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## A review article on eco-friendly synthesis through microwave-assisted reactions

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### Abstract

*Linum usitatissimum* L a member of *Linaceae* family, which is used for various medical purposes. Microwave assisted processes are useful instruments for scientific and industrial applications because they provide faster reactions, higher yields, energy efficiency, and improved product quality when compared to conventional heating. Microwave-assisted synthesis is increasingly finding applications in chemistry laboratories due to its ability to shorten reaction times enhance throughput, and yield purer products. When ions and polar molecules are exposed to oscillating electric and magnetic fields, three primary heating processes take place dipolar polarisation, conduction, and interfacial polarisation. Only substances possessing the ability to absorb microwave radiation are influenced.

The two primary equipment types utilized are single-mode and multi-mode systems. Although they are utilized, terms such as microwave-enhanced chemistry and microwave-organic reaction enhancement are not familiar. Selective heating enables concentrated energy transfer because different materials absorb microwave radiation in different ways. Real-time reaction control and optimisation can also be achieved by integrating in-situ monitoring.

**Keywords:** Spencer, Magnetron, Radarange, Monomode microwave reactor, multimode microwave reactor

### Introduction

Microwave technology's capability of rapidly heating water was the first source of inspiration for microwave-assisted processes, which employ the use of microwave energy to accelerate material formation. After spontaneous experiments involving foodstuffs such as popcorn and eggs, Percy Spencer discovered the microwave's heating capacity in the 1940s, which opened the gates for microwave cooking appliances development in the 1950s. The microwave-assisted process heating properties that utilized microwave energy in material synthesis were first discovered by Percy Spencer in the 1940s. A microwave cooking appliance was developed in the 1950s following tests that indicated food heated up rapidly. Home microwaves became increasingly popular in the 1970s and 1980s after the commercial microwave appliance Rearrange came on the market in 1947. Since microwave irradiation yields rapid, uniform, and accurate heating that accelerates procedures, it has been used in chemical synthesis since 1986.

By direct contact with molecules, microwaves convert electromagnetic energy to heat, making it possible to have faster, cleaner, and greener chemistry without producing hazardous byproducts. Heat is created when molecules vibrate as a result of electromagnetic waves called microwaves. They are between infrared and radio waves. Microwave-induced synthesis is unique through its high heat efficacy through direct energy absorption, its capacity to selectively heat up polar molecules, and its capacity to increase chemical reactions through non-thermal or microwave effects.

### History

Later on, oil baths and hot plates took the place of Robert Bunsens burner (1855). After Percy Spencers first employment of microwave energy for heating food during the 1940s, it started to speed up chemical synthesis during the 1980s. In spite of the fact that early microwave synthesis had been dependent on Teflon or sealed glass reactors in domestic ovens, often resulting in explosions due to the rapid heating of the solvents, its use increased exponentially during 15 years.

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### Principle

Microwave radiation falls between infrared radiation and radio waves in the electromagnetic spectrum. Telecommunications and microwave radar employ numerous frequencies within this range. In order to avoid interference with such systems, microwave ovens are designed to work at a specific frequency of 2.45 GHz. Planck's law  $[E=h\nu]$  predicts the quantum energy at this frequency to be approximately 0.3 cal mol.

### Mechanism of microwave heating

Materials react differently to microwave radiation, which means not all can be heated by it. They can be classified into three groups:

- Substances that is transparent to microwaves, such as sulphur,
- Materials that reflect microwaves, like copper, and
- Substances that absorb microwaves, such as water.

Heating primarily occurs through ionic conduction, dipolar polarization, and interfacial polarization. For microwave chemistry, it is crucial to have materials that can absorb microwaves.

