Chapter

Microwave Assisted Synthesis of Organic Compounds and Nanomaterials

Anjali Jha

Abstract

In the Conventional laboratory or industry heating technique involve Bunsen burner, heating mental/hot plates and electric heating ovens. To produce a variety of useful compounds for betterment of mankind, the Microwave Chemistry was introduced in year 1955 and finds a place in one of the Green chemistry method. In **Microwave chemistry** is the science of applying microwave radiation to chemical reactions. 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. Polar solvents are heated as their component molecules are forced to rotate with the field and lose energy in collisions i.e. the dipole moments of molecules are important in order to proceed with the chemical reactions in this method. It can be termed as microwave-assisted organic synthesis (*MAOS*), Microwave-Enhanced Chemistry (*MEC*) or Microwave-organic Reaction Enhancement synthesis (*MORE*). Microwave-Assisted Syntheses is a promising area of modern Green Chemistry could be adopted to save the earth.

Keywords: Green synthesis, Microwave synthesis, Organic reactions, Nanomaterials

1. Introduction

Chemistry play major part in our daily life in the form of medicines, food colours, soaps, detergents, sunscreen lotions, toothpaste, pharmaceuticals, etc. what we use in our daily life literally everything. Scientist from various disciplines had used their knowledge and skill to produce these materials. To produce these chemicals in industries and laboratory several techniques are used like stirring, heating, refluxing, protection of specific functional group and deprotection of them. Several harmful materials were also generated during the above processes causing environment pollution. To combat these problems, Green Chemistry has emerged as a challenge for scientist from industries and academia to develop the synthetic process for sustainable development of society in last few decades. In this context, several green synthetic techniques were developed to generate a vast library of organic compounds.

In the Conventional laboratory or industry heating technique involve Bunsen burner, heating mental/hot plates/furnace/oil bath and electric heating ovens. These process comprise the heating of walls of vessels first by convection or conduction of energy then the reaction content, leading to much longer time to achieve the target temperature. These techniques not only give useful materials but also give so many by products in the form of solid, liquid and gases, resulting to lot of chemical pollution to mankind as well as for other living creatures on Earth. Furthermore, to produce these useful materials, several resources are also going to be consumed. To combat these problems the various arena of Scientist are trying to develop new Green techniques.

Conventional heating usually involves the use of a furnace or oil bath, which heats the walls of the reactor by convection or conduction method. The core of the sample takes much longer to achieve the target temperature, e.g. when heating a large sample of ceramic bricks (**Figure 1**). The microwave portion of electromagnetic radiation is emerged a significant area in the acceleration of chemical reactions in 1980s. A number of reviews were reported by various scientists [1–10]. There are three main reasons for its special place of microwave (MW) assisted synthesis. The first point is high heating efficiency caused by MW energy, which reaches and absorbed directly by substance. The second is selectivity i.e. having dielectric polar molecule will absorb this energy. And thirdly enhancement of chemical syntheses by the MW effect or non-thermal effect. In other sense, the microwave irradiation energy acts as internal heat source, which is able to heat the target compounds without heating the entire furnace or oil bath, consequently, saves time and energy. It produces more uniform heating. This unconventional energy source (MW) has been used to heat food stuff since more than 5 decades and now the concept is also utilized to accelerate a wide range of chemical reactions. The organic synthesis has very special and significant role in the improvement of day to day life of human being from morning toothpaste to all useful medications/drugs, plastic/polymer materials etc. To produce these useful substance in reasonable time and environment friendly manner is the prime responsibility of the scientist of several discipline. In this regard Microwave heating technique has blossomed in a variety of applications in Organic Synthetic methods. Although the initial synthetic work was performed in domestic Microwave ovens but recently most organic reactions are carried out in dedicated high speed specialized Microwave equipment with appropriate pressure and temperature control.

Microwaves (MW) are the electromagnetic waves, having shortest wavelength of radio spectrum. The word microwave means "a very short wave". Microwave irradiation is a low energy phenomena as compared to other radiations. The energy of MW is about 0.0016 eV at 2450 MHz, which is very low to break any chemical bond. However, a microwave energy can effect a number of chemical transformation. When a substance is exposed to microwave energy, then three process can occur. Firstly, if it is a conductor metal, then MW will be reflected and it cannot be heated. Secondly, if insulator material is exposed to MW it will be not heated since it is MW transparent. Thirdly if material is dielectric in nature then MW energy will be absorbed and gets heated. This case is very interesting for a Chemist.

