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MICROWAVE ASSISTED GREEN ORGANIC SYNTHESIS

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ABSTRACT

With its twelve guiding principles, green chemistry calls for modifications to traditional chemical synthesis and the use of less hazardous raw materials. Green Chemistry aims to make synthetic processes more effective using less harmful solvents, fewer steps in the process, and as little waste as is practically possible. This is how chemical synthesis will be a component of the sustainable development approach. Since the initial papers on microwave irradiation's use to synthetic chemistry, it has been obvious that this approach will have a significant impact on all facets of this field. Reaction times were sped up, yields were enhanced, selectivities were changed, product purity increased, and work-up procedures were made simpler, and in most cases, these conditions and outcomes could not be obtained using conventional heating. Because one component of the reaction strongly absorbs microwave radiation, this methodology can be incorporated into the idea of green chemistry. This results in faster reaction times and greater energy efficiency. Additionally, the synergy of this unconventional energy source with solvent-free conditions, solid catalysts, and environmentally friendly solvents has increased the scope of its green applications. The Use Of Thermal Microwave Irradiations To Achieve Rapid Heating & High Bulk Reactions Is Vital To This Method. Reaction vessels are used to carry out the microwave synthesis. Instead of using Borosilicate glass, this process uses the silicon carbide (SiC). Nanomaterials, nanocatalysts, and prototype equipment are used to carry out the microwave assisted synthessis. Nanomaterials come in variety of Shapes including Nanorods, Nanoprisms, hexagons, spheres, and stars. Noble and transition metals can be synthesized in a repeatable manner using microwave assisted synthesis.[2,4,6]

KEYWORDS: Green chemistry, Microwave Assisted Organic Synthesis, Microwave Irradiation, Catalysts, Environmentally Friendly Solvents, Rapid Heating.

INTRODUCTION

Green chemistry is defined as the design, manufacture and use efficient, effective, safe and environmental friendly chemical processes and products. Entire 12 principles of Green Chemistry are covered by Microwave assisted organic reactions. Microwave assisted organic reactions are useful in the synthesis of Active Pharmaceutical Ingredients (API's), drug intermediates, and other compounds with chemical and medicinal importance. This technology improves the chemical processes and reduces the pollution (solvents free reactions). Microwave-assisted reactions maximizes the efficient use of safer raw materials and reduces the generation of toxic materials.

The ability of microwave assisted organic synthesis approach to improve the productivity of chemical reactions by improving speed, higher yields, lesser byproducts, pure desired products and cleaner reactions has moved the scientists on the finer track representing one of the important dimensions of modern chemistry. Microwave heating refers to use of 0.01 m to 1 m wavelength of frequency 30 GHz to 0.3 GHz to generate heat in the material. Microwave is present between the electromagnetic spectrum of IR and Radio wave. The basic mechanism of microwave is dielectric polarization and conduction.

The phrase green chemistry is spreading throughout the world is used to describe how chemical product designs are made, and products that minimises or completely stop the use or production of compounds harmful to human health in the scientific community. A term that was developed by the US Environmental Protection Agency has been described using set of principles that decrease or stop the production or use of hazardous substances in the development, production and use of chemical products that can be used to accomplish this.[1,2,3,6,7]

Microwave and "12" Principles of green chemistry[2]