### Dipolar polarization

Microwave heating through dipolar polarization happens when polar molecules attempt to align themselves with the fluctuating electric field, leading to molecular friction and the production of heat. Effective heating requires a dipole moment and the correct frequency (2.45 GHz) for

alignment. Gases reorient quickly, while molecular interactions in liquids slow the process. Polar solvents (e.g., water, methanol, and ethanol) and solutes (e.g., ammonia, formic acid) efficiently absorb microwave energy. At very high frequencies, polar molecules cannot reorient effectively due to intermolecular forces, while at low frequencies they can align with the field. The microwave range (0.3-30 GHz) is ideal, allowing proper oscillation and interaction, making it optimal for heating polar solutions.

### Ionic polarization

Ionic conduction in microwaves occurs when dissolved ions oscillate with the electric field, colliding and generating heat through kinetic energy. This mechanism produces faster heating and greater energy release than dipolar polarization, often causing superheating. Ionic liquids and ion-containing solutions, like tap water, absorb microwaves efficiently and heat more rapidly than non-ionic solutions like distilled water.

### Interfacial polarization

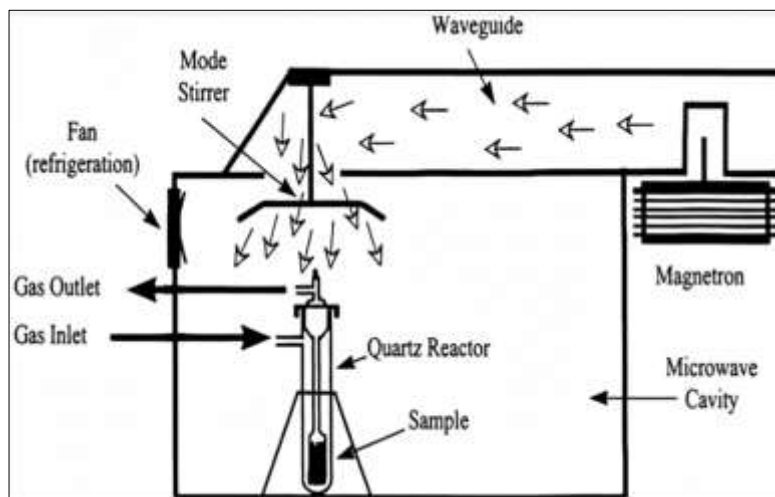
Interfacial polarization Interfacial polarization combines conduction and dipolar polarization, occurring in systems containing conductive particles suspended in non-conductive media. For instance, metal powders suspended in sulphur absorb microwaves well compared to bulk metals or pure sulphur. The metal powder forms a polar-like environment in which ion mobility under the oscillating field produces heat, to render dispersion necessary for effective microwave absorption.

### Conventional versus Microwave synthesis

S. No	Conventional synthesis	Microwave synthesis
1	A heat source first heats the reaction vessel, after which heat is transferred to the reaction medium.	The reaction vessel is transparent to microwave radiation; the reaction mixture is heated directly.
2	Physical contact between the reaction vessel and reacting materials is required.	No need for physical contact between the reaction vessel and reacting materials.
3	Thermal or electric source of heating takes place.	Electromagnetic wave heating takes place
4	The heating mechanism involves conduction.	The heating mechanism involves ionic conduction and dipolar interaction
5	The rate of heating is lower.	The rate of heating is significantly higher
6	The highest temperature of the reaction is limited to the boiling point of the solvents or reaction mixture.	The temperature of reaction can be raised above the boiling points of the solvents or reaction mixture.

**Microwave reactor instrumentation:** The laboratory can use one of two kinds of microwave reactors, Monomode and

multimode microwave reactors.



