An Overview of Microwave Assisted Technique: Green Synthesis

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An Overview of Microwave Assisted Technique: Green Synthesis

Author(s): Jain A K, Singla R K

Abstract

Green chemistry efficiently utilizes (preferably renewable) raw materials, eliminates waste, and avoids the use of toxic or hazardous reagents and solvents in the manufacture and application of chemical products.

Microwave assisted technique opens up new opportunities to the synthetic chemist in the form of new reactions that are not possible using conventional heating and serve a flexible platform for chemical reaction viz. Aldol condensation, Knoevenagel condensation, Beckmann rearrangement, Vilsmeier reaction, Benzil - Benzilic acid rearrangement, Thia – Fries rearrangement, Fischer cyclization, Vilsmeier-Haack reaction, Mannich reaction, Claisen – Schmidt condensation, Gould-Jacob reaction etc.

1. Introduction

Microwaves are defined as electromagnetic waves with vacuum wavelengths ranging use of between 0.1 to 100cm or, equivalently, with frequencies between 0.3 - 300GHz. Microwave dielectric heating uses the ability of some liquids and solids to transform electromagnetic radiation into heat to drive chemical reactions. This technology opens up new opportunities to the synthetic chemist in the form of new reactions that are not possible using conventional heating. The interest in the microwave assisted organic synthesis has been growing during the recent years1-2.

With the easy availability of microwaves its use in chemistry has gained momentum and this has led the microwave heating to emerge as powerful technique to promote a wide variety of chemical reactions.

1.1 Green Chemistry: Benign by Design

In the mid - 1990s Anastas and coworkers4 at the United States Environmental Protection Agency (EPA) were developing the concept of benign by design, that is designing environmentally benign products and processes to address the environmental issues of both chemical products and the processes by which they are produced. This incorporated the concepts of atom economy and E factors and eventually became a guiding principle of Green Chemistry as embodied in the 12 Principles of Green Chemistry5, the essence of which can be reduced to the useful working definition: “Green chemistry efficiently utilizes (preferably renewable) raw materials, eliminates waste, and avoids the use of toxic and/or hazardous reagents and solvents in the manufacture and application of chemical products”.

Raw materials include, in principle, the source of energy, as this also leads to waste generation in the form of carbon dioxide. Green Chemistry is primary pollution prevention rather than waste remediation (end - of - pipe solutions). More recently, the twelve Principles of Green Engineering were proposed6, which contain the same underlying features – conservation of energy and other raw materials and elimination of waste and hazardous materials – but from an engineering standpoint. Poliakov and coworkers7 proposed a mnemonic, productively, which captures the spirit of the twelve Principles of Green Chemistry in a single slide.

1.2 Principle involved in microwave assisted technique

Following twelve basic principles of green chemistry have been formulated by Paul T. Anastas8.

1. It is better to prevent waste than to treat or clean up after it is formed.
2. Synthetic processes, materials 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 the environment.
4. Chemical products should be designed to preserve efficacy while reducing toxicity.
5. The use of auxiliary substances should be made unnecessary whenever possible and, when used it should be innocuous.
6. Energy requirement should be recognized for their environmental and economic impacts and should be minimized.
7. A raw material or feedstock should be renewable rather than depleting, it should be technically and economically practicable.
8. Unnecessary derivatization should be avoided whenever possible.
9. Catalytic reagents are superior to stoichiometric reagents.
10. Chemical products should be so designed 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. Substance should be chosen so as to minimize the potential for chemical accidents, including explosions and fires.

1.3 Microwave assisted technique- A new tool for Green chemistry

Although first reported by the group of Gedye8 and Gigure Majetich9 in 1986, the use of microwaves in organic synthesis was initially hampered by a lack of understanding of the basic principle of MW dielectric heating and the inability to obtain reproducible results with domestic microwave ovens. Today microwave irradiation in organic synthesis has readily gained popularity as it accelerates variety of organic reactions10. Solvent free organic reactions (dry media techniques) under microwave irradiation are one of the main concerns of research in recent time11-13 which was originally developed in late eighties14. Synthesis without solvent, in which reagents are absorbed on mineral support, has a great potential as it offers an eco-friendly green protocol in synthesis.

