer catalysis: An efficient solvent free method for the synthesis of cyclopropane derivatives

Experimental method:

- ^{1}H NMR spectra were recorded on Bruker spectrometers in $CDCl_3$ with TMS as standard, IR spectra on a Perkin-Elmer Infracord, and melting points on a Thermonik Campbell apparatus.
- Reactions were carried out in an 800 W BPL microwave oven, and products purified by column chromatography using silica gel (60–120 mesh).
- Active methylene compounds were reacted with dibromoethane, K_2CO_3, and Aliquat-336 under microwave irradiation (70% power, 10 min).
- After workup with ethyl acetate, washing, and solvent removal, the products were purified by silica gel column chromatography.



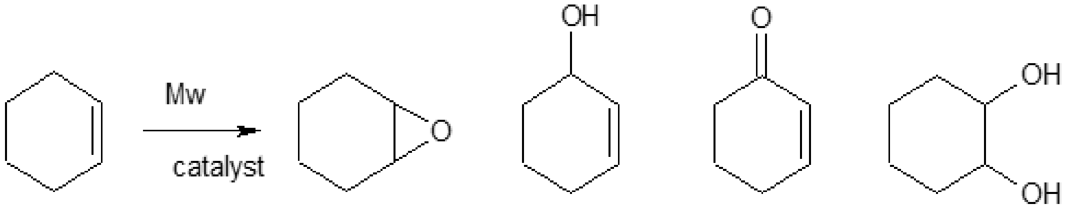
Scheme: a, R I = R2= COIEt b, RI = COCH3; R2= COIEt c, RI = CN; Rl = C02Et d, RI = 4-MePhS02; R2= C02Et e, RI = R2= COCH3

Controlling selectivity in catalysis: Selective greener oxidation of cyclohexene Under microwave conditions

Experimental method:

- Melting points were measured on electrothermal equipment; IR spectra (KBr) on a Perkin-Elmer 457, and $^1H/^{13}C$ NMR on a Bruker 400 using DMSO-d_6 or $CDCl_3$ with TMS as standard.
- TLC was performed on Merck silica gel 60 F_{254} plates with solvent systems A (acetone/toluene/cyclohexane, 5:2:3) and B (ethyl acetate/n-hexane, 4:6), while RP-HPTLC used methanol/water (75:25).

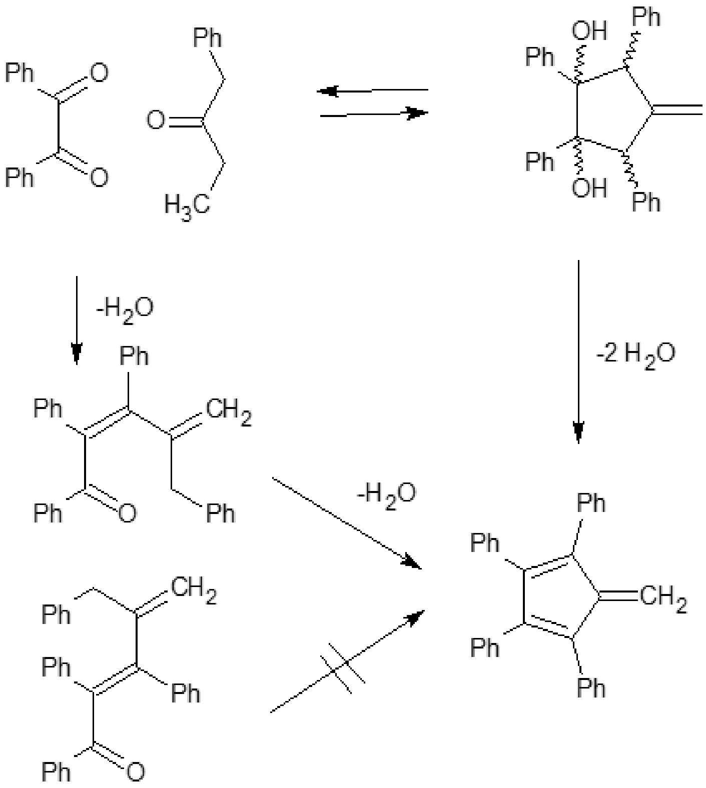
- Microwave reactions were performed on a CEM Discover monomode unit (2450 MHz, 0–300 W) with continuous-feedback temperature control and either magnetic or mechanical stirring.
- Closed 10 mL septum-sealed vessels (pressure regulated) or open 100 mL vessels were used, with TLC/HPTLC confirming compound homogeneity; all solvents and reagents were Aldrich ACS grade.