A microwave works by directly coupling with polar molecule in a reacting species. Microwaves radiation are a form of non-ionizing electromagnetic radiation

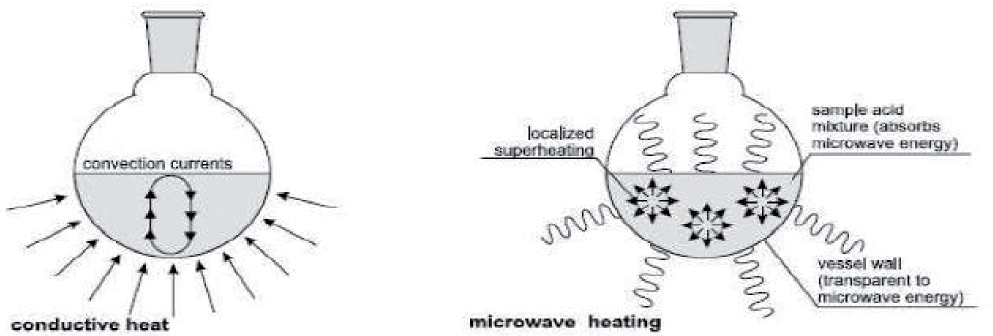


Figure 1.
Process of heating.

with 300 MHz to 300 GHz frequency with the corresponding wavelength 1 mm to 1 m, which places MW in between Infrared radiations and radio waves. The molecules get heated in microwave irradiation process by a dielectric heating phenomenon. The electric dipoles, i.e. a partial positive charge at one end and a partial negative charge at the other, rotate as they try to align themselves with the alternating electric field of the microwaves. Additionally, molecular rotation occurs in a material/food (containing polar molecules) causing electric dipole moment which can partially/completely aligned themselves with an applied electromagnetic field. Since electromagnetic field alternates, the rotating molecule resulting they change their directions by pushing/pulling/colliding. Rotating molecules hit other molecules and put them into motion, thus dispersion of energy happens. This energy, dispersed as molecular rotations, vibrations and/or translations in solids and liquids, raises the temperature of the food, in a process similar to heat transfer by contact with a hotter body. It is a common misconception that microwave ovens heat food by operating at a special resonance of water molecules in the food. As noted microwave ovens can operate at many frequencies.

Additionally, nanomaterials or nanoparticles (NPs) are the materials having a single unit small sized between 1 and 100 nm. During the past few decades nanoparticles research have become a subject of intensive interest because of its potential applications in various fields i.e. biosensing, drug delivery, bioimaging, catalysis, lubrication, electronics, textile manufacturing, water treatment systems. Nanoparticles (NPs) are synthesized from several types of materials i.e. inorganic, organic, hybrid and biological by adopting various methods. The main methods are ball milling, vapor deposition, electro-spraying, reduction of metal salts, sol–gel, coprecipitation and thermal decomposition for inorganic NPs. While organic NPs are synthesized by microemulsion, nanoprecipitation, dialysis and rapid expansion of supercritical solutions. Hybrid NPs are synthesized from both organic and inorganic materials. However, the present trend for the synthesis of nanomaterials endures a challenge to redesign the synthetic strategies offering the use of less hazardous chemicals and reduction in the reaction time and required energy. In this context, microwave (MW)-assisted methods can be considered as a promising green approach for synthesis of nanomaterials and nanocomposites. Besides, MW-assisted strategies offer a homogenous heating with reliable nucleation and growth environment leading to the formation of NPs with uniform size. Several reviews were reported on microwave assisted synthesis of NPs [11, 12].

