

Review

A Concise Review of Multicomponent Reactions Using Novel Heterogeneous Catalysts under Microwave Irradiation

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Abstract: Multi-component reactions for the construction of heterocycles have been fascinated by microwave energy as an alternative technique of heating, owing to the advantages over traditional reflux methods. The heterogeneous catalysts contribute significantly towards recycling, harmless, easy filtration, catalyst preparation, more life span, abundance, and product yields. With novel and creative uses in organic and peptide synthesis, polymer chemistry, material sciences, nanotechnology, and biological processes, the usage of microwave energy has rapidly increased during the past 20 years. This article covers multicomponent reactions involving construction of chromenes, pyridines, pyrroles, triazoles, pyrazoles, tetrazoles, *trans* and *cis* julolidines using heterogeneous catalysts under microwave. It provides an overview of contemporary microwave-assisted heterogeneous catalytic reactions. Microwave chemistry is now an established technology with several advantages regarding reaction rate and production yield, improving energy savings as confirmed by many applications. Due to the widespread curiosity in medicinal chemistry, the heterogeneously catalysed construction of heterocycles under microwave irradiation is explored to reduce time and energy. By considering various aspects of economy, eco-friendly, and user-friendly factors, this review focuses on recent advances in the multi-component construction of heterocycles using heterogeneous catalysts under microwave irradiation. This review also discusses the benefits and limitations of reaction conditions and yields from the literature reports for the past five years.

Keywords: multicomponent reactions; heterogeneous catalysts; microwave-assisted synthetic procedures; heterocycles



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1. Introduction

Based on a critical analysis of the published original research articles, the insightful reviews on the most recent developments in the specialised fields help the researchers be aware of the advances. Review articles are academic works that gather, condense, analyse, and synthesized knowledge on a particular area of research. Reviews can expose research gaps, give proof of applications, create guidelines for policy and practice, serve as a foundation for knowledge development, and, if done correctly, inspire new ideas and lay the groundwork for future research initiatives. Concisely, thorough review papers provide the crucial foundation for all studies and applying that knowledge.

Multicomponent reactions are progressive reactions in which easily accessible or commercially available starting components are used to produce desired single compound [1]. These reactions have been deliberately used in a variety of synthetic transformations, whereas traditional approaches typically include numerous steps and laborious procedures [2]. The target compounds can be obtained in one pot with much fewer steps.

Therefore, multicomponent reactions have received significant interest in research areas like medicinal chemistry and combinatorial chemistry to obtain heterocyclic scaffolds. In general, the multicomponent reactions are synthetic hub to produce novel cyclic compounds such as quinazolines, diazepines, naphthyridines, dihydropyridines, quinolines, pyrimidines, spiro-heterocycles and imidazole's which are widely recognized in clinical evaluation [3,4]. The multicomponent reaction techniques offers excellent yields, economy, shortened reaction time, environmentally safe, and serves as a useful mechanism for the creation of a library of novel chemical entities [5–10]. Further, to increase the reaction rate and product yield and decrease reaction time and economy, many researchers have been focusing on the utilisation of microwave techniques in multicomponent reactions.

Although there is considerable debate regarding how microwave irradiation (MI) can improve or affect the result of chemical reactions, microwave-assisted chemical reactions are increasingly widespread in the laboratory. Most of the discussion concentrated on whether the observed effects can always be explained by purely thermal Arrhenius-based phenomena (thermal microwave effects), emphasising the significance of the quick heating and high bulk reaction temperatures made possible by microwave dielectric heating in sealed reaction vessels.

The sustainable creation of chemical molecules is known as “green chemistry”. Green chemistry seeks to reduce the harmful impact that the production and manufacturing of diverse chemical compounds have on the environment. It aims to produce compounds with minimal harmful components by improving chemical synthesis efficiency and generating as little trash as possible. Utilising numerous powerful techniques, such as MI, is part of the future of green chemistry. The study of using MI effectively started in 1950 [11] and spread to organic synthesis procedures after 30 years. Chemical research and production have undergone a revolution thanks to the usage of microwave-assisted methods. This method allows the creation of even smaller chemicals and molecules in a concise amount of time.

1.1. What Are Microwaves?

A microwave is a type of electromagnetic energy that operates between the range of 300 to 300,000 megahertz. Only molecule rotation, not molecular structure, is impacted by this area of electromagnetic energy. For industrial, scientific, or medical purposes, 2450 MHz is chosen over the other three frequencies because it has the necessary penetration depth to interact with laboratory-scale samples and is close to power sources that can produce microwaves at this frequency. An electric and a magnetic field contribute to microwave energy, but only the electric field may be used to heat objects. Chemical synthesis does not typically include magnetic field interactions. The result of microwave absorption on the excitation of molecules is solely kinetic.

