



Review Article

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## Microwaves in Organic Synthetic Chemistry- A Greener Approach to Environmental Protection: An Overview

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### ABSTRACT

Microwave assisted organic synthesis is an environment friendly approach to synthesis as it is simple, innovative, gives high yield at low cost, and reduces the use of solvents. Conventional methods used in chemical synthetic processes involve the use of substances that are harmful to the environment. To cope with this issue, chemists were in search of a green alternative to conventional chemical practices that resulted in the development of a new branch of chemistry known as "Green chemistry". It foresees minimum impact to the environment as a primary criteria while developing any new chemical process. This predetermined target of green chemistry is achieved by considering different gist areas such as elimination of the use of traditional organic solvents if possible, finding alternative reaction media, conditions to minimize undesirable chemical waste formation, reaction rate enhancement using microwaves as energy source, etc. Microwave technique involves energy transfer that leads to rapid and uniform heating of the dielectric materials which often results in homogeneity and increased yield of the resultant products. It has numerous advantages over conventional thermal process such as shorter reaction time, better yield product, and less energy consumption. The present article describes microwave irradiation as a valuable energy efficient alternative to the conventional heating for greener organic synthesis.

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## Graphical Abstract



## Introduction

Environmental protection has been of prime importance recently due to the persistent increase in toxic and hazardous pollutants generated. Green chemistry also known “environmentally benign chemistry” is the emerging field of chemistry that focuses on the principle of four R’s, i.e. Reuse, Reduce, Recycling, and Recovery. The main goal of green chemistry is minimization of the use of toxic solvents and non-use of hazardous materials in chemical synthesis [1-3].

Also called sustainable chemistry, it is a philosophy of chemical research and engineering that encourages the design of products and processes that minimize the use and generation of hazardous substances [4].

Green chemistry counts on environmentally benign substance utilization to avoid the production of harmful wastes. Horvath *et al.* have described sustainable chemistry as “resources including energy should be used at a rate at which they can be replaced naturally and the generation of wastes cannot be faster than the rate of their remediation [5]. In the 1990’s, Paul Anastas and John C. Warner put forward 12 principles of green chemistry [6]. These principles propose environmentally favorable actions right from the planning of the product to its synthesis, processing, analysis, and its destination after use [7]. These 12 principles are as follow:

1) It is better to prevent waste than to treat or clean up waste after it is formed.

2) Synthetic methods should be designed to maximize the incorporation of all materials used in the process into the final product.

3) Wherever practicable, synthetic methodologies should be designed to use and generate substances that possess little or no toxicity to human health and environment.

4) Chemical products should be designed to preserve efficacy of function while reducing toxicity.

5) The use of auxiliary substances (e.g., solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.

6) Energy requirements should be recognized for their environmental and economic impacts and should be minimized. Synthetic methods should be conducted at ambient temperature and pressure.

7) A raw material or feedstock should be renewable rather than depleting, wherever technically and economically practicable.

8) Reduce derivatives- unnecessary derivatization (blocking group, protection/deprotection, and temporary modification) should be avoided whenever possible.

9) Catalytic reagents (as selective as possible) are superior to stoichiometric reagents.

10) Chemical products should be designed so that at the end of their function, they do not persist in the environment and break down into innocuous degradation products.

11) Analytical methodologies need to be further developed to allow for real-time, in-process monitoring, and control prior to the formation of hazardous substances.

12) Substances and the form of a substance used in a chemical process should be chosen to minimize potential for chemical accidents, including releases, explosions and fires. One of the main goals of Green chemistry is to develop mild and the modest reaction conditions that consume the minimum energy to make the

chemical process more economic. While exploring various alternatives to traditional chemical transformations, different aspects of green chemistry are applicable. Microwave Assisted Organic Synthesis (MAOS) is considered as a "green" technology as it has shown broad applications as a very efficient way to enhance the course of many organic reactions; no need of prolonged heating, producing high yields and greater selectivity, formation of cleaner products and many organic reactions can be carried out in solvent-free conditions [8-12]. A chlorination plant, hydrogen cyanide preparation, drying of pharmaceutical powders, pasteurization in food industry, etc. are some major examples of the industrial applications of microwave assisted rate enhancement in chemistry.