2. Microwave equipment

Though the range of MW frequencies is 0.3 to 300 GHz, out of this most of the frequencies are used in Radar and Telecommunication equipment. However very limited frequency range can be used in microwave heating equipment i.e. 2.45 GHz corresponding to 12.2 cm wave length. So almost all domestic and commercial microwave heating equipment either for domestic or scientific purpose has a fixed frequency 2.45 GHz. In general MW accelerated reactions are carried out either domestic microwave oven or especially designed microwave equipment. These equipment works in between 500 to 1500 W power. In these ovens Microwaves are generated by magnetron, and the temperature is maintained by turning this on and off. The generated microwaves are travelled into vessel (cavity) and reflected back by the walls of cavity. If the generated microwaves are not absorbed, it may reflect back down and damage the magnetron. Thus it is essential to have microwave active dummy load should be used during the process, which will absorb excess microwave and prevent such damage.

3. Solvent used in MOAS

In Microwave solid and liquid phase reactions can be performed at various conditions. The boiling point of solvents will be raised by 25^0 C with their actual boiling points during the process of microwave heating [13]. Although all type of solvents can be used in MOAS, however DMF, ethanol and water are the best solvents for this process, since they absorb MW radiation and heat the content efficiently. All polar solvents comprising OH groups are suitable for MW reactions, since they can able to absorb MW energy. Water, alcohols (ethanol & isopropanol), amides, acetonitrile and acids (acetic and formic) are some of the commonly used solvents in this process. The polarity order of these solvents are like

$$\text{Water} > \text{Acids} > \text{Amides} > \text{Alcohols} \quad (1)$$

The less polar or non-polar solvents are transparent to microwave irradiation i.e. could not absorb MW energy and consequently will not get heated in the pure form. However in general the reaction mixture content contains enough polar or ionic substance, which can absorb microwave irradiation and generate heat to complete the reactions. Moreover small amount of polar or ionic additives could be added to enhance the absorption capacity of reaction content having less dielectric properties of less polar solvents. The energy transfer between polar molecule and non polar solvents under microwave irradiation is so rapid and efficient leading to proceed a variety of chemical transformation and thus a very good alternative for conventional techniques in tune with green chemistry [14].

4. Types of microwave energy assisted synthesis

In general two types of Microwave assisted synthesis were reported in literature: -

I. Microwave assisted synthesis using solvents

II. Microwave assisted synthesis without solvents or solvent free conditions

The reports on Microwave assisted synthesis are well documented in the literature since last 5 decades, which includes mainly following reactions **Figure 2.**

There are several synthesis which can be performed in presence of solvent under microwave conditions:.

a. **Hydrolysis:**

The hydrolysis of benzyl chloride can be successfully done within 3 minutes in MW while normally it takes 35 min.

$$C_6H_5CH_2Cl + H_2O \xrightarrow{\text{MW, 3 min}} C_6H_5CH_2OH \quad (2)$$

Similarly hydrolysis of benzamide in presence of sulphuric acid can be completed in 7 mins instead of one hr. under MW.