1.2. How Are the Reactants Heated by Microwaves?

Chemical synthesis is usually accomplished using conductive heating from an outside heat source. In order to reach the solvent and reactants, heat is first forced into the substance through the vessel's walls. Introducing energy into the system is slow and ineffective since it depends on the thermal conductivity of the numerous materials that must be pierced. Until enough time has passed to allow the container and contents to reach thermal equilibrium, the outside temperature of the vessel will be higher than that of the reaction mixture inside. Hours may pass throughout this process. The chemist's ability to regulate the reaction is likewise hampered by conductive heating. To lower the bulk solution temperature, the heat source must be physically removed, and cooling must be applied.

On the other hand, microwave heating is an entirely different approach. The molecules in the reaction mixture directly pair with the microwaves, rapidly raising the ambient temperature. The outcome is a quickly localised superheating of anything that will react to either dipole rotation or ionic conduction. These are the primary mechanisms for

transmitting energy from microwaves to heated substances. This is because the process is independent of the thermal conductivity of the container's materials.

Additionally, microwave heating allows for simple reaction control. "Instant on-instant off" is an excellent way to explain it. Latent heat is the only thing left over once the microwave radiation is switched off. Polar molecules seek to align themselves with the microwave's rapidly shifting electric field through an interaction known as dipole rotation. Energy is transferred due to the molecule's rotating motion to align itself with the field. The molecules' polarity and capacity to line up with the electric field impact this mechanism's ability to couple. Any polar species (solvent and substrate) present experience this method of energy transfer; the exact parameters that influence the dipole rotation coupling efficiency are numerous.

In organic synthesis, MI has gained popularity as a heating method primarily because of its quick reaction times, solventless processes, and occasionally greater yields. MI also reduces energy usage, making it perfect for procedures of optimisation. Furthermore, there is proof that MI can enhance the crucial characteristics of regio-selectivity and stereo-selectivity involved in synthesizing bioactive molecules. These fundamental characteristics of MI enable its use in green chemical processes.

Besides, the MI technique is contactless [12], substantial, and the best operational condition [13] to obtain products quickly through greenery way [14,15]. Naturally, compounds possessing permanent dipole moments (movable electric charge) are microwave active. Especially the polar molecules, which contain portable electric charges, are typically heated during the reaction process by MI, increasing their reaction rate several folds [16] in minimal time [17,18] and producing greater yields [19] with an easy workup process [4] via this user-friendly methodology [20]. The reactants accelerate their excitation processes in the presence of high-frequency electric fields by decreasing their activation energy [21,22] to yield products. For a fruitful reaction, the stereo- and regio-selectivity of products are highly desirable. The MI technique fulfils this criterion and is thus called the most efficient and eco-friendly modern technique [18,23,24]. The microwave setup is being employed in almost all fields of chemistry [25], with its high energy efficiency and excellent selectivity [26]. The organic heterocyclic reactions under MI produce better yields by increasing reaction kinetics [27] with minimal environmental impacts [20].

A few significant research areas in modern physics utilising the MI technique are nanoscale coatings [28], solid dispersions [29], comparing and scaling up dielectric processes [30], synthesis of γ -MnO₂ used in supercapacitors [31], n-type benzotriazole semiconductor material synthesis [32], tin-oxide synthesis for sensitised solar cells [33], and magnetic nanoparticle synthesis [34]. The MI method is also crucial in bioscience, pharmaceutical and medicinal fields like medicine applications [35], diagnostic pathology [36], extraction process of bioactive compounds [37], medicinal chemistry [20,38], and drug invention processes [39–41]. The emerging surface engineering technology also utilises MI as a significant protocol [42], material engineering [43], Ni-Al-Ti coatings [44], and surface fibrous decoration processes [45].

The MI has tremendous applications in various chemistry research areas such as, heterogeneous catalytic reactions [46], solid state chemistry [47], different heterocyclic organic synthesis involving O, N, and S [48], nitrogen containing heterocycles [49], heterocyclic synthetic reactions [50], solvent free heterocyclic synthesis processes [51], colloidal inorganic nanocrystal synthesis [52], bioactive six membered heterocycles synthesis [53], five membered nitrogen heterocycles synthesis [54], Knoevenagel condensation [55], indoles synthesis [56], triazoloquinazolinones and benzimidazoquinazolinones synthesis [57], extraction of volatile compounds [58], oxazoles and diastereoselective oxazolines [59], synthesis of quinazolines and quinazolinones [60], polymer synthesis [61], ferrocenyl chalcone synthesis [62], arylidene acetophenones synthesis [63], photo oxidation of sulfoxides [64], potential biological compounds [65], organic nanoparticle synthesis [66], synthesis of amino-quinazoline derivatives [67], coumarin-purine derivatives [68], various ring opening polymerisation reactions [69], nucleoside protide analogues synthesis [70], organic trans-

