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Review Article

Microwave Assisted Organic Synthesis of Heterocyclic Compound

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ABSTRACT

Microwave-assisted organic synthesis (MAOS) has transformed the preparation of heterocyclic compounds, offering significantly reduced reaction times, milder conditions, enhanced yields, and improved product purity compared to conventional heating methods. This review summarizes key advances in MAOS for nitrogen-containing heterocycles such as pyrroles, pyrrolidines, oxazole, and indoles, emphasizing green chemistry approaches including solvent-free and aqueous processes. The principles, mechanisms of dielectric heating, and distinctions between microwave and conventional heating are discussed. Recent progress in multi-component reactions, condensations, decarboxylation's, and practical applications in pharmaceuticals, materials, and nanotechnology are illustrated, highlighting the accelerating impact of microwaves on environmentally sustainable synthesis.

INTRODUCTION

Microwave technology, though introduced in chemistry in the late 1970s, gained prominence in organic synthesis only from the mid-1980s after initial challenges with reproducibility and understanding of dielectric heating were addressed. Today, microwave-assisted organic synthesis (MAOS) has rapidly evolved, resulting in over 3500 publications and significant interest in the synthesis of nitrogen-containing heterocycles such as pyrroles, indoles, and quinolines, which are vital for pharmaceuticals and natural products. Pyrroles, in particular, serve as key structural elements in biologically active compounds like hem, vitamin B12, bile pigments, and chlorophyll, and are utilized in various pharmaceutical applications and polymer chemistry. MAOS offers substantial advantages over traditional methods, including shorter reaction times, higher yields, milder conditions, enhanced product purity, and greener processes,

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with many syntheses now occurring in solvent-free or aqueous environments.

1.1 PRINCIPLES OF MICROWAVE HEATING

- Preventing waste is better than managing or cleaning it up after it has been produced.
- It is important to design synthetic procedures so that all of the materials used are completely incorporate into the finished product .
- To enable real-time monitoring and control during processes and prevent the creation of hazardous compounds, analytical techniques need to be further developed.

1.2 Mechanism of heat generating by microwave:

1.2.1 Dipolar polarization

In microwave-assisted synthesis, heat generation is primarily driven by dipolar polarisation, where molecules with a persistent dipole align with the microwave's electric field. This alignment leads to molecular oscillation and collisions, creating heat through friction. Only reagents that are polarisable and possess a dipole moment are responsive to microwave heating. Compounds with higher polarizability show stronger heating effects. Thus, microwave heating is mainly effective for polar molecules such as water, methanol, ethanol, ammonia, and formic acid, while non-polar molecules remain unaffected.

1.2.2 Ionic conduction

Ionic conduction occurs when an electric field causes ions in a substance to move, leading to rapid superheating through frequent collisions and heat generation. As temperature rises, energy transfer during this process becomes even more efficient. Ionic liquids are particularly adept at absorbing microwave energy, enabling quick and effective heating via ionic conduction. In microwave heating, tap water, which contains dissolved ions, heats to a higher temperature than distilled water due to this effect. The combined action of ionic conduction and dipolar polarisation increases the overall temperature in ionic solutions subjected to microwave fields.

1.3 Comparison between conventional heating and microwave heating

In addition to being slow, heating reactions with conventional apparatus such heating mantles, sand baths, and oil baths produces a heated surface on the reaction vessel where products, substrates, and reagents frequently break down over time. It causes the temperature of the reaction vessel to be greater than the temperature of the reaction mixture and is dependent on the thermal conductivity of the different materials that need to be penetrated. On the other hand, microwave energy is remotely added to the chemical reactor and heats the reactants and solvents directly through the reaction vessel's walls. As a result, there is less of a tendency for boiling to begin, and even at atmospheric pressure, superheating above the solvent's boiling point is feasible [9] in closed vessels usually composed of (almost) microwave-transparent materials, like Teflon, quartz, or borosilicate glass, superheating can be produced quickly to temperatures up to 100°C over the conventional boiling point of a given solvent. The rate of the reaction about doubles for every 10°C rise in temperature. Because there is no hot vessel surface due to the extremely effective internal heat transmission, wall effects are reduced, which could result in the detection of so-called particular microwave effects. However, by heating particular reagents or catalysts selectively, macroscopic or microscopic hotspots may form, enabling even faster conversions and the realisation of

chemistries that are not conceivable with traditional heating.