2. Comparison of Microwave Versus COnventional Heating 15-16

Microwave dielectric heating is a non quantum mechanical effect and it leads to volumetric heating of the samples. Therefore, it is necessary to question whether it has any significant advantages compared to thermal heating of chemical reactants. There are significant differences in thermal interaction and these may confer advantages for dielectric heating; however, the effects require a greater understanding of the temperature profile and the nature of the interaction.

1. The introduction of microwave energy into a chemical reaction that has at least one component, which is capable of coupling with the microwaves can lead to much higher heating rates than those that can be achieved conventionally. Using very cheap and readily available microwave cavities, heating rates of 2–4°Cs−1 may be readily achieved even for common organic solvents. Such heating rates are more difficult to achieve using conventional heating, although of course dropping sealed tubes into heated sand furnaces at >1000°C could result in comparable heating rates.

2. The microwave energy is introduced into the reactor remotely and therefore there is no direct contact between the energy source and the sample undergoing heating. This combined with first point may lead to very different temperature–time profiles for the reaction and as a consequence may lead to an alternative distribution of chemical products in the reaction. Microwave dielectric heating resembles a flash heating process, whereby the energy is generated much more rapidly and the sample cools more rapidly at the end of the reaction. The different profiles may therefore lead to significantly different products, particularly if the reaction product distribution is controlled by complex and temperature-dependent kinetic profiles.

3. Chemicals and the containment materials for chemical reaction do not interact equally with the commonly used microwave frequencies for dielectric heating and consequently selective heating may be achieved. Specifically, it is possible to cool the outside of the vessel with a coolant that is transparent to microwaves (solid CO2 or liquid N2) and thereby have cold walls that still allow the microwave energy to penetrate and heat the reactants, which are microwave active, in the vessel. Also for solid-state reactions contamination from the crucible walls may be minimized.

4. The degree of selective heating should not be overdone for solvent mixtures. For example, if a mixture of MeOH (2%), which has a high loss factor, and benzene (98%), which is transparent to microwaves, is exposed to a microwave field, the whole mixture heats up very rapidly. The microwave process involves translation and rotation, and although the effect may have its origins in the vicinity of methanol molecules the rate of energy transport is so fast that benzene molecules are also heated rapidly. Therefore, it is not possible to store the microwave energy selectively either within parts of a molecule or in active molecules in two component mixtures.

5. The boiling phenomenon is a kinetic as well as a thermodynamic process and therefore solvents heated under microwave conditions often boil at elevated temperatures even though they remain contained under 1atm pressure. The precise elevation of this nucleation-limited boiling point depends on the power input, the occurrence of effective stirring and the limitation of the number of nucleation sites. In solid samples, the rate of energy transport is less and consequently the development of hot spots is more significant. A careful analysis of heterogeneous catalysis suggests that hot spot formation around the
catalyst not only enhances the reaction rate but may also contribute to shifts in the equilibrium constant. In a microwave cavity a standing wave pattern is generated, which depends of course on a multiple of the wavelength of the radiation (12.5cm at 2.45GHz), and therefore depending on the dielectric properties and size of the sample one can get considerable variations in temperature. Therefore, for large samples it is possible to develop quite large temperature gradients and it is necessary to introduce electronic and mechanical perturbations to even out the field pattern.

To maximize the difference between microwave and conventional heating, one generally needs to use relatively high power levels, develop a temperature profile that ramps up and cools off more quickly than the conventionally one and work under conditions where higher pressures may develop in the reaction vessel. It is, therefore, not surprising that there have been a number of suggestions that account for the differences between conventional and microwave heating in terms of specific microwave effects.

3. A Flexible Platform for chemical reactions

3.1 The Microwave Oven

A microwave oven consists of three parts(Illustration 1) ·
1. A high power source
2. A waveguide feed
3. The oven cavity.

1. A high power source - A magnetron is a source to generate microwaves. Magnetron is a thermo ionic diode having an anode and a directly heated cathode. As the cathode is heated, electrons are released and are attracted towards the anode which is made up of an even number of small cavities and the gap across the end of each cavity behave as a capacitance. A strong magnetic field is induced axially which bends the path of electrons as they travel from the cathode to the anode.