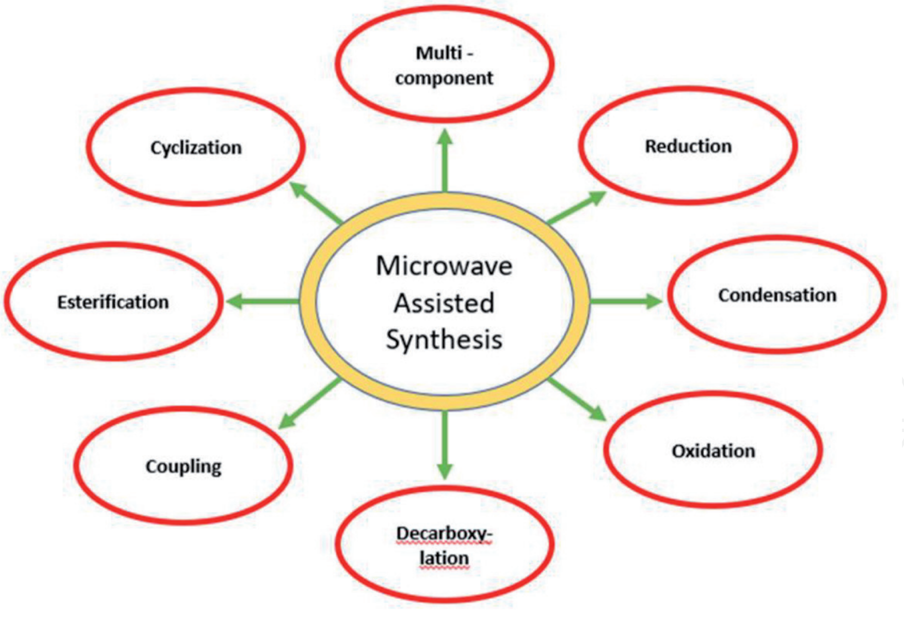


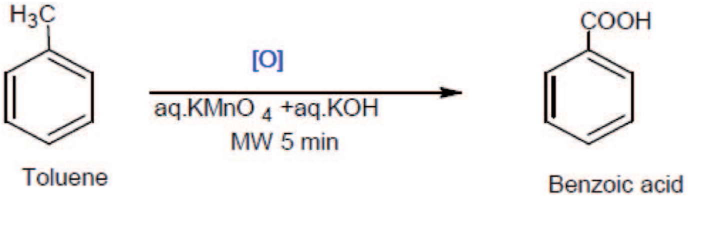
Figure 2.
Various types of Microwave assisted synthesis.

$$\underset{\text{Benzamide}}{C_6H_5CONH_2} \xrightarrow[\text{MW 7 min}]{20\%H_2SO_4} \underset{\text{Benzoic acid 99\%}}{C_6H_5COOH} \quad (3)$$

The acid-sensitive 3-hydroxyacetals and 3-methoxyacetals being hydrolyzed efficient method in minutes in good yields. In this paper reports on efficiently hydrolyzed acetals with silica gel-supported pyridinium tosylate moistened with water in solvent-free conditions under microwave irradiation were reported [15]. Since the hydrolysis of carboxylic acid esters is one of the most studied chemical reactions Safari et al. had reported Microwave-assisted expeditious hydrolysis of isobenzofuranone derivatives using silica supported acid under organic solvent-free conditions [16].

b. **Oxidation:**

The oxidation of Toluene with potassium paramagnet usually takes 10–12 hrs in normal condition, however under MW condition within 5 mins it is converted to benzoic acid [17].



(4)

Since the oxidation of alcohols plays a significant role in organic synthesis and as carbonyl compounds are widely used as intermediates both in manufacturing and laboratory. The progress of new oxidative methods continues to be a focus of research area in spite of the availability of several protocols to achieve such objectives. Bogdał and Łukasiewicz reported [18] an interesting protocol of oxidation of primary and secondary alcohols into equivalent carboxylic acids and ketones within 10–20 min using 30% aqueous hydrogen peroxide and commercially available catalysts under microwave irradiation. The application of hydrogen peroxide as an oxidant is appreciated because water is the sole expected side product.

Further, alcohols are adsorbed on clayfen easily oxidized to carbonyl compounds under solvent-free conditions in microwaves. By this rapid, selective and environmentally benign method, the use of excess solvents and toxic oxidants usually employed can be avoided. Varma and Dahiya reported the oxidation of alcohols to the corresponding carbonyl compounds in presence of clay-supported iron(III) nitrate, under solvent-free dry microwaves conditions [19].

c. **Esterification:**

The esterification of benzoic acid can be achieved in 6 mins only [20] in the presence of catalytic amount of sulphuric acid under MW conditions.

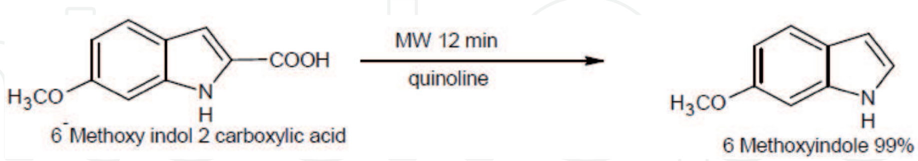
$$\underset{\text{Benzoic acid}}{C_6H_5COOH} + nC_3H_7OH \xrightarrow[\text{MW 6 min}]{\text{Conc } H_2SO_4} \underset{\text{Propylbenzoate}}{C_6H_5COOC_3H_7} \quad (5)$$

Since esters are one of the important products for all industrial applications. In this context convenient and feasible technique for synthesis of esters was reported [21, 22] in dry media and using heteropoly acid as catalyst respectively under microwave irradiation in the absence of organic solvents.