1.4 Microwave theory

Microwave irradiation, operating at a frequency of 2.45 GHz to avoid interference with telecommunications, is a form of electromagnetic radiation with photon energy too low to break chemical bonds or directly induce chemical reactions. Instead, microwave-enhanced chemistry relies on dielectric heating, where materials such as solvents or reagents absorb microwave energy and convert it into heat. This heating occurs primarily through two mechanisms: dipolar polarisation, where molecular dipoles attempt to align with the oscillating electric field and create heat due to friction and dielectric loss; and ionic conduction, where charged particles oscillate and cause heating through collisions with neighboring molecules. The frequency used allows molecules enough time to partially align with the field, maximizing heat generation without following the field perfectly.

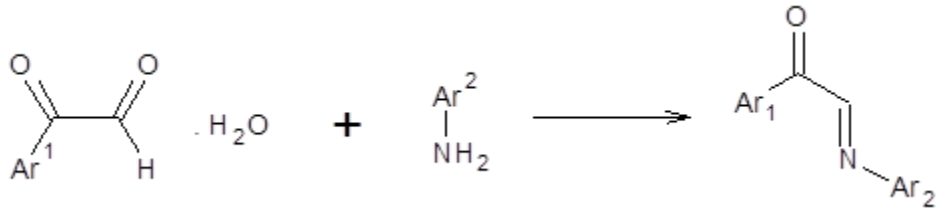
2. TYPES OF MICROWAVE ENERGY ASSISTED SYNTHESIS-

2.1 Microwave assisted reaction using solvents

In microwave-assisted reactions, solvents dissolve reactants and efficiently transfer microwave energy. Temperatures often exceed 100 °C to exploit effects like hydrophobic interactions. Water is increasingly studied as a green solvent due to its high dielectric constant (78 at 25 °C), which decreases to 20 at 300 °C, similar to organic solvents. At elevated temperatures, water behaves like a pseudo-organic solvent, offering a safer environmental alternative. Additionally, cooling after the reaction reduces organic solubility, facilitating easier product isolation.

2.1.1 Hydrolysis:

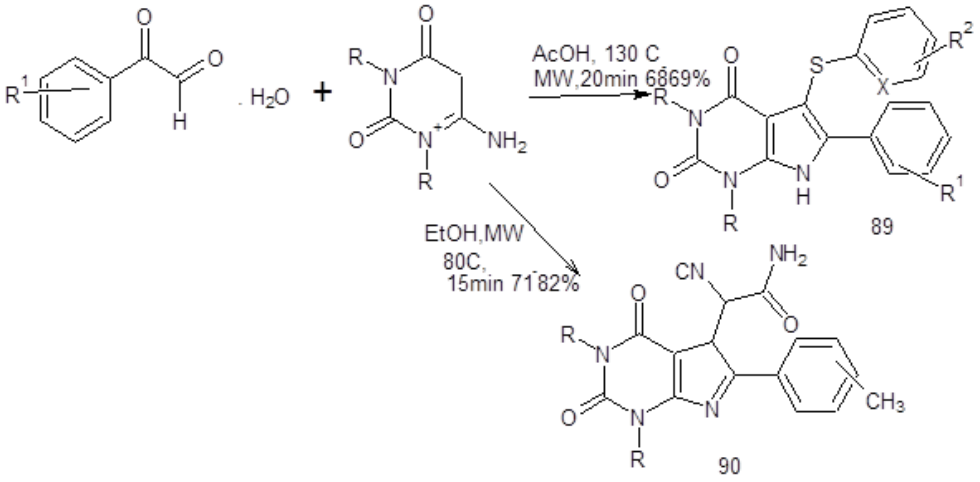
The synthesis is described by a logical mechanism in which molecules and condense to produce an intermediate A, and an acid undergoes nucleophilic addition to form an intermediate B. With the help of an acid, this intermediate proceeds through an intramolecular cyclisation C to produce intermediate D, which, when a water molecule is lost, products



disclosed a three-component reaction of substituted aryl glyoxal monohydrate , 6-amino-1,3-disubstituted uracil and substituted thiols under microwave conditions using acetic acid as a solvent to successfully furnish 5,6-disubstituted pyrrole [2,3-*d*] pyrimidine-2,4-diones . Similarly, excellent yields were obtained when the thiol was replaced by malononitrile [11] even in the absence of catalyst or any promoter.