Today, microwave assisted organic synthesis (MAOS) has become an important tool to chemists for rapid and efficient organic synthesis. A huge work on MAOS exists in the published and patent literature [13-18]. Many reviews [19-33], several books [34-40] and information on the world-wide-web [41] provides extensive coverage of the subject. Microwaves are being used as a heat source for chemical synthesis [42] and it is predicted that Microwaves would be the heat source in all laboratories in the next coming years [34].

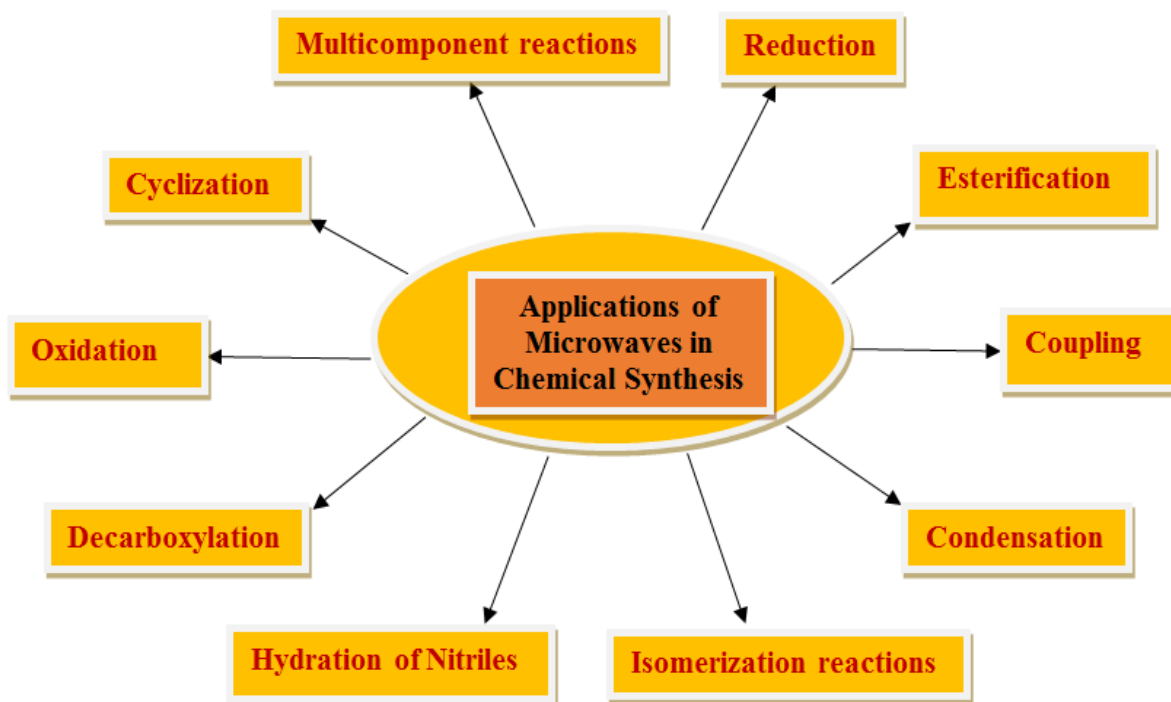
D. M. P. Mingos *et al.* gave a thorough explanation of the theoretical aspects of microwave dielectric heating [43]. Gedye [13] and de la Hoz [44] have discussed the suggested 'specific microwave effect'.

Loupy *et al.* [8] published a number of reviews on solvent-free microwave assisted reactions and Strauss has reported an organic synthesis in high temperature aqueous systems [20]. Recently, Microwave-Assisted Condensation Reactions of Acetophenone Derivatives and Activated Methylene Compounds with Aldehydes Catalyzed by Boric

Acid under Solvent-Free Conditions was reported [45]. Synthesis of multicomponent compounds like 3-(4-Arylmethylamino)butyl-5-arylidene-rhodanines using microwave irradiation method has been reported recently [46].

It is important to note that the country in which the technique seems to be most accepted, according to the number of publications, is India [27]. The growing popularity of this technique is

attributed to acceleration in reaction rates and hence quicker synthesis. For example, the hemolysis of 2,2,6,6-tetramethylpiperidinyl-1 (dimethyldicarboxylpropyl)ol and subsequent reaction with malonyl radical to form carboaminoxylation product takes three days under conventional conditions (DMF, sealed tube, and 135 °C) and the same transformation occurs in 10 minutes in microwave at 180 °C [24].