Recently Reilly et al. [23] demonstrated the microwave assisted esterification reaction to undergraduate student, which is a good initiative to develpo the interest among students about green chemistry.

d. **Decarboxylation:**

The conventional decarboxylation of carboxylic acid usually carried out in presence of quinolone by refluxing the acid however in microwave it can be done in very less time [24].



(6)

The reports on microwave assisted decarboxylation of malonic acid derivatives are available in the literature [25]. They carried out these reactions under solvent-free and catalyst free conditions. This new method produces the corresponding carboxylic acid in a pure manner and with a high yield within short time: 3–10 min.

e. **Multicomponent reactions**

The multi component reaction (MCR) approach has emerged as a promising alternative to incorporate the various diversity in a single step reaction to produce diverse library of important compounds. A rapid one-pot synthesis of imidazo [1,2-a] annulated pyridines, pyrazines and pyrimidines was described by Varma and Kumar, in the presence of recyclable montmorillonite K 10 clay under solvent-free conditions using microwave irradiation [26, 27]. They condensed three components viz.: aldehydes(aliphatic/ aromatic/vinylic); isocyanides (aliphatic/aromatic/ cyclic) and amines (2-aminopyridines, 2-aminopyrazines and 2-aminopyrimidines in presence of catalytic amount of clay in solvent free microwave irradiation.

Mahmood et al. 2017 conveniently synthesized thiazolidinones from aldehydes, thiosemicarbazide and maleic anhydride in the presence of KSF@Ni as heterogeneous catalyst in one-pot three-component reaction under microwave irradiation in good yield in short reaction time [28].

The metal free the microwave assisted synthetic multicomponent approach for N-unsustituted-1,2,3-triazoles was recently reported by Garg et al. [29]. They used aromatic aldehydes, nitroalkane and sodium azide for synthesis, which demonstrates great scope to be utilized by future generation for production of novel biological active molecules.

Since acridines and acridinium ions are one of the important compounds, as they are used in pharmaceuticals, materials, dyes and photo-catalysis. Mandal et al., 2020, reported [30] an unconventional $FeCl_3$-alcohol catalysed one-pot synthesis of these materials by aldehydes, 1,3-cyclohexanedione and amines under microwave conditions. They beautifully merged high atom-economy and multicomponent reaction with novel iron catalyzed dehydrogenation, using aerobic oxygen as the terminal oxidant, in alcoholic solvent to produce water as the by-product.

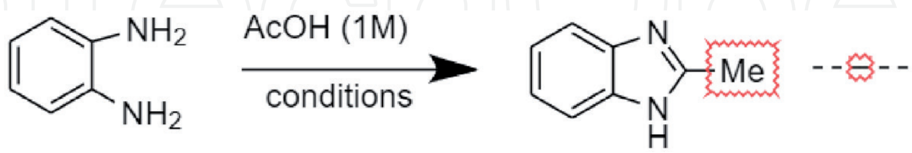
f. **Condensation reaction**

Several known transformation such as hydrolysis of ester or amide, Diels-Alder cycloaddition, Fisher Indole synthesis were investigated in a microwave reactor, in near critical water (NCW) under microwave assisted synthesis and reported [31].

Ashry and Kassem, 2006 has given a comprehensive account [32] of synthesis of Six, Seven membered, Spiro and Fused Heterocycles under microwave conditions.

Naqvi et al. 2009 were developed and synthesized some Schiff bases under microwave irradiation by taking 3-chloro-4-fluoro aniline and various substituted benzaldehydes [33]. They used other green chemistry approach also for their synthesis.

The thermal effect of microwaves were shown in 2006 by condensing ortho-diphenylamine in acetic acid and observed that by conventional means it took 9 weeks while in MW only tem minutes [34]. It was a great findings and opportunities for synthetic chemists.



(7)

Conditions	Temperature/Pressure	Time
Conv	25°C	9 weeks
Conv	60°C	3 days
Conv	100°C	5 hrs
MW	130°C /2 bar	1 h
MW	160°C/ 4 bar	10 min
MW	200°C /9 bar	3 min
MW	270°C / 29 bar	1s

Sondhi et al., 2010; have developed microwave assisted synthesis of complex tri-an d tetracyclic heterocyclic molecules in a simple, high yielding, one step process [35]. They synthesized these important moieties by condensing of dicarboxylic acids with diamines under solvent-free condition.