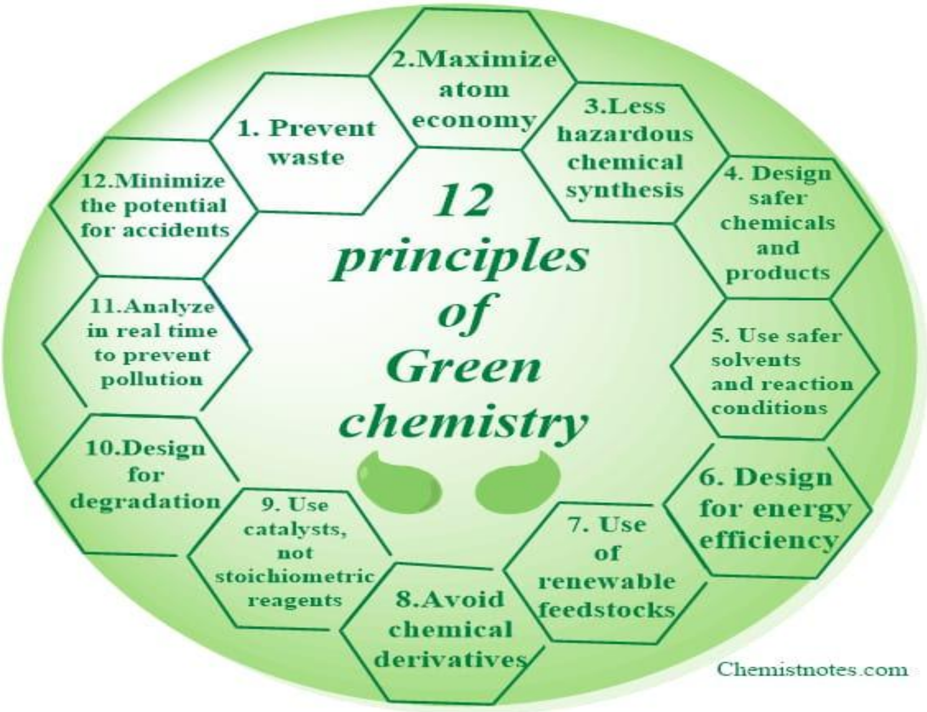


Fig.. 1

1) **Preventing waste:-** It is preferable to treating and reducing up waste that has been produced.
2) **Maximize Atom Economy**:- Synthetic techniques must be created to incorporate all resources as much as possible, used to create the finished products.
3) **Less hazardous Synthesis:-** Artificial ways to be created to use and produce less harmful or hazardous substances.
4) **Design Safer Chemicals and Products:-** Chemicals out to be designed have the desired effect while being as minimal as possible their harmfulness.
5) **Use Safer Solvents:-** Using supplementary materials and solvents. Wherever feasible, substances should be rendered unneeded and harmless while in use.
6) **Design for energy Efficiency:-** The energy needs of minimizing chemical processes and increasing synthetic procedures should be carried out at room temperature and try to apply pressure.
7) **Use of Renewable Feedstocks:-** Whenever possible, a raw materials used should be renewable rather than finite.
8) **Avoid chemical Derivatives:-** If at all feasible, avoid or minimize the need of derivatives.

9) **Use Catalysts:-** Stoichiometric reagents are inferior to catalytic reagents so that catalysts should be used instead of stoichiometric reagents.
10) **Design For Degradation:-** Create chemicals that degrade & can be Easily discarded. Verify that neither chemicals nor the products of chemical degradation are toxic, Bioaccumulative, or Environmentally Persistent.
11) **Real Time Analysis to Prevent Pollution:-** To prevent the formation and release of any hazardous and polluting substances, monitor chemical reactions in real-time.
12) **Minimize the Risk of Accidents:-** Select and create safer chemical processes that automatically lower the risk of accidents.

The development of chemistry has been strongly linked to the identification of novel reagents and strategies for supplying energy to chemical Processes. Energy could only be produced thermally by using fire throughout the era of alchemy, but with the discovery of the lens that could direct sunlight onto a continer for a chemical processs demonstrated that the energy essential to complete a chemical reaction could be acquired from other sources. The separation of sodium, potassium, calcium, strontium, and other elements by Humphrey Davy as employing electrolytic techniques on barium as Pure metals in the early nineteenth century, gave a great example of the opportunities that a new energy source could create, completely new chemical fields. Since then, chemists have kept a close eye on emerging energy sources in the hopes that they can also give rise to creative synthetic possibilities. The first trust worthy generator of microwave radiation with a set frequency was De-At The University of Birmingham During World War-II, Randall and Booth signed the document. During the war, the magnetron was created in vast quantities because it constituted the foundation for radar transmitters, especially those found in air craft and anti-air craft batteries. As a result, When visible light and infra red radiation were First Recognized as having the potential to it did not came as great surprise when a substance was found to be capable. It was initially noted that food might be heated by microwave irradiation.

In 1986, a paper detailing the first use of microwave irradiation in chemiccal synthesis was published. One of the primary functions of chemistry research is the organic synthesis, from plastics to Medication helps to improve everyone's quality of life. In Recent Decades, numerous Novel synthetic methods have made substantial progress in organic chemistry's practical aspects, strategies, tactics, and the development of a wide range of analytical methodologies. These days with everyone concerned about the environment. Technological Advancements are Focused on Cleaner and More Ecologically Friendly Processes.

1) In the last Ten Years, Microwave Irradiation has become Increasingly Popular as a potent method for quick and efficient synthesis of a wide range of chemicals due to microwaves selective absorption polar molecules absorb energy from microwave Irradiation.

2) Which Is Used to give increased Chemical Synthesis is highly beneficial because of the increased reaction rate and better product Yield, Succeeded in producing a number of different carbon-heteroatom linkages.

Microwave in organic Synthesis[1]

Researchers have been studying the mechanism of microwave dielectric heating and identifying the benefits of the approach for chemical synthesis as a result of using Microwave to speed up chemical reactions in laboratories. Microwaves have been widely Used to conduct chemical reactions in recent years, especially from 2003, linked due to the wide availability of specialized and innovative technologies, to the applications of Microwaves instruments that are reliable. Butyl acrylate, acrylic acid, and methacrylic acid were polymerized in an aqueous emulsion using pulsed electromagnetic radiation, marking the first known use of microwave energy in organic Synthesis. Microwave-Assisted Techniques For Organic Synthesis first appeared and quickly expanded was sparked in 1986 by ground breaking articles by Gedye and Colleagues and Giguere and Colleagues. The Usage of this innovative technology has increased significantly during the past two decades has undergone rigrous examination and exponential growth.