formations and synthesis [71], benzannulation reactions [72], heterocyclic phosphonate synthesis [73], azha heterocycle synthesis [74], one pot three component pyrazole synthesis [75], heterocyclic hydrazone synthesis [73], degradation reactions [76], heterogeneous catalysis [77,78], 1,2,3 triazolobenzodiazepinones synthesis [79], silica material synthesis [80], 2-aryl and 2,5-diarylthiophene derivatives preparation [81], decarboxylation of malonic acid derivatives [82], Pd-carbon catalysed reactions [83], and carbon supported-Co catalytic reactions [84].

The MI-assisted multi-component heterocyclic synthetic reactions are environmentally benign and vital to modern organic chemistry [85]. The MI protocol assists the reactants towards yielding the products in the presence of heterogeneous catalysts.

2. Applications of Microwave-Assisted Heterogeneous Catalysed Multi-Component Reactions

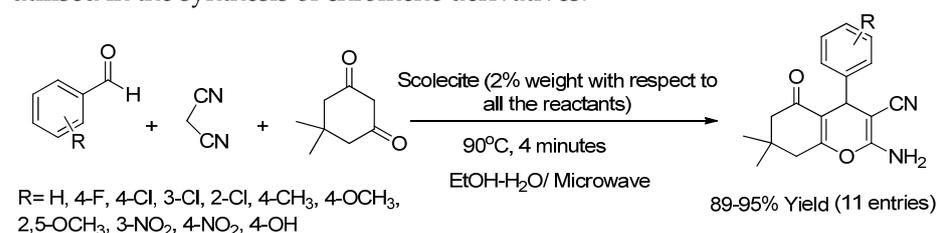
2.1. Synthesis of Nitrogen and Oxygen Containing Heterocyclic Compounds

Heterocyclic compounds can be prepared through one pot MI technique using heterogeneous catalysts and have emerged as a desirable replacement in contemporary organic chemistry.

2.1.1. Preparation of Chromenes

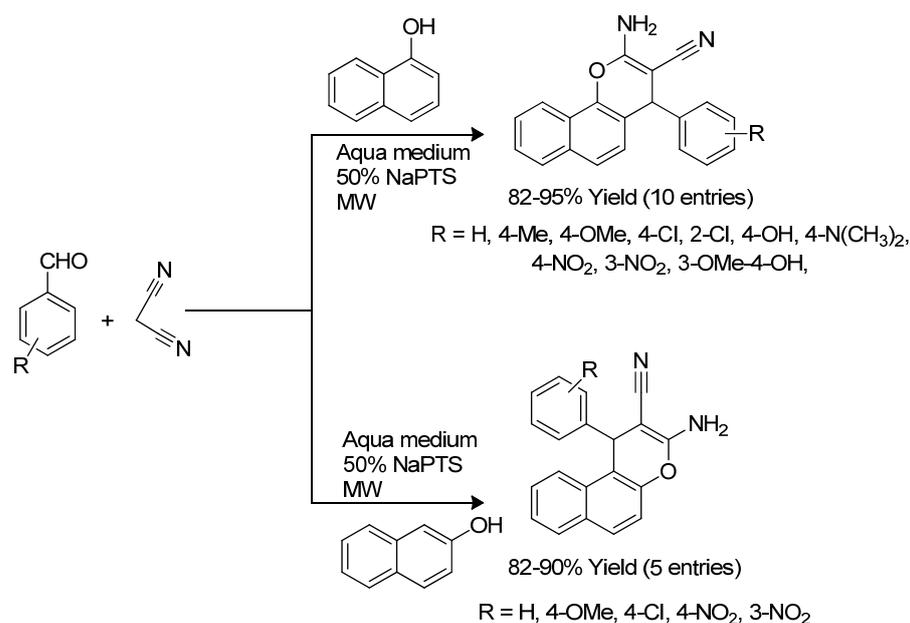
According to the most recent literature research, between five and particularly the six-membered heterocyclic compounds, chromene chemistry is very lucrative from a synthetic and pharmaceutical perspective due to its broad biological effects [86]. The chromenes possess potential biological characteristics like anticancer, food additives, antidiabetic activities, antifungal, anti-inflammatory, antimicrobial [87] and anticoagulants [88], applications in cosmetics, chemicals [89] and agrochemicals [90].