The malononitrile undergoes hydrolysis forming an amide, thus giving rise to a series of pyrrole[2,3-*d*] pyrimidine-2,4-diones acetamides under microwave irradiation. An interesting observation by the authors surfaced that the conventional approach to the malononitrile protocol delivered comparable yields in longer reaction time (5–8 h). A gram-scale attempt under

microwave conditions delivered the desired product in better yields than the reflux strategy.



Scheme-

Synthesis of subtition pyrrole [2,3-d] pyramidine -2,4 diones

R	R^1	R^2
H, Me	H,4-NO_2	H,4-OMe

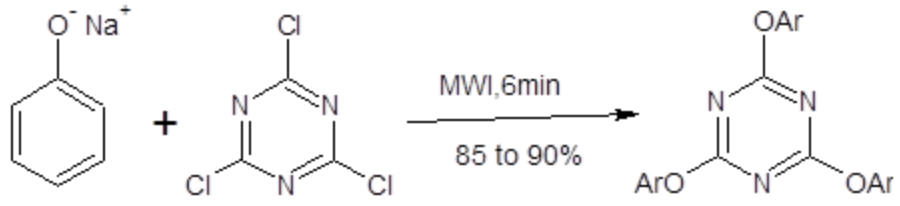
2.2 Microwave assisted synthesis under solvent free

There is increasing demand for solvent-free and efficient synthetic methods to address environmental concerns. Microwaves have become an important energy source for cleaner laboratory processes. Microwave-assisted solvent-free organic synthesis (MASFOS) combines waste-free techniques with selective reactions, avoiding organic solvents. Green chemistry advances focus on safe reagents and conditions to reduce environmental risks. Key approaches include reactions with neat reactants, solid-liquid phase transfer catalysis, and reactions using solid mineral supports.

Following are the Examples of Microwaves assisted Reactions with neat reactants.

2.2.1 Aromatic nucleophilic substitions

Substituted Triazines' Formation Under microwave irradiation (6 min), sodium phenoxide and 1,3,5-trichlorotriazine are used to perform aromatic nucleophilic replacements. The yields of 1,3,5-triarlyoxytriazines, the products, range from 85 to 90%.



2.3 Interaction of Microwave with Different Materials

Microwaves are electromagnetic waves with wavelengths from 1 mm to 1 m, lying between infrared and radio frequencies. Due to their

widespread use in telecommunications, only certain frequencies are permitted for industrial, scientific, and medical applications. Common microwave oven frequencies are 2.45 GHz (12.2 cm) and 915 MHz (32.7 cm) to avoid interference. Most commercial microwave reactors for chemical synthesis also operate at 2.45 GHz. Heating in microwave cavities depends on the ability of liquids and solids to absorb and convert microwave energy into heat. Materials exhibit three different behaviours when interacting with microwaves, depending on their nature. This interaction underpins microwave heating technology.

2.4 Advantages

Speed: microwave reactions can be finished in a matter of minutes. a few chemical processes that happen in a matter of seconds. Reaction times are frequently shortened from hours to minutes to seconds.

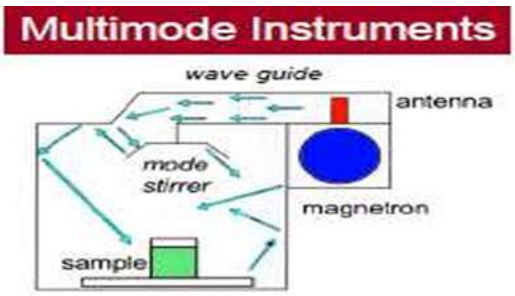
Economical: Microwave reactions use little or no solvent.

Cost-effective: Microwave reactions lower costs by primarily raising reaction rates, which results in higher yields. Simplicity: In most situations, the products of microwave reactions don't need to be purified (recrystallised) and can be easily isolated.