Conventional Heating Vs Microwave Heating[1,8]

In conventional synthesis oil bath or furnace is typically used to heat the reactor walls by convection or conduction. The samples core takes substantially longer time to reach the desired temperature. This is slow and inefficient method for transfer Since a number of chemical processes may be carried out by with a significant reduction in the reaction time, the use of Microwaves in chemistry is so alluring that it was discovered from the very beginning. Reaction times that are often hours or days long, under several minutes or even less can be used to test the impact of Microwave radiation. Many hypothesis that have been put out to explain why reaction times under Microwave is less in compared to the process under standerd circumstances. Despite the fact that the course of the chemical process contraindicates the first group of beliefs, Microwave conditions is significantly shorter than

Conventional Conditions. The reaction time is Shorter as a outcome of the reaction mixture and it's rapid and often unmanageable temperature rise when exposed to Microwave Irradiation. Which inturn causes a rise in reaction rates after typical kinetic laws. It is important to note that, in contrast to Conventional Heating methods, Microwaves use volumetric heating of materials which results in more extensive internal heating than external heating. This makes it difficult to create a proper seal. It is challenging to quantify temperature when using Microwave, Especially for processes taking place in dense, solvent-free medium. As a result, it is the cause of inconsistent descriptions of the same experiments and improper interpretation of scientific data.

Comparison of Conventional and Microwave Synthesis of Phenyl-1H-pyrazoles and Phenyl-1H-pyrazoles-4-carboxylic Acid Derivatives.

Fig..2

Advantages of microwave heating[1,3]

1) **Speed:** Microwave reactions can be completed in minutes. Some chemical reactions complete in second.
2) **Economy:** Microwave reations utilize no or low volume of solvents.
3) **Cost effective:** Microwave reactions reduce the cost per microwave reactions mainly through increasing the reaction rate there by yields.

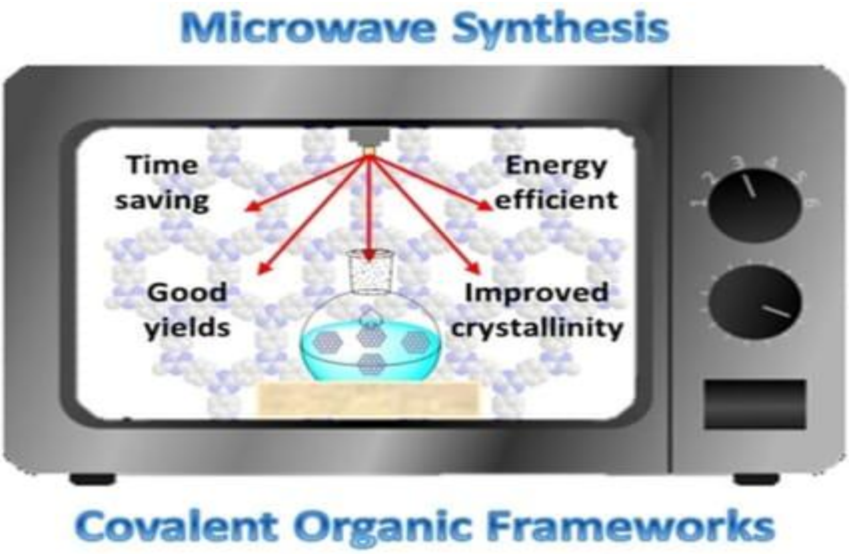


Fig..3

4) **Simplicity:** The products of microwave reactions can be isolated very easily and requires no purification in most cases.
5) **Consistency:** Microwave reactions are reproducible.
6) **Higher yields:** The rapid-efficient reactions inhibits the byproducts formation and hence offers higher yields of the products.
7) **High purity:** The rapid-efficient reactions inhibits the byproducts formation and hence offers highly pure compounds.
8) **Superheating:** It takes the reaction environment to very high temperature. It is very essential for the several reactions such as substitution and coupling reaction.
9) **Versatility:** The microwave heating can be utilize for all kind of organic reactions. It includes substitution, coupling, rearrangement, oxidation and reduction etc.

Disadvantages of microwave heating

1) **Dry food:** Cooking food in a microwave might result in overcooking and drying out certain meals due to the high heat intensity.
2) **Soggy foods:** Microwaves essentially work on an entirely different principle altogether, so they can't give you crispy results. As electromagnetic radiations are used to heat the foods Water molecules in the food are removed.
3) **Not versatile:** One of the most significant disadvantages of microwave is the inability to bake, roast or fry foods. Making Chapattis and rotis is also impossible.

4) **Need special containers:** When using a microwave for cooking, you must be very careful about the cookware or containers you use. It is safe to use microwave bowls explicitly developed for this purpose.
5) **Chances of food poisoning:** Due to Microwave ovens severe food poisoning can be caused. The presence of hot spots cold spots in food occurs due to the standing waves pattern in the microwave

Application of microwave assisted synthesis[1,8]

1) **Microwave irradiation is very much useful in the following chemical reactions**

• Protection and deprotection reactions: Functional group protection and deprotection strategies are important in carbohydrate chemistry and peptide synthesis. Microwave chemistry is more useful in these reactions.

• Named organic reactions: Gabriel synthesis, Suzuki reaction, Williamson- ether synthesis and Pinacol-Pinacolone rearrangement.

• Oxidation, esterification, O-alkylation and aromatic electrophilic substitution reactions.