Bicyclic oxygen heterocycles containing a benzene fusion ring at a 5,6-positioned 4*H*-pyran ring system are called 4*H*-chromene. It has attracted researchers' attention as a valuable structural pattern for finding new drugs. Lambat synthesized 4*H*-chromene derivatives using scolecite as a heterogeneous catalyst under MI through the multi-component one pot method [88] with 95% yield at 90 °C (Scheme 1). This method offers effective and quick reaction times, simple reaction profiles, accessible workup, facile catalytic reusability, and outstanding yield without loss of catalytic nature (up to 3 cycles) are all mainly attributable to the current synthetic approach. The readily abundant reactants have been utilised in the synthesis of chromene derivatives.



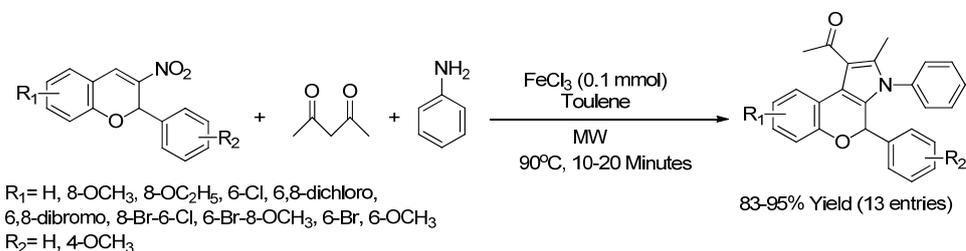
Scheme 1. Microwave-assisted synthesis of 4*H*-chromene derivatives using scolecite as a heterogeneous catalyst.

Through MI, Gaikwad and Kamble [91] reported a new method for preparing 2-amino-4*H*-chromenes in the aqueous hydrotropic medium. A renewable and sustainable energy conservation process is a synthetic methodology exposed to microwave radiation. Various derivatives of 2-amino-4*H*-chromenes were produced in good to outstanding yields by condensing a variety of aromatic aldehydes with malononitrile, α -naphthol, or β -naphthol in an aqueous hydrotropic media. This demonstrates an environmentally friendly process with straightforward experimental and workup steps. The main characteristics of this approach include a quick reaction time, lack of toxicity, reusability, low cost, low temperature (10–28 °C), and high product yield (82–94%) (Scheme 2). The catalyst can be used for five runs with a small change in product yield.



Scheme 2. Synthesis of 2-amino-4*H*-chromenes derivatives.

Baral et al. [86] designed a series of 2*H*-chromene-containing pyrrole derivatives to know the vital bioactive properties and to reduce the laboratory effort. An innovative and effective MI-assisted method for producing the 2*H*-chromene-fused pyrrole derivatives was described using a range of substituted 3-nitro-2*H*-chromene, acetylacetone, aniline, and FeCl₃ as a catalyst and generating excellent yields (83–95%) within 15 min (Scheme 3).

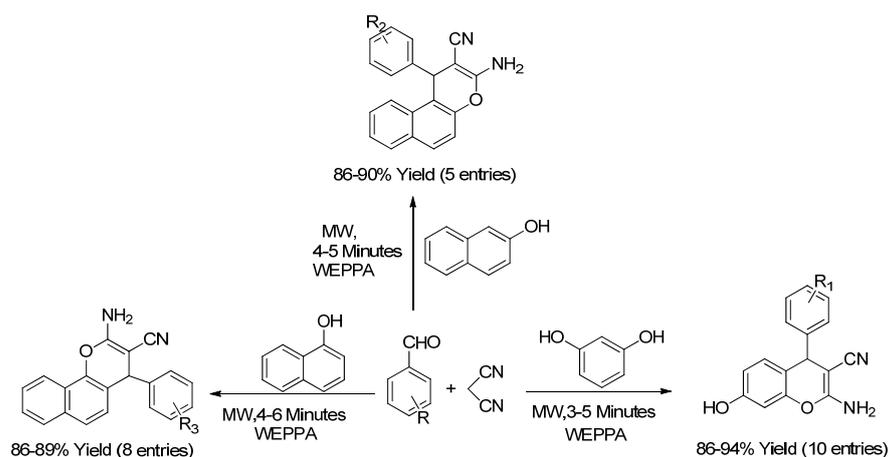


Scheme 3. Synthesis of 2*H*-chromene fused pyrrole derivatives.