The design and synthesis of novel structures having α, β-unsaturated ketone moieties, responsible for the bioactivities of the Mannich bases were carried out by Mete et al. [36]. The target compounds were synthesized by the conventional heating method and also by the microwave irradiation method. They optimized the conditions which can be explored by future researcher.

The reports on the condensation reaction between active methylene group and various aldehydes are well documentated in literature since several decades for their potential diverse biological activities especially for anticancer activities [37]. Furthermore, the various substituted aromatic aldehydes and Thiazolidine-2,4-dione were reacted under microwave irradiation using water as green solvent by simple Knoevenagel condensation. They tried various solvent conditions aqueous ethanol, H2O, DMF, DMSO, and solvent less under MW irradiation [38] and found the water as the best suitable solvent.

The 1,3,5-trisubstituted benzene was synthesized by self-condensation of enamines, enaminones and enaminoester in the presence of pyridinium chloride ([PyH]Cl) in short time in domestic MW oven and ionic liquids in year 2010 with appreaciable yield [39]. Brun et al. 2015 prepared boric acid catalyzed novel acetophenone derivatives with methylene compounds under solvent free microwave assisted condensation reactions [40]. They prepared a large number of compounds having sensitive acid and base function groups in substrates. Knoevenagel Condensation.

A comprehensive review on reactions of cyano malononitrile-derivatives under Microwave Radiation was also reported recently [41]. The Knoevenagel condensation between aromatic aldehydes or ketones and malonic acid in the presence of tetrabutyl ammonium bromide and K2CO3 was carried out and reported by Gupta and Wakhloo 2007 under microwave irradiation in water. The yields of products were excellent with high purity [42].

g. **Cycloaddition reactions**

Since Computational calculations emerged as a significant tool to study reaction kinetics and strategies in last couple of decades. de Cozar et al. [43] studied the thermal and non-thermal effects of microwave irradiation and determined the thermodynamic and kinetic parameters of the reaction. They performed the computational study of two previously reported cycloaddition reactions and analysed the presence of a thermal effect which is responsible for the microwave irradiation that produces changes in the regioselectivity or in the reaction mechanism.

The 1,2,3- triazoles are one of the most active moieties presents in several types of drug candidate. So, the convenient synthesis is a hot topic among various researchers. The catalyst and solvent free synthesis of substituted triazoles via cycloaddition of trimethylsilylazide and acetylenes were carried out recently by Roshandel et al. [44] under microwave conditions In the same year Expeditious microwave assisted synthesis and bio-evaluation of novel bis (trifluoromethyl) phenyl-triazole-pyridine hybrid analogues by click chemistry approach under microwave irradiation was reported from our group [45].

h. **Coupling reactions**

The efficient synthesis of 2,3 disubstituted-6-aminoquinoxaline derivatives were prepared efficiently by applying Microwave assisted Sonogashira Coupling by Lee et al. in 2013. They prepared a library of compounds by very region-selective MW conditions [46]. The mini-review on the Suzuki-Miyaura and on the Heck cross-couplings of nucleosides under microwave irradiations reported as an alternative technology in tune with green chemistry [47]. A simple microwave assisted synthesis of isoindolinones bearing a quaternary C-atom, introducing a Pd-mediated tandem-coupling reactions are reported recently [48]. As Cross-coupling reactions furnishing carbon–carbon (C–C) and carbon–heteroatom (C–X)bond is still one of the most challenging area in organic syntheses. Younis Baqi has reported [49] a comprehensive review on Recent Advances in Microwave-Assisted Copper-Catalyzed Cross-Coupling Reactions very recently. The microwave energy was also used in the synthesis of Ribonucleosides using natural phoshphate as catalyst in 2013 by Ouzebla et al. [50]. The hydrogenation of Levulinic acid to γ-Valerolactone in presence of Ru/TiO2 catalysts was reported very recently by Howe et al. 2019 [51]. The one-pot microwave-assisted polyol method was found to be highly efficient. The preparation temperature, microwave irradiation time and choice of Ru precursor have a significant effect on catalyst activity.

