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Microwave Assisted Green Synthesis

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ABSTRACT

Chemistry has brought about revolution in each field of life whether it is medical field, agricultural field or any other, its impact can be seen everywhere. But the hazardous waste by product so formed and in order to get rid of it, finally marked the beginning of Green Chemistry. The reason why Microwave assisted organic reaction in organic chemistry is getting more popular because it provide better yield, less consumption of energy and cost effective . Altogether it make greener/cleaner chemistry having feature of sustainability in it. . Microwave organic synthesis opens up new opportunities to the synthetic chemist in the form of new reaction that are not possible or if possible take more time by conventional heating and serve a flexible platform for chemical reaction. Microwave heating is playing a vital role in treatment of various kinds of waste like domestic and hazardous nuclear and industrial wastes. Microwave heating can be advantageously used for waste management in areas where human exposure can cause invariable health issues. Altogether it can be justified by saying that microwave is a convenient way towards achieving the goal of green/sustainable chemistry, and is highly recommended to use in organic preparations for various purposes including Pharmaceutical chemistry.

Keywords : Green Chemistry ,Microwave Irradiation, Synthetic Methods, Waste Management

I. INTRODUCTION

Chemistry has brought about revolution in each field of life. In the medical field, with the discovery of drugs and antibiotics, the average life expectancy has risen from 47 years in 1900 to 75 years in 1990s. The world's food supply also increased enormously due to the discovery of hybrid varieties, improved methods of farming, better seeds, and use of insecticides and fertilizers. The quality of life on earth became much better due to the discovery of dyes, plastics, cosmetics and other materials. Soon, the ill effects of chemistry also became pronounced, main among them being the pollution of land, water and atmosphere. This is caused mainly due to the effects of by-products of chemical industries, which are being discharged into the air, water and land. The hazardous waste released adds to the problem. The use of toxic reactants and reagents also made the situation worse. This marked the beginning of GREEN CHEMISTRY. Green Chemistry means environmentally benign chemical

synthesis. The synthetic schemes are designed in such a way that there is least pollution to the environment.

Microwave assisted methods has been proved to be a boon for the society. We can do wonders with its help , we can built large number of polymers from its basic unit in a lesser time when compared to the traditional methods which we follow. Lots of advancement has been made in microwave assisted irradiation reaction for developing wide variety of important organic compounds [1,2] . This technology of microwave assisted organic synthesis came into existence from the mid of 1980s and since then 1990s there has been a boom in the number of publications on this particular method (MAOS) [3-8]as lots of benefits are said to be associated with the above said process. Green Chemistry principles were actually being followed during organic synthesis using microwave technique as it helps in yield improving, reduced energy consumption and also cost effective . Thus in other words it can be said to be a sustainable process [9,10]. Microwave assisted synthesis try to use renewable resources and also attempt to reduce undesirable secondary product generation which supposed to be toxic and to reduce the emission of hazardous gases[11-13].The greatest advantage of this innovative technology of combinatorial chemistry lies in time management , while comparing time taken by traditional method , there is curtail of time by the fraction of 1/10 to 1/100 times or more depends upon a particular reaction[13, 14]. These methods are also applicable in drug discovery[16,17] as it provide simply more alternative pathway to produce variety of products[19,20] in lesser time with no or even less undesirable or toxic byproducts. This leads towards the betterment of the mankind. Hence this article tries to focus on the latest advances in this area of MAOS after providing a brief intro on the Microwave process.

Principles of microwaves activation:

As the name implies microwavesproduces very short waves. However, microwave really indicates the wavelengths in the micron region. Microwave frequencies belong to infrared and visible light region and refer to those from 1GHz to 105 GHz. Most domestic and commercial microwave ovens operate at 2.45 GHz. There areprobably two primary mechanisms for the absorption of microwave energy by a solution.