• Preparation of medicinal compounds such as sildenafil, phenytoin, benzocaine are attempted successfully.

• Drug intermediates, namely, 1,4-dihydropyridines, chalcones, carvones. Thioflavanoids and γ-carbolines also are synthesised.

• Novel cepahalosporins are synthesised using microwaves.

• Asymmetric reactions were also successfully attempted.

2) **In analytical chemistry**

A) Ashing:- The ash content determination such as loss on ignition (LOI) and residue on ignition (ROI) is an important quality control procedure. Microwave ashing provides reduced time, cost and reduced exposure to fumes.

B) Digestion:- Microwave digestion assists in dissolving the metals in minutes during elemental analysis. Microwave digestion can oxidize compounds more effectively than conventional methods.

C) Moisture Analysis:- The microwave-assisted moisture analysis overcome the limitations of Karl-Fishcer (destructive). Microwave analyzer works on higher dielectric constant (attentuate energy transfer) and offers non-destructive moisture measurement.

3) In natural product extraction and isolation:- Extraction and isolation of active principal from plant material is very critical. The conventional methods requires longer extraction procedures and higher solvent volume. Microwave-assisted extraction (MAE) is useful in the extraction of plant tissues using relatively less volume of solvent with higher extraction power. Thus it helps in overcoming the issues of conventional methods.

4) In food industry:- Microwave heating is successfully applied in food processing such as pasteurization and preservation. Microwave pasteurization is an efficient technique for milk and fruit juice. Another important application for food industry is microwave blanching.

Solvents Used In Microwave Assisted Green Organic Synthesis[2]

Table 1:- Solvents used for microwave assisted organic synthesis.

SR.NO	SOLVENTS	DIELECTRIC CONSTANT	BOILING POINT C	DIELECTRIC LOSS
1.	Water	80.4	100	9.88
2.	Formic acid	58.5	100	42.37
3.	DMSO	45	189	37.12
4.	DMF	87.7	153	-
5.	Acetonitrile	37.5	82	2.32
6.	Ethylene glycol	37	197	49.95
7.	Nitro methane	36	101	2.3
8.	Nitrobenzene	34.8	202	20.47
9.	Methanol	32.6	65	21.48
10.	Ethanol	24.3	78	22.66
11.	Acetone	20.7	56	1.16
12.	1-Propanol	20.1	97	15.21
13.	1-Butanol	17.1	118	9.76
14.	2-Butanol	15.8	100	7
15.	Isobutanol	15.8	108	8.24
16.	Dichloromethane	9.1	40	0.38
17.	THF	7.4	66	0.34
18.	Acetic acid	6.2	113	1.07
19.	Ethyl acetate	6	77	0.33
20.	Chloroform	4.8	66	0.43
21.	Chlorobenzene	2.6	132	0.26

Various Types of Microwave assisted Organic Reactions[1,2,8]

The microwave-assisted organic reactions have been broadly classified into two categories: microwave-assisted reactions using solvents; microwave-assisted reactions using solvent-free conditions.

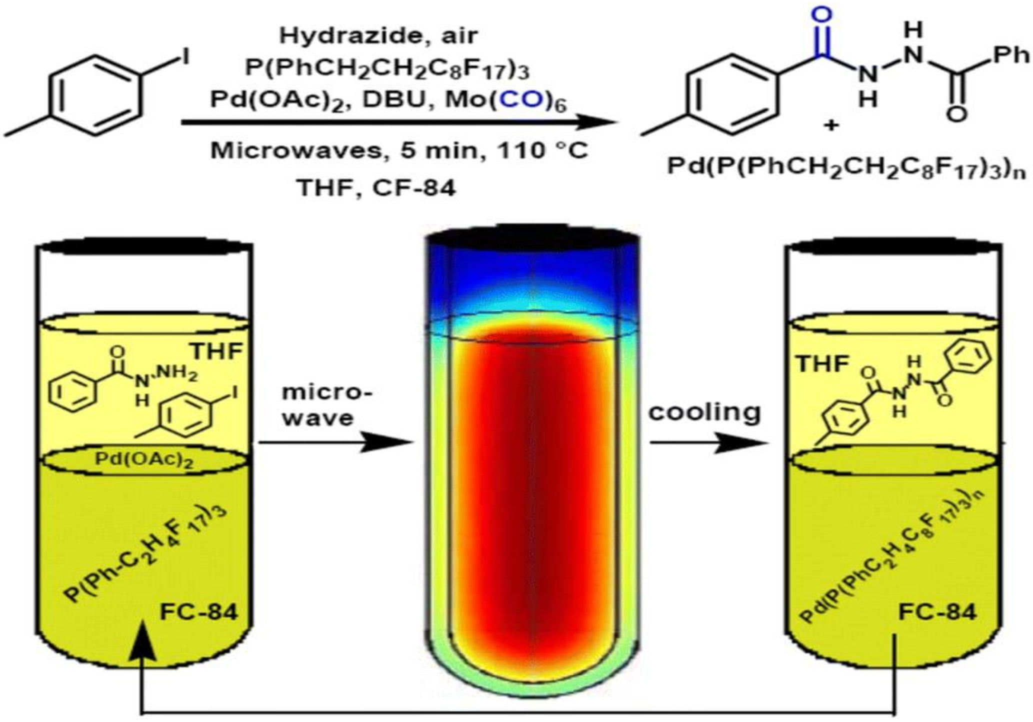


Fig 4.