The extraction of bioactive components from fruits of *Ficus racemosa* was recently carried out by Sharma et al. 2020 under the microwave – assisted extraction (MAE). They developed the optimum condition for MAE at 3.5 pH,360.55 W microwave power in 30.01 s, which can be used as promising green technology to extract other useful materials from other natural sources [52]. The microwave synthesis of hybrid inorganic–organic porous materials was reported by Jhung et al. 2006. They observed phae selective rapid crystallization of hybrid material by MW method, which could be better alternative method to be explored further [53].

The comparative study of conventional and Microwave induced organic reaction enhancement (MORE) methodologies was carried out to synthesize some selected heterocyclic molecules and found more efficient over conventional classical methods in 2010 [54]. Kitchen et al. 2014 reported [55] a comprehensive review on the significant advances in the area of solid-state MW synthesis in context with future scope in the 21st century. They also presented the merits of MW heating along with understanding of MW heating and interaction mechanisms. The major focus of the review is on the use of MW heating to make novel improved materials with MW heating. They analytically discussed the developments in MW techniques and instrumentation and their potential to foster interest among the materials chemistry research and industrial communities and to explore the access to MW method.

However, Guadino et al. 2013, reported a comprehensive review on potential application of MW heating to accelerate heterogeneous reactions under gaseous reagent i.e. CO and CO_2, which gave a new insight to various scientist to explore the MW heating method. Since the poisonous nature of CO and high cost of CO_2 storage restricted various resercher to use as heterogeneous medium, so the concept of dielectric heating can be further explored viaMW heating protocol [56]. Recently Darekara et al. 2020, synthesized a series of derivatives of thiadiazoles and triazoles under microwave conditions. They also explored their antibacterial activities [57].

5. Synthesis of nano particles

As Nanoscience and nanotechnology are new frontiers of this century in all facet of human life and comfort. Nanoparticles are important components in a wide type of applications, including medicine, semiconductors, catalysis, and energy. Microwave energy has the potential to selectively heat either the solvent or the precursor molecules for nanomaterial preparation. In this process of heating rapid temperature rise allows nanomaterial synthesis to take place in a homogeneously mixed precursor solution To produce nano materials or particles by using microwave technology is a need of hour in context of green chemistry also. The silver nanoparticle has an importance place in biomedical and catalytical field. Its easy synthesis is a hot topic of research. The template MW Synthesis of silver nanoparticles were carried out [58] from silver nitrate and starch solution. They performed the synthesis under direct heating, controlled heating, and microwave irradiation. The Starch acted as a reductant as well as a capping material to protect the nanoparticles surfaces and prevents the particles from aggregation.

Additionally, the MW heating technique is also useful for the preparation of superior nanomaterials from direct heating of the various molecular precursors. The microwave-assisted process involve reduction of metal salts either in organic solvents or in aqueous medium in presence or absence of surface-directing agents in one-pot synthesis [59].

As Ionic liquids, due to their high ionic conductivity and polarizability, were an excellent microwave-absorbing agent, have also been used for the synthesis of metal nanostructures. By changing the various parameters i.e. precursor, precursor concentration, solvent, microwave power, pressure and temperature different metallic nanostructures in numerous sizes and morphologies have been prepared. Many simple and complex metal oxides have been also synthesized by using the microwave-assisted route. The microwave hydrothermal method has been used to prepare a large variety of binary and ternary oxides such as:

Sl No	Types of nano particles	References
1	CuO	Zhao Y, et al. [60]
2	ZnO	Huang J et al. [61]
3	PdO	Wang et al. [62]
4	In2O3, Tl2O3	Patra CR et al. [63]
5	SnO2	Jouhannaud J et al. [64]
6	HfO2	Eliziário SA et al. [65]
7	BiVO4	Zhang HM et al. [66]
8	ZnAl2O4	Zawadzki M et al. [67]
9	BaTiO3	Nyutu EK et al. [68]
10	CaTiO3	Moreira ML et al. [69]

Motshekga et al. reported [70] a comprehensive review on the synthesis and applications of carbon-nanotube-coated metal/oxides nanoparticles under the microwave-assisted method. They explained various studies in which the microwave-assisted synthesis method of the composites formation will be completed in a shorter reaction time with uniform and well-dispersed nanoparticle.