(i) Dipole rotation
(ii) Ionic conductance

In the dipole rotation mechanism molecular dipole is altogether aligned with the applied electric field. The electric field oscillates, thus forcing the dipole molecules [10] move which results in frictionally generated heat. At 2.4 GHz, the frequency of almost all laboratory microwave oven, the dipole align and randomize itself which is approximately five billion times per second. The frequency of the molecular rotation happens to be, similar to the frequency of microwave radiation, thus consequently the molecule continuously attempts to realign itself with theso called changing field and the energy is absorbed. Whereas in the ionic conduction mechanism, the ion species migrate in one direction or the other depends on the polarity of the electromagnetic field. Here heating is the natural consequence of the accelerated ions which meet resistance to their flow.

The above mentioned two mechanisms are responsible to heat the solutions much faster as compared to conduction and convection. Here the heating is so fast that in open vessels vaporization alone are sufficient todissipate the excess energy so produced. This results in “super heating” of solutions above their normal existing boiling points as much as 5 °C for water to 20 °C for acetonitrile. Microwave

reactions are specific towards the absorption of microwave (MW) energy by polar molecules, where as non-polar molecules being inert to microwave dielectric loss. The initial experiments revolve around the use of solvents having high dielectricconstant such as water, methanol, N-dimethylformamide (DMF), ethyl acetate, dichloromethane, acetic acid etc. As it is evident from the factlarger the dielectric constant, greater the coupling with microwaves. Hence non polar solvents such as hexane, toluene, diethyl ether and carbon tetrachloride do notshow any coupling and (therefore do not heat with microwave irradiation although it is eventually possible to use mixtures comprising microwave active/inactive solvents.

It is not compulsory to use solvents for heat transportation .In microwave chemistry we can even perform experiments under solvent less conditions, which provide an effective alternative pathway and also comprises of techniques which can be reported as environmentally friendly . This has a great advantage as it avoids toxic residues (organic solvent and mineral acids) generation and consequently enhances yield without taking environment at risk . This emerging environmentally benign technique (MAOS) belongs to the upcoming area of green chemistry.

Principle of conventional heating

Here in this method of traditional heating, reactants are slowly activated by a conventional external heat source. Heat supposed to enter into the substance, but before that it enterthe walls of the container in order to approach the solvent and the reactants. It takes longer time andalso inefficientway for transferring energy into the reacting system. One more drawback Is that most of the heat also dissipated into the surrounding without affecting solvent and reactants.

CONVERTING SOME LAB EXPERIMENTS INTO GREENER EXPERIMENTS

Identification of organic compound is a major experiment of Organic Chemistry in B.Sc.III .One step in compound identification is preparation of its derivative. In addition to the normal methods, some derivatives will also be prepared by Green Chemistry approach.

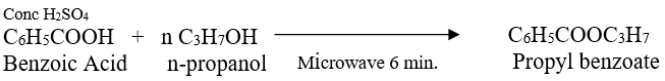
The main emphasis will be on Microwave Induced Green Synthesis. Microwaves may be considered as a more efficient source of heating, since the energy is directly imparted to the reaction medium rather than through the walls of a reaction vessel. In fact, the rapid heating capability of the microwave leads to considerable saving in dissolution or the reaction time. The smaller volume of solvent required contributes to saving in cost and diminishes the waste disposal problem[21, 22].

An attempt will be made to perform some of the following reactions:

A. Microwave Assisted Reactions in Organic Solvents.

Esterification : Reaction of Carboxylic Acid and Alcohol

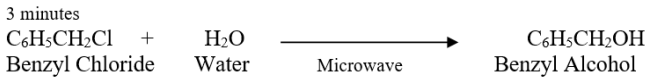
A mixture of benzoic acid and n-propanol on heating in a microwave oven for 6 minutes in presence of catalytic amount of concsulphuric acid gives [23,24,25] propylbenzoate.



B .Microwave Assisted Reactions in Water

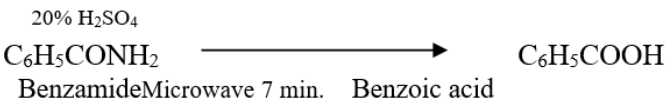
Hydrolysis of Benzyl Chloride

Hydrolysis of benzyl chloride with water in microwave oven gives 97% yield[24] of benzyl alcohol in 3 minutes. The usual hydrolysis in normal way takes about 35 minutes.