A) Microwave Assisted Reactions Using Solvents

In the case of the microwave-assisted reactions using (organic) solvents, the reactants are usually dissolved in the solvent, which often couples effectively with microwaves and thus acts as the energy transfer medium.

The use of aqueous media for organic reactions is also under active investigation and temperatures of up to 100 °C and above have been employed for the syntheses often intended to exploit the hydrophobic effect. Water has a dielectric constant 78 at 25 °C which decreases to 20 at 300 °C; the latter value being comparable with that of the solvents, such as acetone, at ambient temperature. Thus, water at elevated temperature can behave as a pseudo-organic solvent and is a possible environmentally benign replacement for organic solvents. In addition to the environmental advantages of using water instead of the organic solvents, isolation of the products is often facilitated by the decrease of the solubility of the organic material upon post-reaction cooling.

An alternative method for performing microwave assisted organic reactions, termed enhanced microwave synthesis (EMS), has also been examined. More energy can be directly applied to the reaction mixture, By externally cooling the reaction vessel with compressed air, while simultaneously administering microwave irradiation. In the conventional microwave synthesis (CMS), the initial microwave power is high, increasing the bulk temperature (TB) to the desired value very quickly. However, upon reaching this temperature, microwave power decreases or shuts off completely in order to maintain the desired bulk temperature without exceeding it. When microwave irradiation is off, classical thermal chemistry takes over, losing the full advantage of microwave irradiation, which is used to reach TB faster. Microwave enhancement of chemical reactions will only take place during the application of the microwave energy. This source of energy is not desirable to suppress its application because it directly activate the molecules in a chemical reaction. EMS ensures that a high, constant level of microwave energy is applied, resulting in the significantly greater yields and cleaner chemistries.

Namely microwaves and water has become very popular Recently, the combination of two prominent green chemistry principles, and received substantial interest.

A plethora of very recent synthetic applications describes a variety of new chemistries that can be performed with microwave irradiation but a wide range of microwave- assisted applications is still waiting. Many organic transformations proceed via radical chemistry. As chemists wonder if microwave irradiation can promote radical transformations, microwave-assisted freeradical chemistry is increasingly being explored. Microwave irradiation is applicable not only to the solvent phase chemistry, but also to the solid-phase organic synthesis.

Following are the example of microwave assisted reaction using solvents

1) Hydrolysis

Hydrolysis of benzyl chloride with water in microwave oven gives 97% yields of benzyl alcohol in 3 min. The usual hydrolysis in normal way takes about 35 min.

Reaction:1

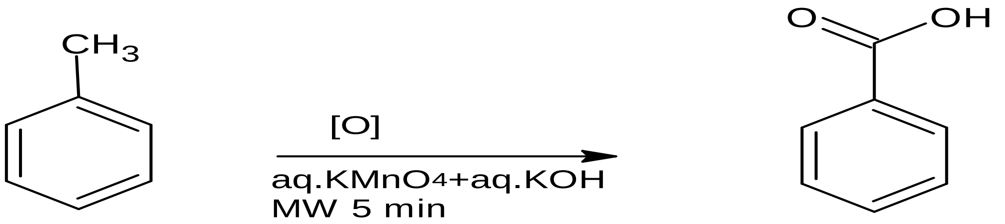
$$C_6H_5CH_2Cl \quad + \quad H_2O \longrightarrow C_6H_5CH_2OH$$

Benzyl Chloride — Benzyl Alcohol 97%

2) Oxidation

Oxidation of toluene with KMnO4 under normal conditions of refluxing takes 10-12hr compared to reaction in microwave conditions, which takes only 5 min and the yields is 40%.

Reaction: 2



Toluene Benzoic acid

3) Esterification

A mixture of benzoic acid and n-propanol on heating in a microwave oven for 6 min in presence of catalytic amount of conc. Sulfuric acid gives propylbenzoate.

Reaction: 3

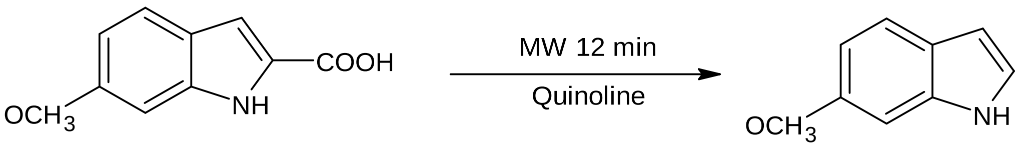
$$C_6H_5COOH + nC_3H_7OH \xrightarrow[\text{MW 6 min}]{\text{Conc } H_2SO_4} C_6H_5COOC_3H_7$$

Benzoic acid Propylbenzoate

4) Decarboxylation

Conventional decarboxylation of carboxylic acid involve in quinolone in presence of copper chromate and the yields are low. However in the presence of microwaves decarboxylation takes place in much shorter time.