**Fig 1:** Multi mode microwave reactor

## Monomode Microwave Reactor

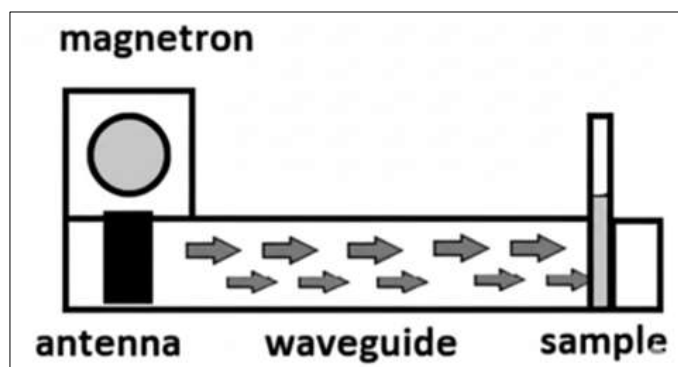


Fig 2: Monopod microwave reactor

### Microwave reactors with multiple modes

Microwave reactors, initially developed for cooking, are now used in organic synthesis on a laboratory scale, often using home microwave ovens with limited power (800-1000 W) and non-uniform electric fields. To improve synthetic applications, modifications like adding condensers and using microwave-inactive coolants have been proposed. Larger multimode reactors can handle multiple reactions simultaneously with in-phase microwave irradiation but have limitations such as heterogeneous electric fields, inaccurate temperature measurement, and non-tunable power. Multimode devices can heat multiple samples and larger volumes, enabling bulk heating and chemical processes, including continuous-flow production at kilogram scales. However, they lack precise control over sample heating.

Monopod microwave ovens are designed to allow only one mode of microwave propagation, achieving a uniform electric field and energy-efficient heating with higher yields in organic synthesis, especially for thermally unstable products. For organic synthesis, specialized microwave reactors such as rotating solid phase microwave reactors

(RSPMR), continuous microwave reactors (CMR), and microwave batch reactors (MBR) have been developed. Single-mode reactors can only irradiate one vessel at a time, but they can produce standing wave patterns with high-energy antinodes, allowing for rapid heating. They are suitable for automation, combinatorial chemistry, and drug discovery on a small scale since they can accommodate the capacities of 150 mol or less in open vessels and 02-50 mol in closed vessels.

### Interaction of microwaves with different materials

Microwaves are electromagnetic waves between radio and infrared frequencies, with wavelengths of 1 m to 1 mm (MHz to 300 GHz). There are only particular frequencies reserved for industrial, medical, or scientific use due to their widespread application in telecommunication (mobile phones, radar). For the purpose of avoiding interference, microwave ovens typically operate at 12.2 cm (2.45 GHz). The frequency is also applied for the warming of chemicals in industrial microwave reactors. Heating occurs based on the material's properties when liquids and solids absorb microwave energy and convert it into heat.

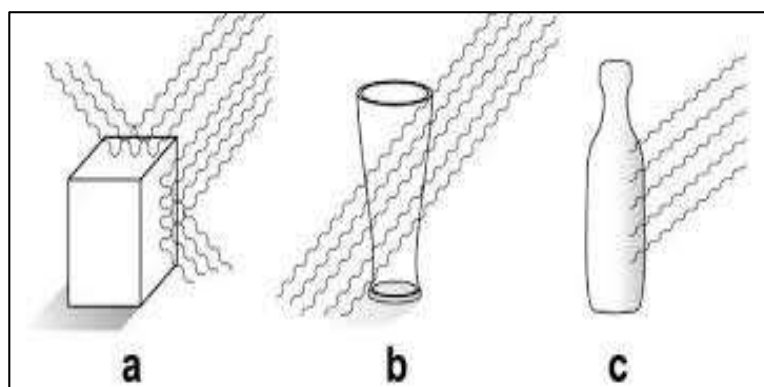


Fig 3: Interaction of microwaves with different materials (a) electrical conductor, b) insulator, (c) Lossy dielectric

### Types of Microwave Assisted Reactions

#### • Microwave Assisted Reactions using solvents

Reactants are generally dissolved in microwave-absorbing solvents that efficiently transfer energy in microwave-assisted organic synthesis. The hydrophobic effect is utilized at temperatures below and above 100 °C, and more research is being conducted on the application of aqueous media for organic reactions. Water at room temperature acts similar to organic solvents such as acetone since its dielectric constant

reduces from 78 at 25 °C to about 20 at 300 °C. Hot water can hence be employed as a safe and eco-friendly alternative to organic solvents.