Scheme: Possible products that can be obtained from the oxidation of cyclohexene.

Solid-Liquid Phase Transfer Catalysis and Microwave-Assisted Green Synthesis of Tetra cyclone

Equipment Melting points are uncorrected and were measured with an electrothermal melting point equipment. KBr disks were used to record infrared spectra on a Perkin-Elmer 457 spectrometer. Wave numbers are measured in cm-1. Using a Bruker 400 spectrometer, 1H-NMR and 13C-NMR spectra were captured at room temperature. DMSO-d6 or CDCl3 were used to dissolve the compounds. Tetramethylsilane, or TMS, is used as an internal standard when expressing chemical shifts in the δ scale. Merck TLC plates (silica gel, 60 F 254, E) were used for thin layer chromatography (TLC) studies. E. Merck, reference 5735, Darmstadt, Germany. All of the compounds listed here were regularly examined in two common solvents for TLC: ethyl acetate/n-hexane (solvent B, 4:6, v/v) and acetone/toluene/cyclohexane (solvent A, 5:2:3, v/v/v). HPTLC plates RP-18 F-254 S (Merck) and methanol: water (75/25, v/v) were the conditions for the reverse-phase thin layer chromatography. The corresponding writers have information about semi-empirical quantum computations.



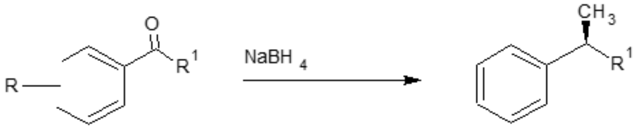
Scheme5: Synthesis Of Tetra Cyclone

Under such TLC and HPTLC conditions, all reported compounds were determined to be homogeneous. We bought all of the reagents from Aldrich in Milwaukee, USA. Every solvent was of the Aldrich ACS reagent grade. The first exploratory small-scale microwave experiments were carried out with a PC computer-monitored CEM Discover Synthesis Unit (monomode system) running at 2450 MHz. A circular waveguide encircling the cavity focuses the microwaves generated by this apparatus. Optical fiber or infrared detection with continuous-feedback temperature control was used to measure the temperature, and power modulation (0–300 W) was used to keep it constant. When reactions were carried out in closed containers with regulated pressure, stirring was supplied by an in situ magnetic variable speed stirrer; for reactions conducted in open vessels, mechanical stirring was used. Glass vessels with a 10 mL capacity that were sealed with a septum or open vessels with a 100 mL capacity were used for the reactions. A load cell that was attached to the vessel via a needle that went through just below the septum surface managed the pressure.

5)Applications

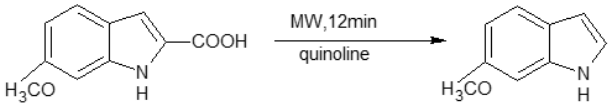
Reduction

In a microwave, acetopehenone and NaBH4 are reduced to generate 92% of benzyl alcohol in 2 minutes.



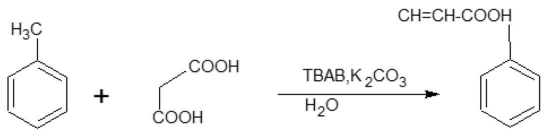
Decarboxylation

The yields are poor when carboxylic acids are decarboxylated conventionally, which involves refluxing in quinoline while copper chromate Is present. However, decarboxylation occurs considerably more quickly in the presence of microwaves.