Reaction:4

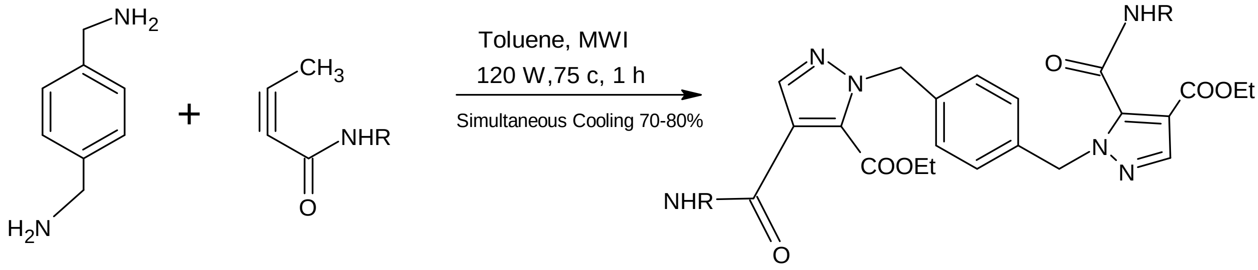


6 Methoxy indole 2 carboxylic acid 6 Methoxyindole

5) Cycloaddition

1,3-Dipolar cycloadditions are important reactions in organic synthesis. Cycloaducts were prepared by carrying out the reaction between an azide and a substituted amide in toluene. This reaction was carried out under microwave irradiation at 120 W at 75 degree C for 1hr. The products was isolated in 70-80% yield.

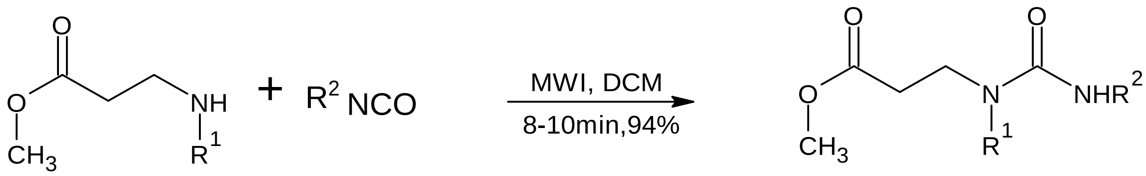
Reaction:5



6)N-Acylations

N-Acylations were carried out using secondary amines and isocyanate in dichloromethane under microwave irradiation (8-10 min), yielding the product in 94% yield.

Reaction:6



A) Microwave assisted Reactions under Solvent-Free Conditions

Due to the environmental concerns, there has currently been an increasing demand for efficient synthetic processes and solvent-free reactions. Some old and new methodologies are being used to diminish and prevent pollution caused by chemical activities. In this context, the microwaves have become an important source of energy in many laboratory procedures.

Furthermore, microwave-assisted solvent-free organic synthesis (MASFOS) has been developed as an environmentally friendly process as it combines the selectivity associated with most reactions carried out under microwaves with solvent and waste-free procedures in which organic solvents are avoided throughout all stages. In these environmentally conscious days, the research and development are directed towards devising cleaner processes. Environmental hazards and the subsequent degradations are instrumental for the rapid evolution of green chemistry concept involving benign reagents and conditions. The MASFOS reactions are of three types.

A) Reactions using neat reactants

B) Reactions using solid-liquid phase transfer catalysis (PTC)

C) Reactions using solid mineral supports.

A) Reactions Using Neat Reactans

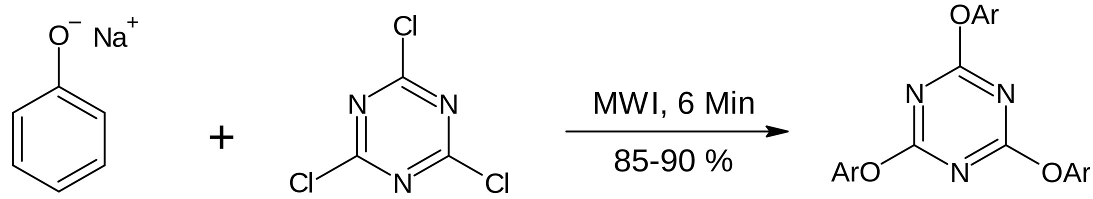
For carrying out reactions with neat reactants i.e without the use of a solvent or a support (heterogeneous reactions), at least one of the reactants at the reaction temperature should normally be liquid. In such a set-up, either the solid is partially soluble in the liquid phase or the liquid is adsorbed onto the surface of solid with the reaction occurring at the interface. There is also another possibility, namely that both the reactants are solid. Usually, during the reaction course they melt and then undergo reaction as described above.

Following are the Examples of Microwaves assisted Reactions with neat reactants

1) Aromatic Nucleophilic Substitution

Formation of Substituted Triazines Aromatic nucleophilic substitutions are carried out using sodium phenoxide and 1,3,5-trichlorotriazine under microwave irradiation (6 min). The products, 1,3,5-triarlyoxytriazines are obtained in 85–90% yields.