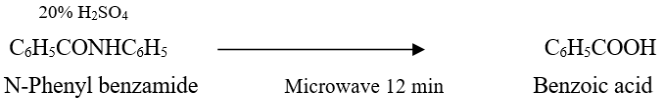
- **Hydrolysis of Benzamide**

The usual hydrolysis of benzamide takes 1 hour. However, under microwave conditions, the hydrolysis is completed in 7 minutes giving[23] 99% yield of benzoic acid.



- **Hydrolysis of N-Phenyl benzamide**

The acidhydrolysis of N-Phenyl benzamide usually takes 18-20 hours. However, under microwave conditions, the reaction is completed in 10 minutes giving[24] 74% of Benzoic Acid.

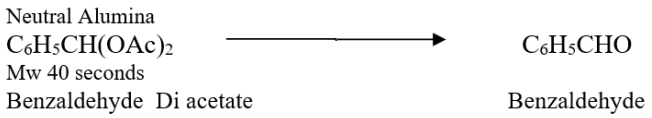


C. Microwave Solvent Free Reactions (Solid State Reactions)

Application of microwave irradiation in organic reactions has added a new dimension to solid phase synthesis. By the use of this technique, it is now possible to carry out reactions without the use of toxic or others solvents, which is one of the main problems associated with chemical synthesis. In these, the reactants are dissolved in a suitable solvent like water, alcohol, methylene chloride, etc. and the solution stirred with a suitable adsorbent or solid support like silica gel. After stirring, the solvent is removed *in vacuo* and the dried solid support on which the reactants have been absorbed are used for carrying out the reaction under microwave irradiation. An attempt will be made to perform the following reactions by solid support synthesis.

Deacetylation

Aldehydes[26], phenols[27] and alcohols[27] are protected by acetylation. After the reaction, the deacylation of the product is carried out usually under acidic or basic conditions. This process takes long time and the yields are low. Use of microwave irradiation reduces the time of deacetylation and the yields are good. One example is given as below:



Use of Microwave irradiation in waste management

Microwave heating is playing a vital role in treatment of domestic and hazardous nuclear and industrial wastes. Microwave heating can be advantageously used for waste management in areas where human exposure can cause invariable health issues for example **microwave irradiation**, a removal of E. coli bacteria in water of up to 100% was achieved in contrast to a low removal rate of 3–5% for nanotubes alone . **Microwave** technology nowadays has gained attention from researchers in a wide spectrum of applications . The major advantage of heating of microwave is that it involve no contact heating thats why the MW and high frequency technology needed for handling such type of hazardous waste is prove to be a boon .Activated carbon can be manufactured form organic wastes such as used paper, wood, waste plastic etc. in high carbonization efficiency using MW heating[28].

Advantage of Microwaves

a. Less side-products.
b. Improved yields.
c. Rapid reactions
d. High purity of products
e. Wider usable range of temperature.
f.. Simplified and improved synthetic procedure.
g. Higher energy efficiency.

Disadvantages of Microwaves

a. Heat so generated cannot be controlled

b. As microwave is a closed container. It is dangerous as it could burst.

c. Water evaporation.

II. CONCLUSION

Altogether it can be justified by saying that for achieving the goal of green/sustainable chemistry microwave assisted synthesis is an efficient alternative pathway which opens a new and exciting door to meet the requirement for transforming our society towards modernization and is highly recommended to use in organic preparations for various purposes including Pharmaceutical chemistry. The examples cited above are of utmost importance and provide a brief intro of the field of microwave assisted organic synthesis.The benefits of microwave-assisted organic synthesis are tremendous, thus making the technique more established worldwide. In order to achieve further development in this field, novel instruments for more accuracy and better management, for reproducible performances and that constitute a minimal hazard should be used instead of the domestic microwave ovens.

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