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(SHORT COMMUNICATION)

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Heating using microwave technique: An approach towards green chemistry

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Abstract

Green chemistry focuses on minimizing waste, minimizing use of chemicals/reagents, reducing pollution and preservation of human health and environment. Using conventional methods of synthesis are creating the said problems in present scenario. To solve these problems new alternate methods must be developed. Many alternatives have been already developed but many more have to do. Use of microwave radiations are one the alternative in which these radiations are used for heating purpose instead of conventional heating methods which uses solvents in large amount and requires much time. Lot of benefits are there in heating using microwave radiations over conventional methods of heating but have limitations too. Not for every reaction/synthesis, but microwaves can be only used for the reactant having specific properties. Single mode and multi-mode microwave ovens are usually used for achieving this purpose. Heating by microwave radiations not only saves time i.e. it converts hours to minutes because it accelerates the reaction, increases the yield, reduces the energy requirement and the most important thing is no or minimum pollution/waste. Health hazards are also been noticed in using these radiations both external and internal. This short communication was aimed to find out the necessity of alternative methods of synthesis that fulfils the concept of green chemistry which will fulfill the need of healthy life and environment.

Keywords: Microwaves; Microwave radiations; Green chemistry; Electromagnetic radiations; Microwave assisted synthesis; Conventional and microwave synthesis

1 Introduction

1.1 Green chemistry: significance, benefits & principles

During the last few centuries, chemistry has become very important in our daily life. From several decades, chemistry is immensely improving the quality of life but also the negative effects of some chemicals affecting both human health and environment. In today's world one of the biggest problems is environmental pollution which includes water, air and soil pollution. Chemicals from factories, toxic gases expelled from factory chimneys, waste water from home, mixed organic solvents.etc are the major cause responsible for environmental pollution. Green chemistry is the most important and the only alternative way to achieve long-term improvement because population is continuously growing and natural resources are limited. Green chemistry represents two main components. First component addresses the problem of efficient raw material utilization and the efficient elimination of waste. Second component deals with the manufacture, use and disposal or reuse of chemicals which directly or indirectly influence health, safety and environment. Green chemistry provides a new approach of synthesis, processing and application of chemical substances that result in reducing threats to health and environment.

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1.2 Twelve principles of Green chemistry

These principles are proposed by Anastas and Warner.

- It is better to prevent waste than to treat or clean up waste after it has formed.
- To maximize the incorporation of all materials used in the process into the final product, such synthetic methods should be designed.
- Practically, synthetic methodologies should be developed to use and generate substances that possess no or minimum toxicity to human health and the environment.
- To reduce the toxicity various chemical products should be designed.
- The use of solvents should be made unnecessary wherever possible i.e. minimum use.
- Use of low temperature and pressure in synthesis is the good method to reduce energy requirements.
- Use of renewable raw materials or feedstock.
- Wherever possible, derivatization (blocking group, protection/deprotection) should be avoided.
- Uses of catalytic reagents are better than stoichiometric reagents (as selective as possible).
- Chemical products should be designed in such a way that after the end of their function they do not continue in the environment and break into degradation products.
- To control the formation of hazardous substances, analytical methodologies should be developed.
- Chemical accidents like releases, explosion, fire etc. can be minimized by using avoiding substance that are inflammable or explosive in chemical processing.

Green Chemistry says to minimize waste formation. Preservation of human health and environment is ensured by new approach that is green chemistry. Energy conservation and consumption has long been known to produce a major environmental effect. Solid state isatechnique like Microwave irradiations are being utilized to affect chemical transformations rapidly, in contrast to those that have classically been conducted in liquid solutions. Solvent-free microwave assisted reactions provide an opportunity to work with open vessels and thus, avoiding the risk of high pressure and increasing the potential for scale up of such reactions. The practical feasibility of microwave assisted solvent free synthesis has been illustrated in various useful transformations and in the synthesis of heterocyclic systems.