Molaei et al. [92] have synthesized 4*H*-chromene derivatives via microwave method using nano-coconut shell-BF₃ as a new heterogeneous catalyst and aryl aldehydes, cyclic 1,3-diketone, and malononitrile as reactants. The authors concluded that the catalyst is used for three successive times without loss of its significant activity. A good yield is obtained at 90 °C for 4 min of reaction time under microwave conditions (Scheme 4). Prashant B. Hiremath et al. [93] have introduced a new technique for synthesizing substituted chromenes by using water extract of pomegranate peel ash (WEPPA) under MI. They produced 2-amino-4*H*-chromenes by condensation of substituted aryl aldehyde, malononitrile and resorcinol. The products were obtained in 45 min with an 85% yield (Scheme 5).

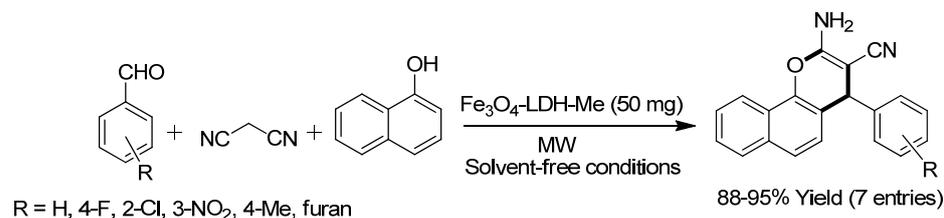


Scheme 4. Synthesis of 4*H*-chromenes catalysed by Nano-coconut shell-BF₃.



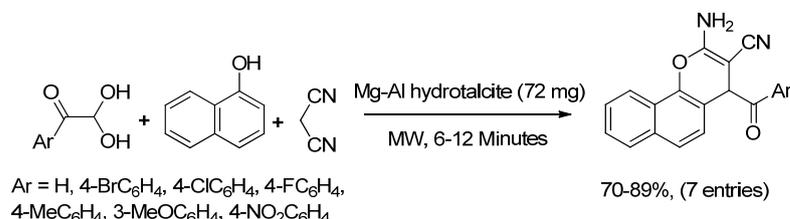
Scheme 5. Synthesis of 2-amino-4*H*-chromenes.

Nope E and co-workers [94] reported a new method for the synthesis of 4*H*-chromenes by using aromatic aldehydes, malononitrile, and naphthol derivatives in the presence of magnetic Fe_3O_4 -based hydrotalcites (50 mg) heterogeneous catalyst. The reaction was carried out in the presence of microwaves, and the 88–95% product yield is found at 80 °C (Scheme 6). They finally concluded that, the catalyst with 50 mg can effectively catalyze the conversion of the Michael adduct into the 4*H*-chromene product and can be recycled up to five times without loss of its catalytic activity.



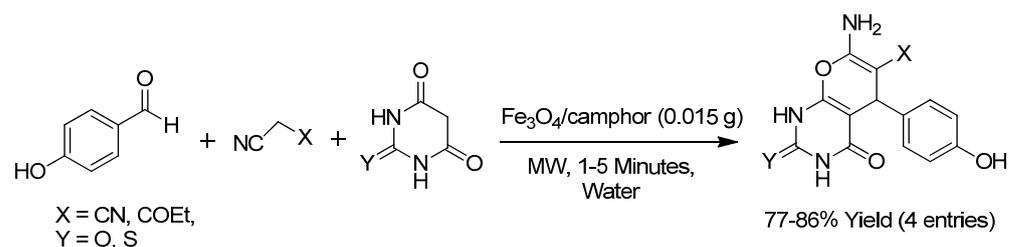
Scheme 6. Synthesis of 4*H*-chromene.

Poursattar et al. [95] constructed 2-amino-4-aryl-4*H*-benzo[*h*]chromene-3-carbonitriles through a multicomponent reaction through MI method in the presence of Mg-Al hydrotalcite catalyst. The process was carried out under solvent-free conditions offering 70–89% yield within 6 to 12 min (Scheme 7). The advantages of this approach include quicker reaction durations, milder reaction conditions, simple workup, good to outstanding yields, an abundance of raw materials, and its usefulness in the synthesis of various heterocyclic compounds.



Scheme 7. Synthesis of 2-amino-4-aryl-4*H*-benzo[*h*]chromene-3-carbonitriles.

Dwi Febriantini and group [96] introduced a new method to synthesize 2-amino-4*H*-chromenes using 4-hydroxy benzaldehyde, malononitrile/ethyl cyanoacetate and barbituric acid/thiobarbituric acid as reactants and Fe_3O_4 /camphor as a catalyst under microwave condition (Scheme 8).