#### Below are some examples of solvents

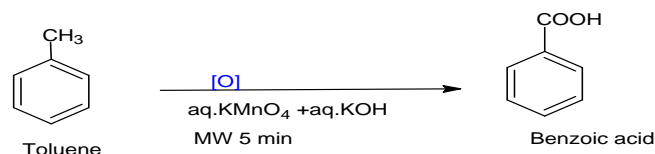
##### • Hydrolysis

Hydrolysis of benzyl chloride with water in microwave oven gives 97% yield alcohol in 3 min. The usual hydrolysis in normal way takes about 35 min.



### • Oxidation

Oxidation of toluene with  $\text{KMnO}_4$  under normal conditions takes 10-12 hr. Compared to reaction in microwave conditions, which takes only 5 min.



### Solid-liquid phase microwave-assisted reactions:

In organic synthesis, solid-liquid phase transfer catalysis (PTC) is a productive method, especially for anionic reactions. It entails mixing a catalytic quantity of a tetralkylammonium salt or cation complexing agent with pure reactants. Only the electrophilic reactant (R-X) is present in the liquid organic phase, where the reaction takes place. R-X functions as both the reactant and the organic phase because it is undesirable to add more liquid because it dilutes reactants and reduces reactivity.

Following are some examples of solid liquid phase:

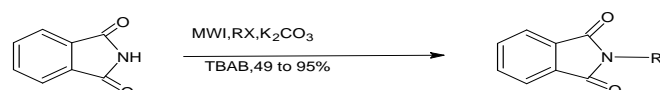
#### O-Alkylation

Under microwave irradiation, ethers were prepared from  $\beta$ -naphtha using benzyl bromide and 1-butyl-3-methylimidazolium tetrafluoroborate; the products were isolated in 75-90% yields.



#### N-Alkylation

Using phthalimide, alkyl halides, potassium carbonate, and TBAB, N-alkylation under microwave irradiation is used to synthesise N-alkyl phthalimides, yielding products in 45-98% yield.



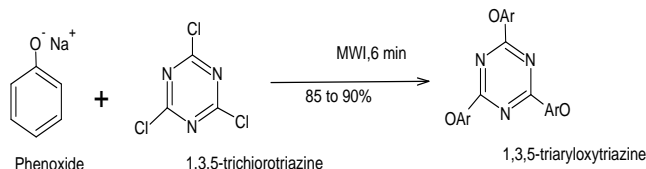
### Microwave assisted reactions under solvent free conditions

The need for solvent-free reactions and effective synthetic techniques is growing as a result of environmental concerns. Both conventional and novel methods are employed to reduce pollution from chemical reactions, with microwaves emerging as a significant energy source in numerous lab applications.

Following are some examples of solvent free conditions

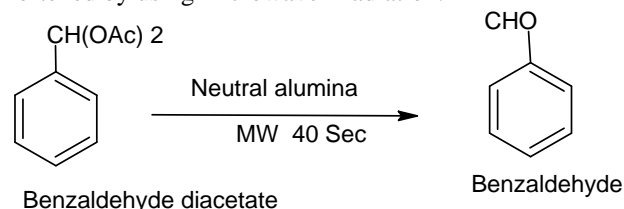
#### • Nucleophilic Substitutions

Under microwave irradiation, sodium peroxide and 1, 3, 5-trichlorotriazine are used to form substituted triazines. 85-90% yields of the products 1, 3, and 5-triaryloxytriazines are obtained.



### Deactivation

The traditional method produces low yields and takes a long time. The yields are good and the deactivation time is shortened by using microwave irradiation.

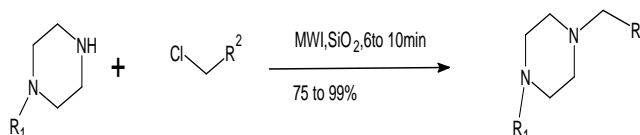


### Microwave assisted reactions on mineral supports in dry media

Solid supports are efficient microwave absorbers that allow for extremely quick and even heating, despite frequently being poor heat conductors. Compared to traditional heating techniques, this potent microwave effect results in improved temperature control, quicker reactions, and less product degradation. Following are some examples on mineral supports in dry media.