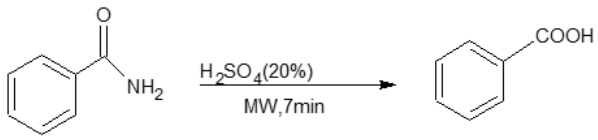
Knoevenagel Condensation

A well-known chemical process called Knoevenagel condensation is also used to create unsaturated acids, which are utilized as building Blocks for numerous heterocycles, flavonoids, and scent precursors. Tetrabutylammonium bromide and potassium carbonate in water were Used to study Knoevenagel condensation between.



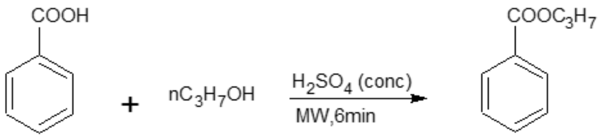
Hydrolysis

When benzyl chloride is hydrolyzed with water in a microwave oven, 97% of the resulting benzyl alcohol is created in 3 minutes. The usual procedure for common hydrolysis takes about 35 minutes. Benzamide hydrolysis usually takes an hour. However, the hydrolysis is completed in 7 minutes with a 99% yield of benzoic acid when microwaved.



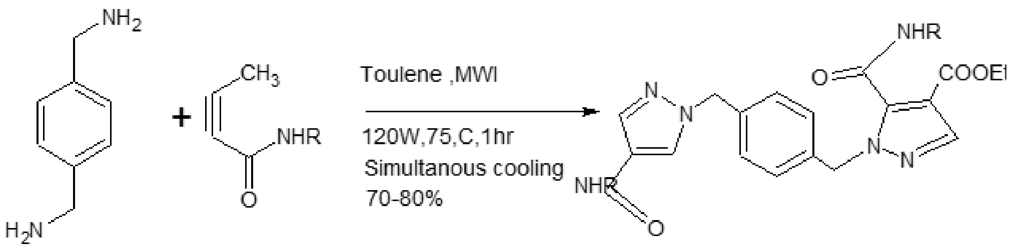
Esterification

a combination of n-propanol and benzoic acid heated for 6 minutes in a microwave in the presence of catalyst, Propyebenzoat is produced by sulfuric acid.30



Cycloaddition:

1,3-Dipolar cycloadditions [31] are important reactions in organic synthesis. Cycloaducts were prepared by carrying out the reaction between an azide and a substituted amide in toluene. This reaction was carried out under microwave irradiation at 120 W at 75 °C for 1 h. The product was isolated in 70–80 % yield.



6)Advantage of Microwaves assisted synthesis:

- Rapid reactions
- High purity of products
- Less side-products
- Improved yields
- Simplified and improved synthetic procedure
- Wider usable range of temperature
- Higher energy efficiency

- Sophisticated measurement and safety technology
- Modular systems enable changing from mg to kg scale.

Drawbacks of microwave assisted synthesis:

• Some solvents are inappropriate because they absorb microwaves much more readily than others.

• Reactions involving volatile compounds require extra caution since pressure in these reactions might also result in an explosion

• Heating reactions much above the boiling point of the solvent can increase the pressure which can cause vials to explode.

• They frequently accomplish inconsistent solvent heating, making yields unpredictable, unless we are employing a very costly microwave Reactor.32

7)CONCLUSION

The conclusion of Microwave assisted organic synthesis of non-heterocyclic compounds highlights it significant advantages in accerlating reactions, influencing yield, and enabling environ mental friendly conditions. Microwave irradiation reduced reaction times drastically compared to conventional heating and often increase the product purity. The technique supports solvent free or solvent condition aligning with green chemistry principles. Micro wave assisted organic synthesis also offers enhanced selectivity and energy efficiency, making it valuable in sustainable synthetic methodologies. Overall microwave assisted synthesis is powerful, efficient and eco conscious approach for preparing non heterocyclic organic compounds across various applications.

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HOW TO CITE: Dr. ND. Nizzamuddin*, Nikhitha Velpula, Microwave Assisted Organic Synthesis of Non-Heterocyclic Compounds, Int. J. of Pharm. Sci., 2025, Vol 3, Issue 10, 2086-2094 https://doi.org/10.5281/zenodo.17394299