Green chemistry is a multidisciplinary research area that involves knowledge from various disciplines like chemistry, chemical technology, biotechnology, molecular biology, ecology, toxicology. The inputs from the various disciplines can help in developing new and innovative technologies and green synthetic routes for chemical production. The greenness of a chemical synthesis can be evaluated quantitatively and qualitatively. This can be easily understood by an example, Perchloroethylene, $Cl_2C=CCl_2$ is commonly being used as a solvent for dry cleaning. It is now known that Perchloroethylene contaminates ground water and is a suspected carcinogen. A technology known as Micell Technology developed by Joseph De Simons, Timothy Romark, and James McClain, make use of liquid CO_2 and a surfactant for dry cleaning clothes, thereby replacing Perchloroethylene. Dry cleaning machines have now been developed using this technique [1].

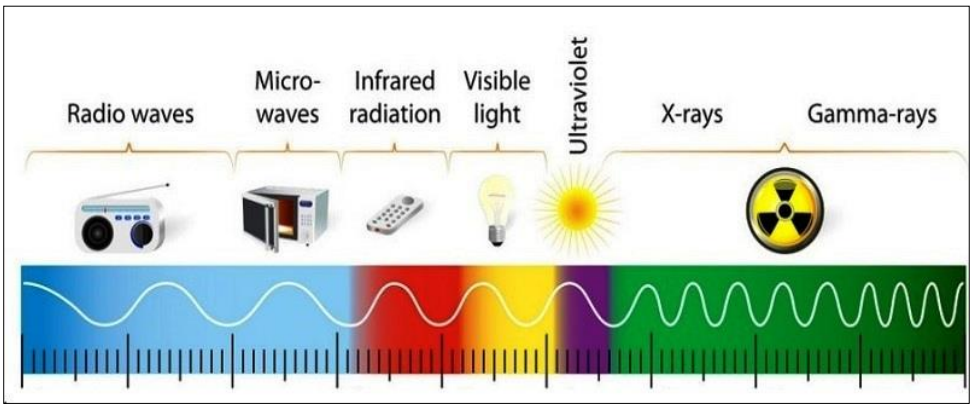
1.3 Microwave radiations or microwaves

Microwaves are electromagnetic radiations whose frequency ranges from 300 MHz to 300 GHz and in terms of wavelength, it ranges from 1 mm to around 30 cm [2]. In electromagnetic spectrum, microwaves fall between infrared radiation and radio waves as shown in Fig. 1.

1.4 Properties of microwaves

- These radiations are capable of radiating electromagnetic energy with shorter wavelengths.
- These radiations are reflected by metal surfaces.
- Refraction, diffraction, reflection and interference are the phenomena which affects the transmission if microwaves.
- Microwaves can easily pass through glass and plastics that's why these are used in heating and cooking in an oven.
- They are easily weakened within shorter distances.
- Microwaves are not reflected by the Ionosphere.

- Microwave radiations can go across the atmosphere. Hence, microwaves are utilized in the satellite communication sector.
- Microwaves propagate in a straight line and are reflected by the conducting surfaces [3].



(Courtesy: https://amhsnewspaper.com/42656/science/cuba-targets-u-s-embassy-with-microwave-radiation/)

Figure 1 Microwaves in electromagnetic spectrum

2 Mechanism of microwave heating

All the materials are not liable to microwave heating as response of many materials to microwave radiation is multiple. Depending on their response to microwaves, materials can be divided as follows:

- Materials transparent to microwaves, example sulphur
- Materials reflect microwaves, example copper
- Materials that absorb microwaves, example water

Table 1 Comparison between the conventional and microwave assisted techniques of heating

Conventional synthesis	**Microwave assisted synthesis**
Conventional synthesis typically involves the use of a furnace or oil bath that heats the walls of the reactors by convection or conduction (Figure 2).	Microwave penetrates inside the material and heat is caused through direct microwave material interaction (Figure 2).
The centre of the sample takes much longer to attain the target temperature.	Microwave permits for an increase in reaction rate, speedy reaction optimization, and speedy analogue synthesis.
This is a slow and incompetent method for transferring energy into the reacting system.	Less energy and solvent is used and it allows difficult compound synthesis.