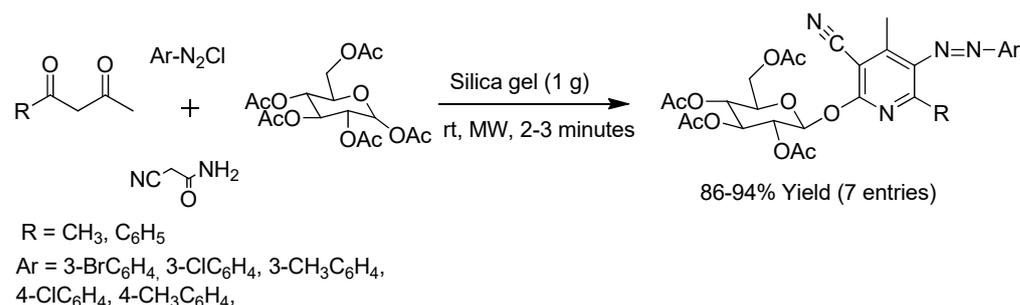


Scheme 8. Synthesis of 2-amino-4H-chromens.

2.1.2. Preparation of Pyridines

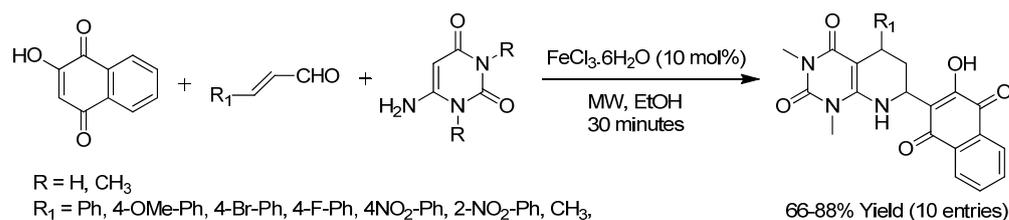
The pyridines have a wide range of applications, frequently being used in fluorescence sensors, laser dyes and molecular switches [97], confocal microscopy [98], medical applications [99,100], anti-inflammatory [101], DNA and RNA structural constituents [102–104], antioxidants [105], antimicrobial [106,107], fungicidal [108] to treat hepatitis B [109] and C [110], arterial thrombosis [111], Alzheimer's [112], tumours [113], and kidney diseases [114].

Hany A. Eldeab [115] designed pyridine nucleosides under microwave and solvent-free conditions using an efficient and environmentally friendly solid silica gel catalyst. The products were reported as 94% in 3 min (Scheme 9). The method emphasised the recyclable silica gel catalyst.

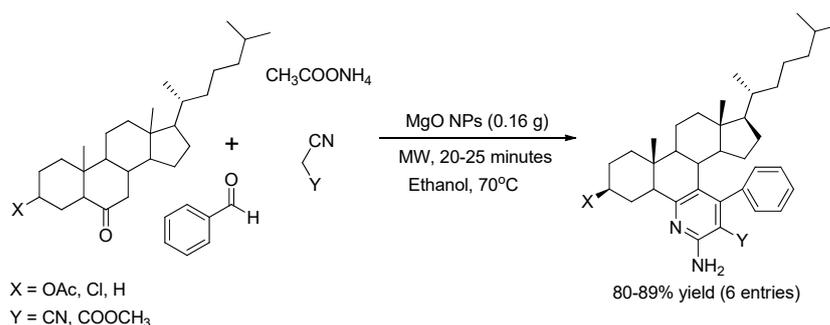


Scheme 9. Synthesis of pyridine nucleosides.

Pooja Kumari and co workers [116] have put effort to synthesize pyrimidine derivatives by utilizing cyclic 1,3-diketones, α, β unsaturated aldehydes and 6-aminouracils in the presence of 10 mol% FeCl_3 catalyst under the microwave conditions (Scheme 10). Their procedure comprises less reaction time, moderate-to-good yields with cheap starting materials. The MnO_2 catalyst produced desired amounts of products. Ansari et al. [117] synthesized steroidal pyridines by microwave one-pot multi-component reaction using MgO NPs as a heterogeneous catalyst. The method was successful at 70 °C in 20 min with an 89% yield. The MgO NPs heterogeneous catalyst was reported as an alternate and sustainable catalyst for synthesizing substituted steroidal pyridines (Scheme 11). The catalyst was reused for five subsequent cycles without major loss of its catalytic nature.



Scheme 10. Preparation of pyrido[2,3-d] pyrimidines.