**Figure 1.** Applications of microwave assisted synthesis

### *Microwaves in organic synthesis*

#### *History*

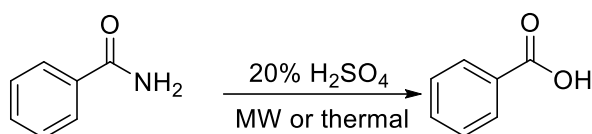
During the World War II, Randall and Booth (University of Birmingham) made an outstanding contribution by inventing the cavity magnetron used to generate microwaves. This innovation was used to build microwave radar transmitter equipments and led to the development of a prototype microwave oven that was installed in Boston restaurant for testing. The Raytheon Company patented

Microwave dielectric heating [47] in 1946 and made available commercial microwave oven in 1947 called Radarange. The use of microwave dielectric heating in different branches of chemistry has expanded very rapidly when R. Gedye and G. Majetich [48-50] and their coworkers in 1986, suggested that the rate of many organic reactions is enhanced under microwave conditions.

Nowadays, there are more than thousands of research articles describing the applications of this technique for the synthesis of new

compounds. Microwaves have a lot of applications in the chemical and industrial field since the 1950s, particularly in the food processing, drying, and polymer industries. Microwave energy has only been implemented in organic synthesis since the mid-1980s by Richard Gedye [48] and J. Giguere/George Majetich [50].

The first example of microwave assisted organic synthesis was published in 1986. It is the hydrolysis of benzamide, as shown in the following:



Thermal: 1 h, 90% yield (reflux)  
MW: 10 min, 99% yield (sealed vessel)

**Scheme 1.** Hydrolysis of benzamide

In the 1990s, several groups started to experiment with solvent free microwave irradiation, which eliminated the danger of explosion [8-12].

#### *Microwave frequency range for dielectric heating*

Microwaves are electromagnetic radiations that belong to the lower frequency end of the electromagnetic spectrum and are located between infrared radiation and radio waves. The microwave region of electromagnetic spectrum range was between wavelengths of 1 cm to 1 m (30 GHz to 300 GHz) [51]. In this region of electromagnetic energy, only molecular rotation is affected and not molecular structure.

A frequency of 2.45 GHz (12.2 cm) is the preferred frequency for industrial, scientific, and medical applications because it has the

right penetration depth to interact with the materials. Hence, domestic and commercially available microwave applicators for chemical purposes generally operate at this frequency (2.45 GHz).

#### *Theory of microwaves*

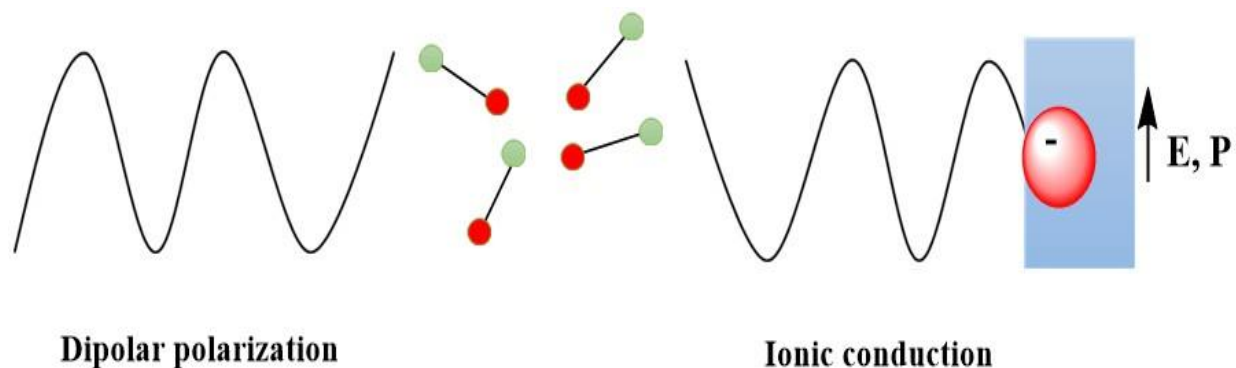
In microwave dielectric heating, the microwave radiation passes through the walls of the vessel and heats only the reactants and solvent, not the reaction vessel itself. If the apparatus is properly designed, the temperature increase will be uniform throughout the sample, which can lead to less by-products. Microwave dielectric heating [43] is a very efficient process due to the microwave couple directly with the molecules that are present in the reaction mixture, leading to a fast rise in temperature, faster reactions, and cleaner chemistry.

Microwave dielectric heating involves aggregates of molecules of the substance which have the capacity to transform electromagnetic energy into heat for carrying out the chemical reactions.