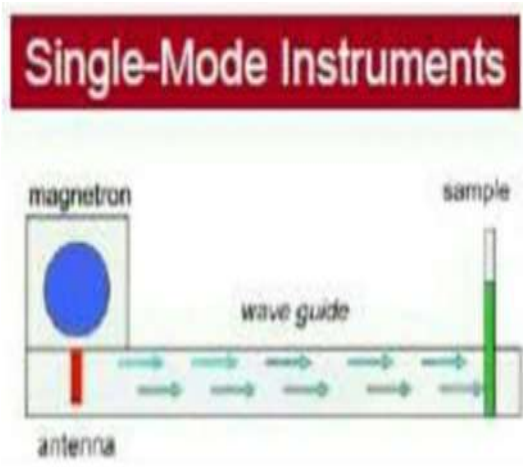
2.5 Drawback of microwave heating:

1. Using sealed containers could lead to explosions, which would be dangerous.
2. Evaporation of water can provide difficulties.
3. It's frequently challenging to control the intensity of heat.
4. It has been noted that some issues can occasionally arise when synthesis is carried out utilising microwaves.

2.6 Instruments of microwave reactors



Most microwave-assisted organic synthesis uses home ovens with multiple modes and variable power due to on-off cycling. Multimode ovens have non-uniform electric field distribution caused by reflections from the oven's iron walls. This heterogeneity requires mapping of the field for synthesis. High-energy hot spots are identified using cobalt chloride–saturated filter paper.



Single-mode microwave instruments create standing wave patterns with nodes (zero energy) and antinodes (maximum energy). Samples are placed at antinodes for optimal heating, but only one vessel can be irradiated at a time. They handle volumes from 0.2 to 50 mL in sealed vessels and up to 150 mL in open vessels. Single-mode ovens are commonly used in small-scale drug discovery, automation, and combinatorial chemistry.

3. HETEROCYCLIC NUCLEUS SYNTHESIS

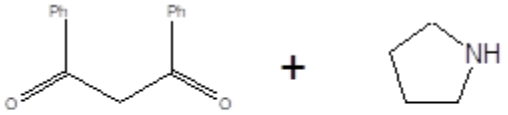
Microwave is used in synthesis of

- Five-Membered Heterocyclic Rings: Pyrroles, Pyrazoles, Imidazoles, Oxazolines, Triazoles and Tetrazoles, Oxadiazoles, Isoxazolines and Pyrazolines
- Benzo-Derivatives of Five-Membered Rings: Benz-imidazoles, Benz-oxazoles, and Benz-thiazoles, Indoles
- Six-Membered Rings: Dihydropyridines, Dihydropyridopyrimidinones, Tetrazines, Dihydropyrimidines
- Polycyclic Six-Membered Rings: Quinolines, Pyrimido [1, 2-a] pyrimidines

Microwave is used in heteocyclic C-Alkylation, heterocyclic N-Alkylation reactions.

3.1 Microwave assisted synthesis of heterocyclic compound

Microwave-assisted synthesis has transformed organic chemistry, especially for heterocyclic compounds containing sulfur and nitrogen. Traditional methods often involve complex steps and materials. Microwave techniques offer faster reactions with higher yields and simplify purification. This approach provides an efficient alternative to conventional synthesis methods.



11 12

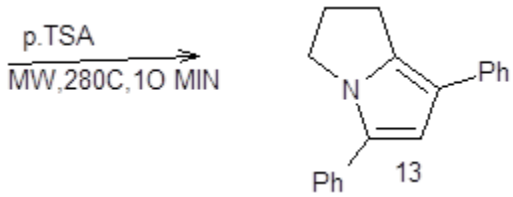
Deb et.al produced ring-fused pyrrole in a single step by reacting 1,3-diketone 11 with cyclic aniline 12 at 280 degrees Celsius when exposed to microwave radiation. Within ten minutes, the process, which was catalysed by 0.5 equivalent of p-toluenesulfonic acid (p-TSA), produced a 53% yield. Using 20 mol% ZnCl2 (1.0M in ether) to assist the ligand-free 5-endo-dig cyclisation of homopropargyl azide 14 in CH2Cl2 at 105 C, pyrroles (eight examples) were formed in 40–60 minutes with yields ranging from 91% to 41%. In contrast, the same result was obtained after 16 hours of traditional heating at 160 °C.

3.1.1 Pyrrole

One important chemical compound is pyrrole, which has the formula C4H4NH with a five-membered ring. Its many actions make it important in medicinal chemistry. Many techniques have been developed by researchers to create a variety of substitutions in pyrroles.