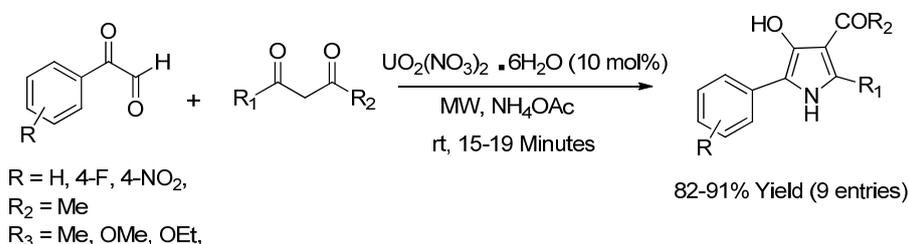


Scheme 11. Synthesis of poly-substituted steroidal pyridines.

2.1.3. Preparation of Pyrroles

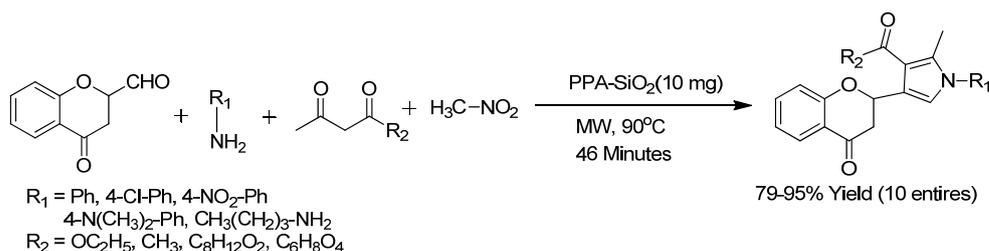
The 5-membered heterocyclic compounds of nitrogen include pyrroles, pyrrolidines, oxazoles, indoles, and pyrazoles, where pyrrole is discovered to be the most significant of them. Any organic molecule in the heterocyclic group with a ring structure of four carbon atoms and one nitrogen atom is called a pyrrole. Pyrroles have widespread usage in pharmaceutical sectors and are essential targets in chemical synthesis [118]. In bio-applications, these pyrroles demonstrate fungicidal, antibiotic, antipsychotic, anxiolytic, beta-adrenergic antagonist, anti-inflammatory [119], anticancer, antiprotozoal, anti-malarial, anti-tumour agents [120], and antimicrobial activities [121]. It is also used as a corrosion inhibitor in polymer chemistry and an organic conducting inhibitor in many engineering applications.

Venkatesan et al. [122] have synthesized pyrrole derivatives catalysed by uranyl nitrate hexahydrate and obtained 85% yield in 15 min at room temperature (Scheme 12). The method offers good product yield. The scheme was tried in many solvents, and excellent results were obtained in the presence of ammonium acetate.



Scheme 12. Synthesis of functionalised pyrrole derivatives.

Sumit kumar and group [123] have introduced a new technique to synthesize tetrasubstituted-1H-pyrrol derivatives catalyzed by heterogeneous reusable silica gel supported polyphosphoric acid (PPA/SiO₂) under microwave-irradiation (Scheme 13). Authors concluded that the methodology was produced excellent yields with inexpensive catalyst in shorter reaction time. The catalyst was studied for 5 successive cycles where more than 80% product yield was noticed.

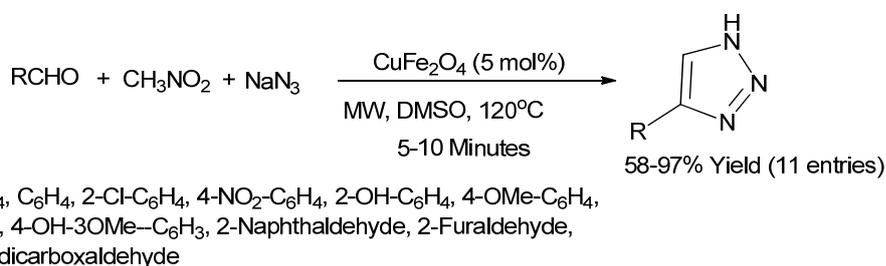


Scheme 13. Synthesis of tetrasubstituted-1H-pyrrol derivatives.