Energy transfer is rapid between these molecules and this limits the extent of localization of the heating. The interaction between electromagnetic waves and matter is quantified by the dielectric permittivity and magnetic susceptibility.

Like all electromagnetic radiation, microwave radiation can be divided into an electric field component and a magnetic field component. The electric field component is responsible for microwave dielectric heating.

Fundamental mechanisms responsible for transferring energy from microwaves to the substance are Dipolar polarization and Ionic conduction (Figure 1).



**Figure 2.** Mechanism of microwave heating

### *Dipolar polarization*

The phenomenon of dielectric heating in the microwave region is strongly connected with the dipolar polarization. It is the interaction in which polar molecules try to align themselves with the rapidly changing electric field of the microwave. For a substance to generate heat when irradiated with microwaves, it should possess a dipole moment, as in case of water molecules.

A dipole is sensitive to external electric fields and will try to reorient itself with the electric field by rotation. The applied field provides the energy for this rotation. In gases, the alignment of their molecules with the applied field is rapid as the molecules are spaced far apart while in liquids, the instantaneous alignment is prohibited by the presence of other molecules. The ability of liquid molecules to align with the applied electric field will vary with different frequencies and with the viscosity of the liquid. If two samples containing water and dioxane, respectively, are heated in a single mode microwave cavity at a fixed radiation power and for a fixed time the final temperature will be higher in the water sample (Figure 2).

This is because, dioxane which lacks the dipole characteristics necessary for microwave dielectric heating, does not heat while water,

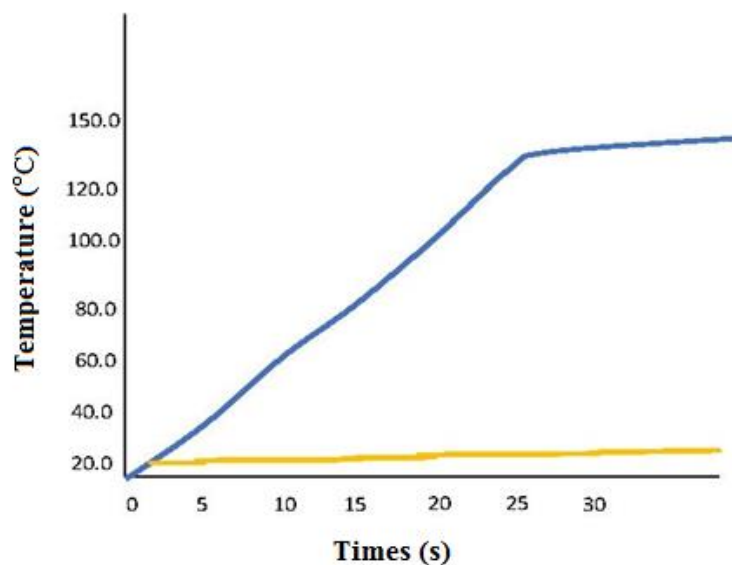
which has a large dipole moment, heats readily. Furthermore, gases could not be heated under microwave irradiation, since the distance between two rotating molecules is long enough for the molecules to be able to follow the electric field perfectly so that no phase difference will be generated.

Thus, microwave heating is only possible for polar molecules like water, methanol, ethanol, ammonia, formic acid, etc. The non-polar molecules fail to interact with the microwave radiation.