As the deflected electrons pass through the cavity gaps, they induce a small charge in to the circuit, resulting in the oscillation of the cavity till high amplitude has achieved of 1200W of electric line power used by magnetron, around 600 W is converted into electromagnetic energy. The remainder is converted into heat that may be dissipated through air or water cooling. The variable power available in domestic oven is produced by switching the magnetron on- and- off according to a duty cycle.

2. A waveguide feed - It is a rectangular channel made of sheet metal having reflective walls which allows the transmission of microwaves from magnetron to microwave cavity. These walls prevent leakage of radiations and increase the efficiency of the oven.

3. The oven cavity - Some area of oven cavity receives large amount of energy in the form of electric energy and in some it is neglected. For smoothing the incoming energy in the cavity, a stirrer is sometimes used.

3.2 Reaction vessel and reaction medium

3.2.1 Reaction vessel

The preferred reaction vessel for microwave induced organic reactions (MORE), is a tall beaker, loosely covered with a capacity much greater than the volume of the reaction mixture. Teflon and polystyrene vessels can be used as these are transparent to microwaves. No metallic container can be used as it gets heated soon due to preferential absorption and reflection of rays.

3.2.2 Reaction medium (in presence of solvent)

In MORE, the reactions are carried out in a solvent medium or on the solid support (in which no solvent is used). For a reaction in solvent medium, the solvent must have a dipole moment so as to absorb microwaves and a boiling point at least 20-30 higher than the reaction temperature. In this respect N,N-dimethylformamide (DMF), is an excellent solvent used in domestic microwave oven with high b.p. (160°C) and high dielectric constant ($\epsilon = 36.7$) other solvents used are: formamide (b.p. 216°C, $\epsilon = 111$), methanol (b.p. 65°C, $\epsilon = 32.7$ ), 1,2-dichlorobenzoic (b.p. 83°C), dioxane (b.p.101°C). The presence of salt in polar solvents can frequently enhance, microwave coupling. Hydrocarbon solvents such as hexane ($\epsilon = 1.9$), toluene ($\epsilon = 1.9$), because of less dipole moment are unsuitable as they absorb microwave radiation poorly but addition of small amounts of alcohol or water can lead to dramatic coupling effect.

3.2.3 Reactions without solvent (over solid supports)

Recent years have witnessed the importance of microwaves in mediating organic reactions because of the advantages with respect to classical organic chemistry in terms of shorter reaction times, minimum waste, generally higher yields, possibility of carrying the reactions in the absence of solvents and in safe conditions. In view of this, more interest has now been focused on dry media synthesis, involving
the coupling of MWI with solid supported reagents. Solid support method was initially described by Keinan and Mazur. Solvent free techniques represent a clean economical, efficient and safe procedure which can lead to substantial saving in time, money and products. The expeditious and solvent-free approach involves the exposure of neat reactants to microwave irradiation in conjugation with the use of supported reagents or catalysts, which are primarily of mineral origin. Mineral oxides are poor conductor of heat but they absorb microwave radiation very effectively with significant improvement in temperature, homogeneity and heating rates which enables faster reactions and less degradation of final products. Depending on the type of organic reactions aluminas, silicas, clays, zeolites or envirocates EPZG and EPZ 10 are selected as acidic or basic supports.\(^{21, 22}\)

Alumina alone can act as base support towards a rather acidic molecule, but if a strong base is necessary, KF on alumina can be used and ionize carbon acids up to pKa@35\(^{23}\) montomorillonites (clays) such as K-10 and KSF offers acidities very close to nitric or sulphuric acids. Envirocates EPZ1O and EPZ210 are solid supported catalysts which have Bronsted and Lewis acid characteristics where as EPIC is solid supported strong Bronsted acid. Some of the supported reagents, namely clay supported Iron (III) nitrate (clayfen) and copper (II) nitrate (claycop) are also useful and prepared according to literature procedure\(^{24}\). The recyclability of various solid supports from the reaction medium is the most interesting feature as it renders these processes truly eco-friendly, reduce the pollution at source and make the process to fall in the domain of important green chemical theme. Due to less time requirement, ease of workability, dramatic rate enhancement and increased selectivity, microwave technology provides a promising alternative to environmentally unacceptable conventional procedures.