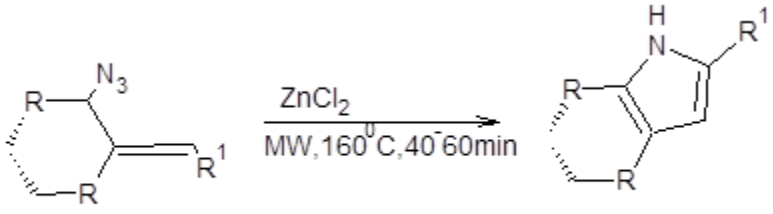
Scheme:

Syntesis of Ring Fused Pyrrole Under Microwave Irradation



13

Scheme synthesis of pyrrole by 5-endo-dig cyclisation of homopropargyl azide:

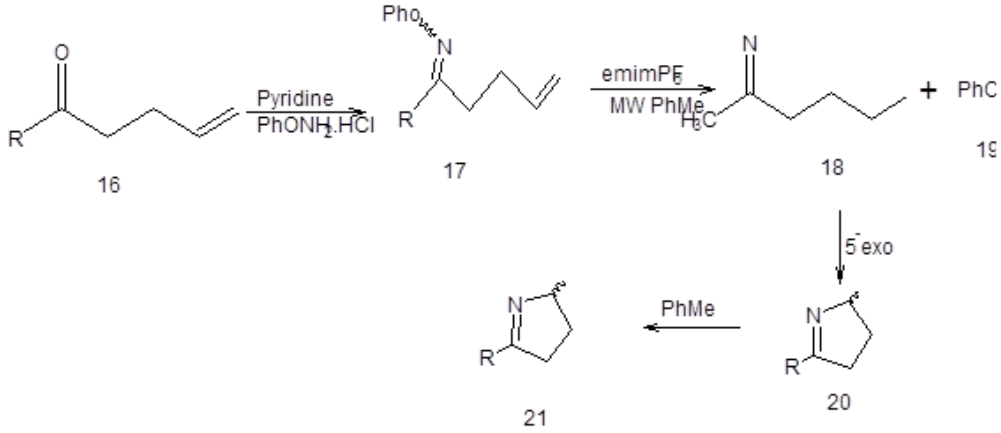


14 15

R=alkyl, aryl; R^1=H, alkyl

Using functionalised O-phenyl oxime ethers 17, which were stimulated by microwaves (MWs) to generate dihydropyrrole 21 (for example), Portela-cubillo et al. investigated the production and cyclisation of iminyl radicals with yields ranging from 68% to 82%. One equivalent of the ionic liquid 1-ethyl-3-methyl-1H-imidazol-3-ium hexafluorophosphate (emimPF6) was used in the reaction, which took place at 160 °C for 15 minutes. Notably, extended reaction durations, ambiguous product generation, and unsatisfactorily low yields made conventional thermolysis of O-phenyl oxime ethers difficult.