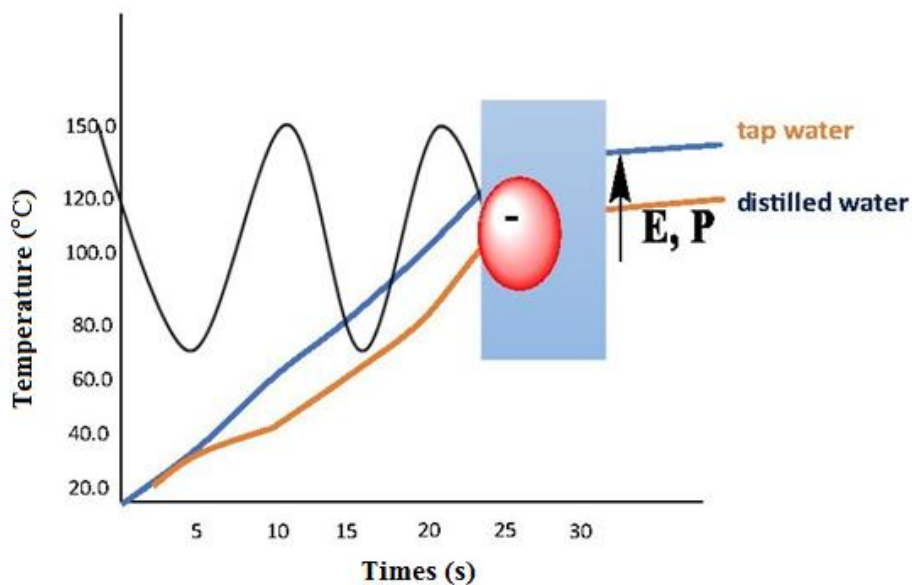
### *Ionic conduction*

This mechanism consists in the instantaneous superheating of the ionic substance due to the translational motion of the electric charges when an electric field is applied. Ions cause increased collision rate and convert kinetic energy to heat. When the temperature increases, the energy is transferred more efficiently. Ionic liquids absorb microwave radiation extremely well and transfer energy quickly by ionic conduction. If two samples containing distilled water and tap water, respectively, are heated in a single mode microwave cavity at a fixed radiation power and for a fixed time, the final temperature will be higher in the tap water sample (Figure 3).





**Figure 3.** The temperature increases in water and dioxane, respectively, at 150 W microwave irradiation. The upper curve represents water and the lower plot represents dioxane



**Figure 4.** Conduction: Tap water vsersus distilled water

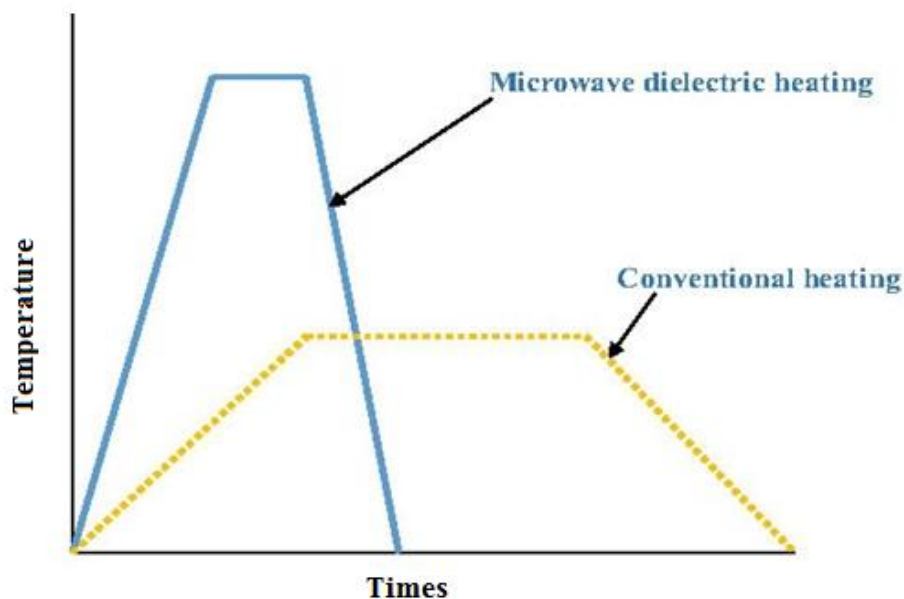
This conduction mechanism is due to the interaction of the electric field component with the sample. In the above example, the heat generated by the conduction mechanism due to presence of ions adds to the heat produced through the dipolar mechanism, resulting in a higher final temperature in tap water.

#### *Rapid reaction rates*

Based on the study of the available experimental data, it can be concluded that the microwaves can accelerate the heating rates thousand times as compared to conventional heating methods. Microwave-enhanced chemical reaction rates are much

faster since higher temperatures may be readily achieved which is difficult using conventional heating. In the microwave reactor, there is no direct contact between the microwave energy source and the sample subjected to heating. This leads to the completion of the reaction in very short time, i.e. in few minutes instead of some hours. Microwave heating technique is a

highly efficient process that saves significant amount of energy. This is due to the fact that microwaves only heat the sample molecules and provide them with instantaneous energy to cross the activation energy barrier and hence complete the reaction quickly with better product yield.