### 3.3 Some important name reactions

Following name reactions have been successfully carried out under MW conditions.

1. Aldol condensation\(^{25}\) (Illustration 2)
2. Knoevenagel condensation\(^{26, 27}\) (Illustration 3)
3. Beckmann rearrangement\(^{28, 29}\) (Illustration 4)
4. Vilsmeier reaction\(^{30}\) (Illustration 5)
5. Benzil - Benzilic acid rearrangement (Illustration 6)
6. Thia – Fries rearrangement\(^{31}\) (Illustration 7)
7. Fischer cyclization\(^{32}\) (Illustration 8)
8. Vilsmeier-Haack reaction\(^{33}\) (Illustration 9)
9. Mannich reaction\(^{34}\) (Illustration 10)
10. Claisen – Schmidt condensation\(^{35}\) (Illustration 11)
11. Gould-Jacob reaction\(^{36}\) (Illustration 12)

Other than above, following reactions have also been carried out in microwave:
1. Diels-Alder reaction\(^{37, 38}\)
2. Bischler Napieralski reaction\(^{39}\)
3. Cannizzaro’s reaction\(^{40, 41}\)
4. Prins reaction\(^{42}\)
5. Michael addition\(^{43, 44}\)
6. Ene reaction\(^{45}\)
7. Niementowski reaction\(^{46}\)
8. Wittig reaction\(^{47}\)
9. Willgerodt-Kindler reaction\(^{48}\)
10. Fries rearrangement\(^{49}\)
11. Claisen rearrangement\(^{50}\)
12. Doebner condensation\(^{51}\)

### 4. Conclusion and Perspectives

Microwave assisted green synthesis is a very good technique in the field of green chemistry by governs a flexible platform for many named organic reaction. The compiled review gives an idea on MW green chemistry, their principle, their assembly and enlist named organic reaction which is beneficial for research work.

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Illustrations

Illustration 1

Illustration 1 The Microwave Oven

Illustration 2

Illustration 2 – Aldol Condensation
Illustration 3

Illustration 4
Illustration 5

Illustration 6
Illustration 7

Illustration 7

Illustration 8

Illustration 8
Illustration 9

Illustration 10
Illustration 11

Illustration 12
Illustration 13

Table: 1 R&D Philosophy in Harmony with the Principles of Green Chemistry

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<td>Atom Economy</td>
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<td>Solvent Reduction</td>
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<td>Reagent Optimization</td>
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<td>Safety</td>
<td>Non-hazardous materials reduce risk of exposure, release, explosions and Fires</td>
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Reviews

Review 1

Review Title: Self Evaluation of Article

Posted by Mr. Rajeev K Singla on 16 Dec 2011 05:15:22 PM GMT

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Rating: 8

Comment:
This work is meant for those scientists who work on green chemistry and believes that environment should not get disturbed by our research.

Competing interests: No, I don't have any competing interest

Invited by the author to make a review on this article? : No

Experience and credentials in the specific area of science:
I am experienced

Publications in the same or a related area of science: No

How to cite: Singla R. Self Evaluation of Article[Review of the article 'An Overview of Microwave Assisted Technique: Green Synthesis ' by ].WebmedCentral 1970;2(12):WMCRW001275
Review 2

Review Title: An Overview Of Microwave Assisted Technique: Green Synthesis

Posted by Mr. Amrit B Karmarkar on 15 Dec 2011 07:50:08 AM GMT

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Rating: 5

Comment:
Dear Authors, Thanks for writing this article in WMC. In comparison section, can you please include Comparison of Microwave Versus Conventional Heating in table format. Also you can add recent advances section in the paper.

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Experience and credentials in the specific area of science:
I have backup of some recent things in this field

Publications in the same or a related area of science: No

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Review 3

Review Title: An Overview of Microwave Assisted Technique: Green Synthesis

Posted by Dr. Varadaraj B G on 28 Sep 2011 04:36:04 AM GMT

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Rating: 6

Comment: Nil

Competing interests: Nil

Invited by the author to make a review on this article? : Yes

Experience and credentials in the specific area of science:
I have synthesised few molecules using MW synthesis

Publications in the same or a related area of science: No

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