A simple and microwave irradiation method for the biosynthesis of silver nanoparticles (SNPs) using aqueous leaf extract of *Origanum majorana* and *Citrus sinensis* as a novel bio source of cost-effective, non-hazardous reducing, and stabilizing agents was reported [71]. The microwave heating made the synthesis of SNPs fast, uniform, and reproducible. They also exhibited good antibacterial activity against *E. coli* and *B. subtilis* pathogens.

Similarly five plant extracts of *Syzygium aromaticum, Origanum vulgare, Origanum majorana, Theobroma cacao* and *Cichorium intybus* were used [72] for microwave assisted synthesis of alumina nanoparticles from Aluminum nitrate.

On analysis of XRD pattern of particles synthesized with *S. aromaticum* showed semi-crystalline structure however others showed nano dimension of particles or amorphous structure nanoparticles.

The microwave-assisted sol–gel synthesis of high-quality and uniform ZnFe2O4 nanocrystals were performed by Suchomski et al. [73]. They described these novel nano particles. Additionally, the synthesis of magnetite and maghemite nanoparticles with well-controlled size, high crystallinity and good magnetic properties, in less reaction time was developed under microwave irradiation method [74]. It was observed that microwave power and heating time are the main parameters controlling the size of the nanoparticles and the presence of maghemite, while ammonia concentration does not strongly affect it.

The magnetic metallic nickel nanoparticles were synthesized by reacting simple nickel chloride as precursor, ethylene glycol as solvent and ethanol used as reducing agent following microwave-assisted method using a monomode microwave reactor by Zuliani et al. [75]. They achieved metallic nickel in five minutes at 250°C under MW conditions, which showed high catalytic activity for the hydrogenolysis of benzyl phenyl ether.

Further, fabrication of small anatase titanium dioxide (TiO2) nanoparticles (NPs) attached to larger anisotropic gold (Au) morphologies by a very fast and simple two-step microwave-assisted synthesis was performed by May-Masnou et al. [76]. They also evaluated the photocatalytic activity of the two Au/TiO2 NPs, in the photoproduction of hydrogen from gaseous water/ethanol mixtures at ambient temperature and pressure.

The amazing utilization of microwave energy and nanotechnology to improve the printability and performance of cotton prints via screen printing technique was reported [77]. They pre-treated each cotton sample separately by microwave power in the range of 300 to 700 watts for a period of 1 to 9 min. Then the optimum sample was printed by printing paste comprising a reactive dye Remazol™ and silver nanoparticles (Ag-NPs) with diverse concentrations. Further, the printed samples were fixed using MW energy lateron subjected to steaming or thermo-fixation. On the analysis of results indicated that, the prints obtained using microwave and Ag-NPs were found to have better colour strength, fastness properties, antibacterial behaviour and surface morphology as compared to the conventional techniques.

Though the conventional wet chemical synthetic method is still the most widely used method for nanoparticle synthesis due to its simplicity, inexpensive processing, and scalable nature. But nanoparticles produced by this route tend to agglomerate after some time, in particular at high temperature and pressure, resulting to a gradual loss of activity. To tackle these challenges a facile and scalable thermal shock synthesis method based on microwave irradiation for the rapid synthesis of nanoparticles on a reduced graphene oxide (RGO) substrate was demonstrated by Xu et al. 2019 very recently [78]. By loading precursors onto RGO, with medium amount of defects can efficiently absorb microwaves leading to rapid temperature change resulting uniform nanoparticles synthesis. This technique is also known as defect engineering. The beauty of this technique is scalable i.e. has potential in large-scale production nanoparticles.

6. Conclusion

In last couple of decades, the application of Microwave enegy has emerged a promising tool for synthesis of novel molecule in the area of drug research, in synthesis of nano material, in catalyst field, in inorganic synthesis, in CO and CO2 capture reactions, paint industry, in extraction of products from natural sources etc. It blossemed as a reliable alternavite of conventional heating due to high speed or shortening of reaction time; simplicity i.e.reaction can be performed in sealed and open glass vials with wide range of options; with high productivity i.e. almost 200–400% more than conventional means. The MW assisted reactions will be a one of the best method to synthesise diverse type of molecules of interest.

Conflict of interest

None.

Author details

Anjali Jha
Department of Chemistry, GIS, GITAM (deemed to be University) Rushikonda, Visakhapatnam, India

*Address all correspondence to: ajhamani@gitam.edu

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