**Figure 5.** Comparison of conventional thermal heating and microwave dielectric heating

*Advantages microwave heating over conventional heating [52, 53]*

The benefits of microwave heating method in chemical synthesis can be summarized as follow:

1. Increased reaction rates,
2. Decrease in reaction time,
3. Improved chemical yields,
4. Uniform and Selective heating,
5. Milder reaction conditions,
6. Energy consumption is less,
7. Lesser side-products and higher purity so easier work up and purification step is faster,
8. Green and solvent-free chemical synthesis than conventional methods, and
9. Simplified synthetic procedure.

*Disadvantages microwave heating*

1. Closed container could burst so may be dangerous,
2. Water evaporation could be a problem, and
3. Heat force control is difficult.

*Effects of solvents in microwave assisted organic synthesis*

Microwaves heat only the sample or solvent and not the apparatus. Selective and uniform heating is done by microwaves and therefore in microwave heating energy consumption is less. Selective heating unlike conventional heating is based on the fact that different materials respond to the microwave radiation in a different way. Some may absorb the microwaves, but other materials may be transparent.



Microwave assisted synthesis can be carried out in the presence or absence of solvents. Every solvent absorbs the microwave energy differently, depending upon its tendency to couple with the microwaves and act as energy transfer medium. Although the solvent polarity is not the only factor in determining the absorbance of microwave radiation, it is important one. This is because a stronger dipole moment causes more rotation motion to align with the changing microwave field. Polar solvents like water and most alcohols can be used microwave assisted synthesis.

Non-polar solvents however are not heated under microwave irradiation. Another factor to consider while through solvents in microwave synthesis is the use of open or closed vessels.

Seddon K.R. and Welton T. [54] have reported ionic liquids as novel environment friendly and recyclable alternatives to dipolar aprotic solvents for chemical synthesis. Ionic liquids have huge benefits as solvents in organic synthesis because they absorb microwave radiation very efficiently, exhibiting a very low pressure and have enhanced suitability for microwave dielectric heating. Solvent-based methods have several benefits.

#### *Microwave assisted organic synthesis under solvent-free conditions*

Microwave reactions under solvent free conditions are an alternative technique that is environmentally benign. It provides a greener and eco-friendly synthesis that avoids the formation of toxic substances and use of hazardous solvents throughout the process leading to improved yield of products at reduced environmental cost. The use of solvents in the microwave assisted reactions can be eliminated as microwaves directly heat the compounds only. The solvent free microwave reactions have a great potential and these reactions may be carried out using a solid

mineral support, neat reactants, or solid-liquid phase transfer catalyst.

Microwave irradiated reactions under solvent-free conditions have various advantages such as simple work up, cleaner and faster reactions, greater yields, lesser risk of explosions, reduced use of polluting solvent reagents, etc. These methods have disadvantage of having a lack of facility for temperature measurement and for mixing substrate compounds.

#### *Modes*

In Microwave-assisted reactions, we make use of single-mode and multi-mode microwave ovens. The single-mode microwave has a unique ability to form a standing wave pattern inside it. Single mode microwave apparatus gives a high rate of heating as the sample is placed at the antinodes of the field where the intensity of microwave radiation is the highest and not at the nodes where the microwave intensity is zero. However, its disadvantage is that with this single-mode cavity apparatus only one vessel can be irradiated at a time. Cavity used in domestic multi-mode microwave ovens contain 3 to 6 different modes to give a uniform heating of food material. The use of multi-mode technique provides a field pattern with hot and cold spots resulting in the varying of heating capacity drastically between different positions of the load. Hence, in multi-mode microwave cavity, a well-defined heating wave pattern generation is avoided and it is used for bulk heating of samples. Unlike single mode microwave, here, simultaneous heating of a number of samples is possible.

#### **Conclusion**

Microwave Assisted Synthesis has emerged as an important and powerful tool of Green chemistry which we need in our quest to meet

the demand of modern civilization. Many chemical transformations can be carried out by the use of microwave irradiation which is an unconventional source of energy. The chemical processes that experience polar transition state are favored by microwave irradiation. The various advantages of Microwave Assisted Organic Synthesis (MAOS) technique include reaction rate acceleration, solvent-free reactions, minimum energy consumption, and no need of prolonged refluxing, etc. which is not easily achievable by the other conventional synthetic techniques. It appears that this fascinating technique cannot be easily replaced by other modern methods in the coming years due to its ability to superheat the substrates in an eco-friendly and safe manner with superb control on reaction conditions like pressure and temperature.

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### Authors' Contributions

All authors contributed to data analysis, drafting, and revising of the paper and agreed to be responsible for all the aspects of this work.

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