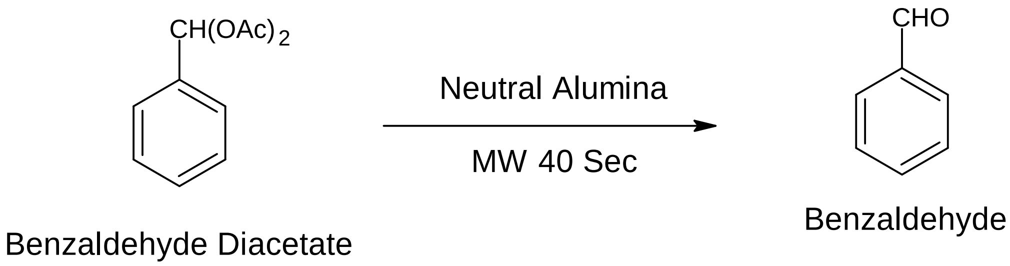
Reaction:7



2) Deacetylation

Aldehydes, phenol and alcohols are protected by acetylation. After the reaction, the deacetylation of the product is carried out usually under acidic or basic conditions the process takes long time and the yields are low. Use of microwave irradiation reduces the time of deacetylation and the yields are good.

Reaction:8



B) Microwave assisted Reactions using Solid Liquid Phase

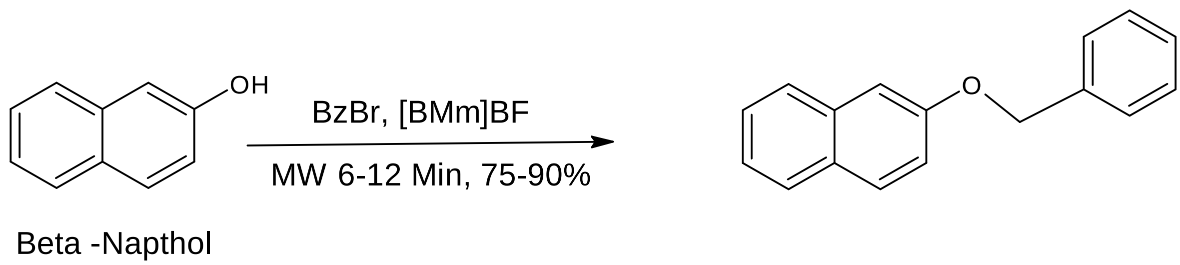
Solid liquid phase transfer catalysis (PTC) has been described as an effective method in organic synthesis and is under active investigation. This methods is specific for anionic reactions as it involves anionic activation. A catalytic amount of a tetralkylammonium salt or a cat ion complexing agent is added to the mixture (in equimolar amounts) of both pure reactants. Reactions occur in the liquid organic phase, which consists here only of the electrophilic R-X. The presence of an additional liquid component is disadvantageous as it induces a dilution of reactants and consequently a decrease in reactivity. The electrophile R-X is therefore both the reactant and the organic phase for the reaction.

Following are the Example of Microwave assisted Reaction using Solid Liquid Phase

1) O-Alkylation

Preparations of ethers were carried out from β-naphthol using benzyl bromide and 1-butyl-3-methyl-imidazolium tetrafluoroborate under microwave irradiation (6-12 min) the products were isolated in 75-90% yields.

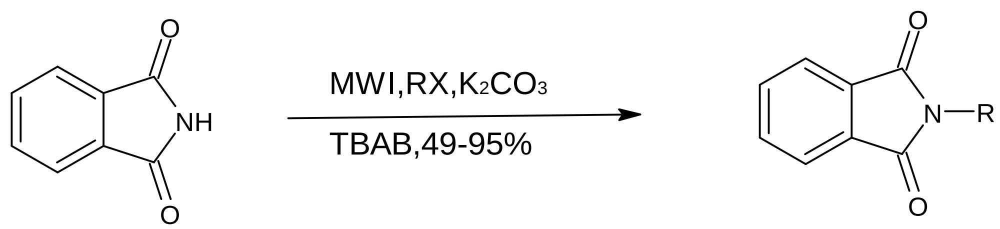
Reaction: 9



2) N-Alkylation

N-Alkylations under microwave irradiation using phase transfer catalysts occupy a unique place in organic chemistry. Bogdal and co-workers reported the synthesis of N-alkyl phthalimides using phthalimide, alkyl halides, potassium carbonate and TBAB; giving products in 45–98% yields.

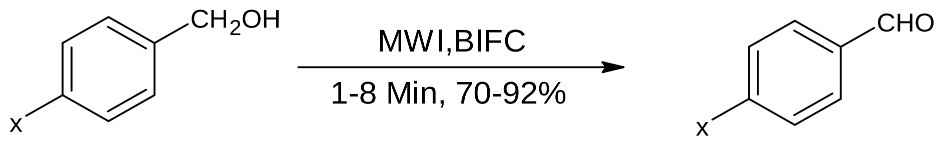
Reaction:10



3) Oxidation

Chakraborty reported the oxidation of secondary alcohol and benzyl alcohols using phase transfer catalysts. Oxidation of secondary alcohols to acetone derivatives was carried out using PCC, tetrabutylammonium bromide and dichloromethane under microwave irradiation (6–8min), products were isolated in 70–99% yields. Oxidation of benzyl alcohols was conducted using BIFC under microwave irradiation (1–8 min) yielding benzaldehyde derivatives in 70–92% yields.