Scheme: **synthesis of di hydropyrrole using o-phenyl oxime ethers.**

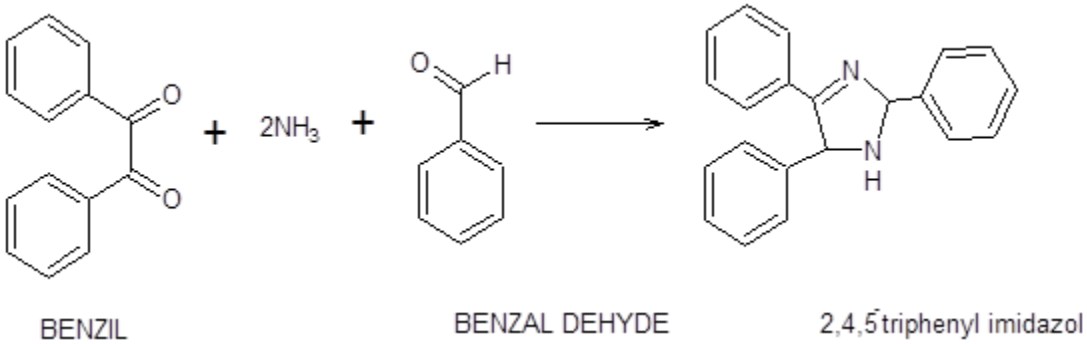


3.1.2 Imidazole

Imidazole is a five-membered aromatic ring with nitrogen atoms at positions 1 and 3, crucial in proteins as part of histidine. It acts as a biological buffer, stabilizing pH in biochemical studies, and participates in coordination chemistry with metal ions. Widely used in chemical synthesis, imidazole is vital for drug development and organic molecule production due to its electron-rich nature. First synthesized by Heinrich Debus in 1858 using glyoxal, formaldehyde, and ammonia, this method is still employed despite low yields. The compound 2,4,5-triphenylimidazole is formed by the condensation of ammonia with benzoile and benzaldehyde. Imidazole's significance spans coordination chemistry, chemical synthesis, and biochemistry.

Scheme:

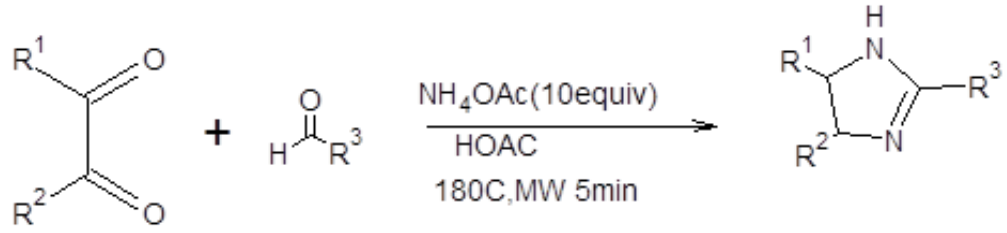
Synthesis of 2,4,5 triphenyl imidazole under microwave irradiation



Using 1,2-diketones and aldehydes, Wolkenberg et al. created a more effective synthesis process for 2,4,5-trisubstituted imidazoles. Their method, which involved using NH40Ac and microwave (MW) irradiation at 180 C for five minutes, produced significant yields (80–90%) in mild circumstances. This is in contrast to traditional procedures, which frequently call for harsher conditions (150-200 C, 4-6 hours) and produce lower yields (40-90%) and product mixes. These chemicals are useful for the synthesis of the platelet aggregation inhibitor trifenagrel and the imidazolium alkaloid lepidine B6.

Scheme:

Synthesis of 2,4,5-trisubstitited imidazole from 1,2-diketone by using MW reaction

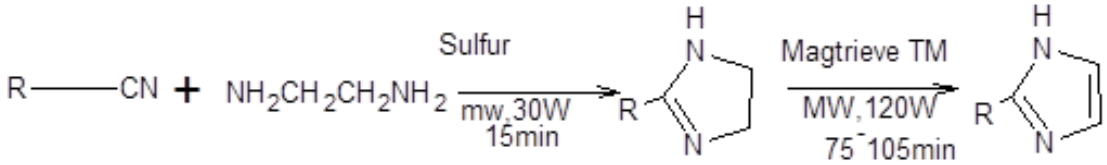


R= ALKYL, ARYL, HETEROARYL

Hoz et al. created 2-imidazolines by cyclizing nitriles with ethylenediamine with the use of a microwave. While conventional heating with MnO2 needed longer reaction durations (24-48 hours) for comparable results (76-93%), this reaction, which was carried out in toluene with magtrieve TM (oxidation), produced imidazoles (five examples) in 75-105 minutes.

Scheme:

Synthesis of 2-imidazole by using mw

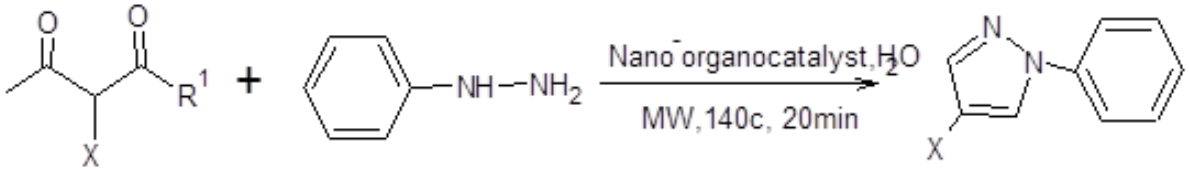


3.1.3 Pyrazole

Pyrazole is a five-membered heterocyclic ring with three carbon and two adjacent nitrogen atoms, known for its anti-inflammatory, anti-cancer, and anti-microbial activities. Researchers focus on synthesizing and modifying pyrazole derivatives to develop potential therapeutic agents. Pyrazolo[3,4-b]pyridines are studied for treating stress-related diseases. Using microwave-assisted synthesis with a nano-organocatalyst, Polschettiwar et al. achieved high yields (84–96%) in just 20 minutes at 140°C in water.