Table 2 Comparison of time taken in reaction using microwave versus conventional heating in respect of time taking and energy efficiency

Compound synthesized	**Microwave Reaction time**	**Conventional Reaction time**
Methyl benzoate	05 minutes	08 hours
4-nitrobenzylester	02 minutes	90 minutes
Zeolitesynthesis	30 seconds	60 minutes
Cubanite	03 minutes	03 days
Sodium aluminium hydride	120 minutes	08 hours
4-nitrobnzyl ester	02 minutes	90 minutes

Microwave absorbing materials are of highest importance and there are three main mechanisms of heating specifically: Dipolar polarization, Conduction mechanism and Interfacial polarization. Table 1 **S**ome reactions that compares techniques of microwave heating and conventional heating

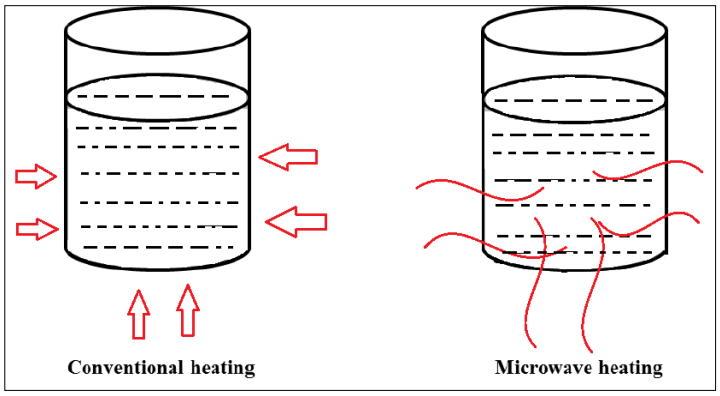


Figure 2 Difference in heating by conventional method and microwave method

2.1 Microwave synthesis apparatus

The apparatus for microwave assisted synthesis comprises single-mode microwave ovens and multi-mode microwave ovens.

2.2 Single-mode microwave apparatus:

The differentiating characteristic of a single-mode apparatus is its potential to create a standing wave pattern. These interfaces give rise to an array of nodes where microwave energy intensity is zero and an array of antinodes in which the magnitude of microwave energy is at its highest. One of the restrictions of single-mode apparatus is that only single vessel can be irradiated once.

2.3 Multi-mode microwave apparatus:

A key feature of a multi-mode apparatus is the intentional avoidance of generating a standing wave pattern inside it. The purpose is to generate as much disorganization as possibleinside the apparatus. The greater the disorganization, the higher is the dispersion of radiation, which enhances the area that cancause effective heating inside the apparatus. Proportionately, a multi-mode microwave heating apparatus can assist a number of samples simultaneously for heating.

3 Benefits of microwave assisted synthesis

As catalysts accelerates the rate of chemical reaction, same like that microwaves can accelerate the rate of reaction, provide better yields and higher purity, uniform and selective heating with lower energy usage, perform greater reproducibility of reactions and help in developing advantageous and cleaner synthetic path. Some advantages of microwave assisted organic synthesis are given below:

3.1 Speedy reaction

The reactions are finished in few minutes instead taking hours in completing reaction e.g. synthesis of fluorescein takes about 10 hours by conventional heating methods which can be achieved in only 35 minutes by using microwave heating.

3.2 Greater yield and elevated purity

Less formation of side products are noticed using microwave irradiation and theproduct is recovered in greater yield e.g. microwave synthesis of aspirin catalyzes the yield of the reaction from 85 % to 97%.