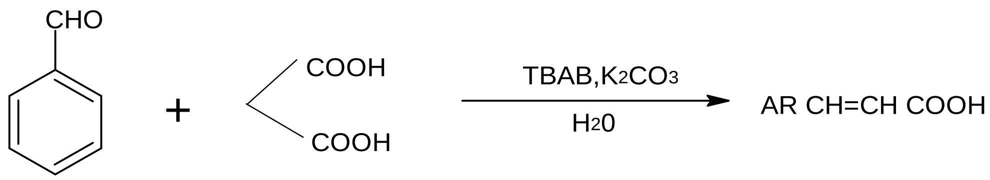
Reaction:11



4) Knoevenagel Condensation

Knoevenagel condensation is a well known organic reaction, other applied in the synthesis of unsaturated acids, which are used as precursors for perfumes, flavonoids and as building blocks of many heterocycles. Gupta and Wakhloo studied knoevenagel condensation between carbonyl compounds and active methylene compounds, such as malonic acid, using tetrabutylammonium bromide, potassium carbonate in water forming unsaturated acids in excellent yield and purity under microwave irradiation.

Reaction:12



C) Microwave assisted Reactions on Mineral Supports in Dry Media

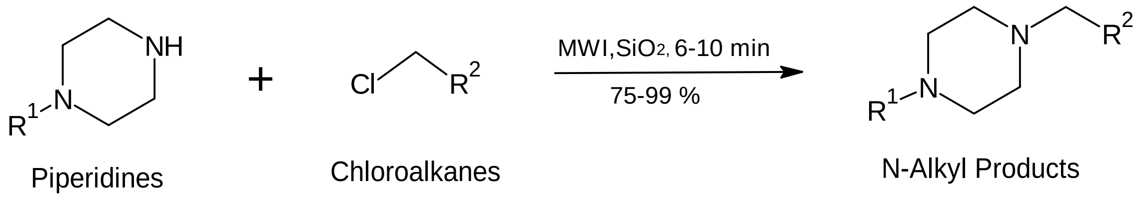
Solid supports are often very poor conductor of heat but behave as very efficient microwave absorbents. This, in turn results in very rapid and homogeneous heating. Consequently, they display very strong specific microwave effect with significant important in temperature homogeneity and heating rates enabling faster reactions and less degradation of final products as compared to the classical heating.

Following are the Example of the Microwave Activation with Supported Reagents.

1) N-Alkylation

N-Alkylation were carried out between piperidines and chloroalkanes in the presence of silica as the solid support under microwave irradiation (6-10 min). N- Alkyl products were isolated in 79- 99% yields.

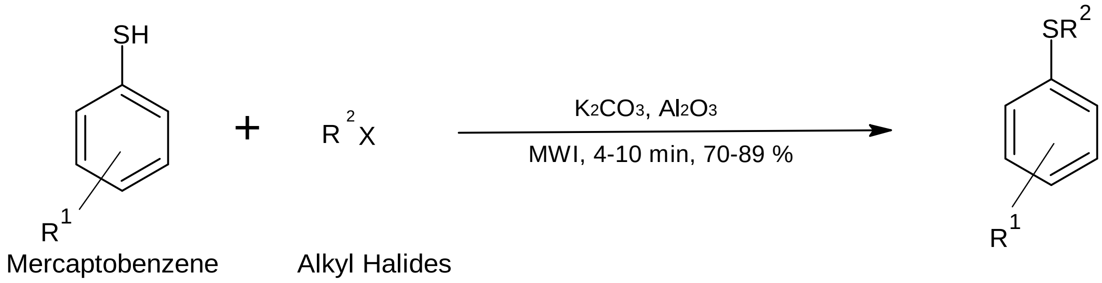
Reaction:13



2) S Alkylation

S-Alkylation was studied and accomplished by carrying out the reaction between mercaptobenzene and alkyl halides using potassium carbonate and alumina under microwave irradiation (4-10 min). Products were isolated in 70-89 % yields.

Reaction:14



CONCLUSION

Microwave heating is very convenient way towards goal of green chemistry used in organic preparation. Microwave heating is instantaneous, very specific and there is no contact required between energy source and reaction vessel. The most important goal of chemist in ensuring that our next generation of synthetic protocol for drugs and fine chemicals are more sustainable and greener that the current generation. The uses of emerging microwave assisted chemistry in conjuction with utility of greener reaction media or solvent free conditions are effective and sustainable techniques to reduce chemical waste & reaction time of organic synthessis. It is technique which can used to rapidly explore chemistry space and increased in the diversity of compound prepared. Now a days, it could be considered that all the previously conventionally carried reactions should be performed using this microwave assisted technique. In the end we can say that microwave mediator synthesis is most efficient and most effective method that of conventional synthesis.

ACKNOWLEDGEMENT

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