Scheme:

Synthesis of pyrazole derivative using Nano-organocatalyst by using MW- assisted



3.2 Microwave heating:

Microwave heating rapidly raises temperature by directly interacting with the molecules in the reaction mixture, especially those with dipole moments or ionic conductivity, causing localized superheating independent of the vessel's thermal conductivity. This selective heating targets the reaction contents, not the vessel, enhancing uniformity. Microwave radiation accelerates chemical reactions through thermal and specific non-thermal effects driven by the material's interaction with the electromagnetic field. Molecules with dipoles rotate to align with the rapidly oscillating electric field (at 2450 MHz), generating internal heat through friction. A material's ability to convert electromagnetic energy into heat depends on its dielectric properties, quantified by the dissipation factor tan⁡δ=ε″ε′\tan \delta = \ frac {\varepsilon"} {\varepsilon'}tanδ=ε′ε″, where a higher value indicates greater microwave susceptibility. Heat also arises from ion conduction as ions move and collide under the electric field, converting kinetic energy to heat and increasing the overall temperature.

3.3 Microwave Extraction

Microwave-assisted extraction (MAE) is one of the most important techniques for separating, recovering, and isolating phytoconstituents. The conventional extraction process requires a larger amount of solvent and a longer extraction time, and also destroys thermolabile compounds. However, microwave-assisted extraction (MAE) reduces solvent volume, speeds up extraction, and prevents oxidative destruction of phytoconstituents. Microwave extraction makes use of the concepts of maceration and percolation. The rate of extraction, or the breakdown of plant cells and tissues, is much higher in microwave extraction. In a conventional extraction procedure, heat is delivered from the heating medium to the sample. MAE provides direct heat to cell phone signals.

4. APPLICATIONS OF MICROWAVE

1. Application of microwave in material chemistry:

In material chemistry, the synthesis of inorganic solids using microwaves is a very effective and practical process. Ceramics have been prepared using microwaves, and Ayappa and groups of Kenkre et al. have examined the theoretical modelling of microwave interaction with ceramic material. Moreover, Lee et al. . According to Rao et al. SiC is produced when Si and C (charcoal) in powder form are placed in a silica crucible and microwaved for 4 to 10 minutes in a home microwave oven set to 2.45 GHz. SiC is a big volume ceramic that finds widespread industrial use in the production of abrasion tools and grinding wheels.

2. Medical application for pyrrole:

One of the most commonly found heterocyclic systems in the structure of natural products and synthetic materials is the pyrrole ring, which can be incorporated as a substituent or undergo a variety of substitutions on the ring itself. This is likely due to the ring's electron characteristics, which improve binding with enzymes and receptors and cause additional scaffold modifications to achieve the desired activity profile. While some medications with a pyrrole moiety are now in clinical testing, others are already on the market. Pyrroles are used in medicine for their strong antimycobacterial properties, and several of its analogues have good therapeutic indices.

FUTURE TRENDS:

The ability to scale up the corresponding processes, initially to a pilot plant scale and then to the production scale, is a frequent prerequisite linked to the introduction of new technologies. In order to prevent negative reaction characteristics (such as lengthy reaction times, secondary reaction times, solvent usage, excess components, etc.), microwave processing aims to speed up processes. Converting a batch operation to a continuous operation following a process analysis is another objective for process improvement. Replicating the established reaction conditions utilised in the traditional reactions in the microwave field is typically the initial step in this process.

CONCLUSION:

Microwave-assisted synthesis has established itself as a reliable and energy-efficient technique for the production of diverse heterocyclic compounds, demonstrating remarkable advantages in speed, yield, and environmental impact. The technology has enabled scalable, reproducible, and greener synthetic procedures for a wide range of valuable molecules, particularly those of pharmaceutical relevance. With continuous process optimization and industry adoption, future research is likely to prioritize sustainable reactions that minimize waste and environmental harm while leveraging microwaves' capacity for selective activation and enhanced chemical reactivity.

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