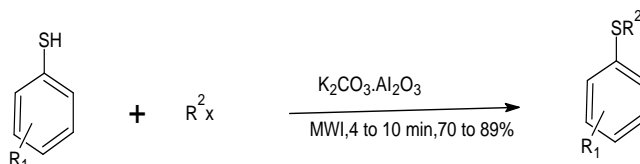
#### N-Alkylation

N-Alkylation were carried out between piperidines and chloroalkanes in the presence of silica as the solid support under microwave irradiation. N-Alkyl products were Isolated in 79-99% yields.



#### S-Alkylation

Under microwave radiation, mercaptobenzene and alkyl halides reacted with potassium carbonate and alumina to perform S-alkylation. Yields of 70-80% of the products were isolated.



### Advantages

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

### Disadvantages

- Heat force control is difficult
- Water evaporation



- Closed container is dangerous because it could be burst
- Sudden increase in temperature may led to distortion of molecules

### Applications of Microwave chemistry

Chemical synthesis is accelerated by microwave radiation, which enables organic chemists to work more quickly, generate greater yields, and obtain purer products. Without changing the reaction conditions, high-capacity microwave equipment makes it simple to scale up from milligrammes to kilogrammes. Microwave-assisted organic synthesis is the technique's main component.

### Organic synthesis

The process of creating desired compounds from readily available starting materials is known as organic synthesis. Richard Gedye and associates initially demonstrated the well-researched process of microwave-assisted organic synthesis when they hydrolysed benzamide to benzoic acid at reaction rates five to a thousand times faster than conventional heating. Since then, microwaves have been used to successfully carry out a number of organic reactions. Chemists have since carried out a wide variety of organic reactions with success.

#### These includes following

- Diels-Alder reaction
- Ene reaction
- Heck reaction

- **Organic synthesis at atmospheric pressure**

Microwave-assisted organic reactions are most effective under reflux and atmospheric pressure. For example, the Diels-Alder reaction between maleic anhydride and anthracene completes in one minute with 90% yield using diglyme, compared to 90 minutes by traditional methods with benzene. Using dipolar, high-boiling solvents is important for efficient microwave-assisted synthesis.

- **Organic synthesis at elevated pressure**

In sealed microwave-transparent containers, solvents can be heated to higher temperatures under increased pressure, significantly accelerating microwave-assisted organic synthesis. Reaction rates depend on factors such as vessel volume, solvent-to-space ratio, and solvent boiling point.

- **Organic synthesis in 'solvent -free 'open vessel reaction**

Microwaves used with solid supports in dry media significantly reduce reaction times in organic synthesis, including condensation, acetylation, and deacetylation. For example, deacetylation of alcoholic acetate on a support takes 2-3 minutes with microwaves versus 40 hours using traditional oil-bath heating at 75 °C.

- **Microwave -assisted synthesis of ceramic product**

In the field of ceramics, the development of microwave-assisted synthesis has significantly advanced, allowing for faster and more efficient processing. Initially, microwave heating was mainly utilized for drying and removing solvents, but it is now valued for its energy efficiency, especially for materials containing less than 5% moisture, as well as its ability to heat materials more evenly compared to conventional methods..

### Conclusion

Microwave-assisted synthesis is a key tool in medicinal chemistry and drug development due to its rapid, uniform heating, eco-friendly nature, and high product yields with minimal by-products. This Green Chemistry approach reduces reaction time, minimizes side reactions, and enhances reproducibility. It relies on polar solvents or solvent-free conditions to accelerate reactions and lower environmental impact. Although complex, multi-step drug syntheses remain challenging, microwave methods significantly improve the efficiency of developing and optimizing new therapeutic compounds.

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