3.3 Energy saving

Heating by means of microwave radiation saves energy as microwaves heat up just the sample and not the apparatus and for that reason energy consumption is less.

3.4 Uniform and selective heating

In conventional heating, there is always a temperature difference between the wallsand the solvent because the walls of the oil bath gets heated first and then the solvent, in addition there is uneven heat distribution too. On the other hand in microwave heating, only the solvent and the solute molecules are excited by heat, which results inconsistent heating of the solvent. Selective heating is based on the principle that different materials reply differently to microwaves. Some materials are transparent though others absorb microwaves.

3.5 Green synthesis

Reactions conducted using microwaves does not produce any pollution, hence eco-friendly. Amount of solvents can be minimized in the reaction because microwaves heat the compounds directly. Synthesis without solvent i.e. reagents are absorbed on mineral support, is potentially nice eco-friendly green technique of synthesis. In addition, use of microwaves facilitates reduction in amount of solvents required for purification of end products which involves toxic-reagents.

3.6 Reproducibility

In microwave heating, the temperature of chemical reactions can be easily monitored. Microwave assisted reactions are more reproducible because of uniform heating and better control of process parameters.

4 Limitations of microwave synthesis

Microwaves are good source of heating but have limitations in application for materials that absorb them e.g. sulphur. It has been observed that improper use of microwave heating for enhancement of rate of chemical reactions may give dangerous end results, for example, microwave irradiation may damage the polymer vessel used for heating. Conducting microwave reactions at high pressure may also result in uncontrolled reactionsand cause explosions. Health hazards are also observed due to penetration of microwaves, low frequency ranges are able to penetrate the human skin while higher frequency-range can reach up to body organs. Research has proven that complete degeneration of body tissues and cells may result on prolonged exposure of microwaves. It has also been established that constant contact of DNA with high frequency microwaves during a biochemical reaction mayresult incomplete degeneration of the DNA strand [4].

5 Practical examples

5.1 Microwave stimulated synthesis of ethyl benzene (Wolfkishner reduction)

10 ml acetophenone, 30 ml diethylene glycol, 9 ml hydrazine hydrate and potassium hydroxide pellets were kept under microwave irradiation for 5 minutes at 110°C temperature and 160 watt power, results in ethyl benzene. The reaction mixture which consisted of upper and lower layer was transferred to a separating funnel and lower aqueous layer was taken in another container. The aqueous layer was then extracted with 10 ml ether. Excess ether was removed by heating on a water bath. The resultant mixture was dried with anhydrous magnesium sulphate and a yellowish solution with gasoline like smell was obtained. Yield was 74%.

5.2 Microwave stimulated synthesis of azlactone from hippuric acid

In a round bottom flask, benzaldehyde (10.3 mol) and hippuric acid (10.3 mol) were taken and mixed. Then mixture of acetic anhydride (10.3 mol) and potash alum (10 mol %) was poured in above round bottom flask. The whole content was irradiated under microwave irradiation at ambient temperature for 8 minutes. After completion of time the yellow solid obtained which got precipitated on addition of 5 ml of 95% ethyl alcohol. The whole solid product was then filtered and washed with hot water. Whole mixture was recrystallized with acetone/water to obtained azlactone as final product. Yield was 96% [5].

6 Conclusion

Although there are lot of benefits of microwave assisted synthesis like speedy reaction, greater yield and elevated purity, energy saving, uniform and selective heating, green synthesis and Reproducibility but it have limitations and health hazards too. Microwave assisted synthesis is a revolution in field of green chemistry because of no or minimum pollution i.e. soil, air and water which is one of major problem all over the world. Alternative methods of synthesis is highly required in today's scenario to reduce pollution and microwave assisted synthesis is one of the potent way to

achieve the aim. More researches are required that follows the principles of green chemistry i.e. eco-friendly, not only to synthesize more products but also to reduce the limitations and health hazards.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that there is no conflict of interest.

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