2.1.4. Preparation of Triazoles

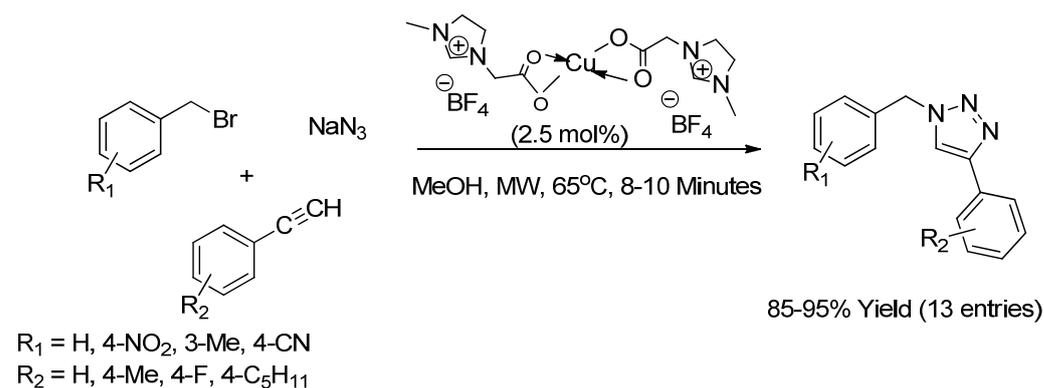
Triazoles have a five-membered heterocyclic ring with two carbon and three nitrogen atoms as their primary structural component. The triazoles are used as antimicrobial, antiviral, anti-tubercular, anticancer, anticonvulsant, analgesic, antioxidant, anti-inflammatory, and antidepressant activities, antifungal agents [124] and drug candidates for various microorganism-causing diseases [125], preferred overazole drugs for the remedy of some fungal infections [126], possess lead structures to design COX-1/COX-2 inhibitors [127], and forms hydrogen bond in various chemical transformations [128].

Bhuyan et al. [129] developed a new procedure to synthesize 4-aryl-1*H*-1,2,3-triazoles using aromatic aldehydes, sodium azide and nitromethane. The reaction was carried out under MI with an active CuFe_2O_4 catalyst by a short reaction time, i.e., 5–10 min, with satisfactory high product yields (60–97%) (Scheme 14). The activity of the reused catalyst was evaluated by the authors and the results were reported with a slight decrease in the catalyst activity up to the sixth cycle.



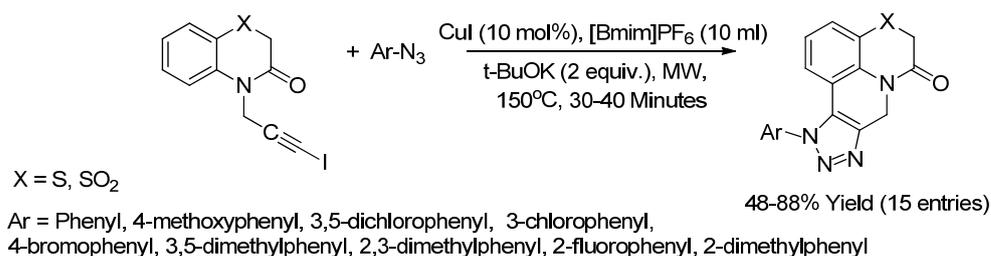
Scheme 14. Synthesis of 4-aryl-1*H*-1,2,3- triazoles.

Saikia and his team [130] created an ionic liquid-based Cu(II) heterogeneous catalyst for 1,2,3-triazole derivative synthesis. This ionic liquid-based Cu(II) catalyst was created by combining $\text{Cu}(\text{OAc})_2$ with 1-(1-carboxymethyl)-3-methylimidazolium tetrafluoroborate under MI in a water medium for 20 min. The catalyst was then converted to Cu(I) using a reducing agent. This catalyst was used for 1,2,3-triazoles derivative from benzyl bromide, NaN_3 and alkynes in methanol for 3 h, with outstanding yields up to 3 cycles (Scheme 15).



Scheme 15. Synthesis of 1,4-disubstituted 1,2,3-triazoles.

Narsimha et al. [131] designed a one-pot microwave-assisted synthesis of 1,2,3-triazole derivatives from 1-iodoalkynes with various aryl azides using CuI catalysts with good yields (Scheme 16). The reactions were completed in <35 min with a simple operating methodology to give the desired product in good result.

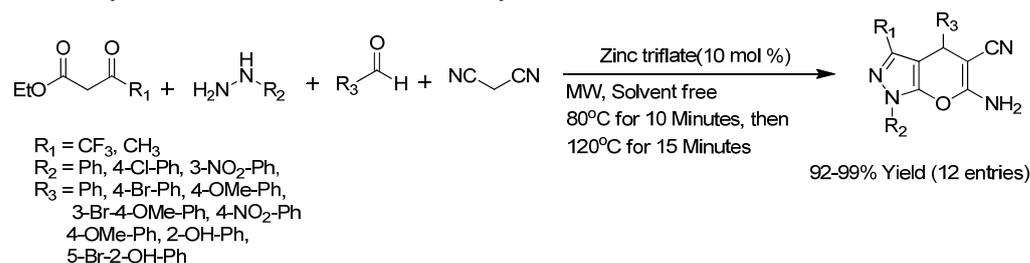


Scheme 16. Synthesis of fused 1,2,3-triazoles.

2.1.5. Preparation of Pyrazoles

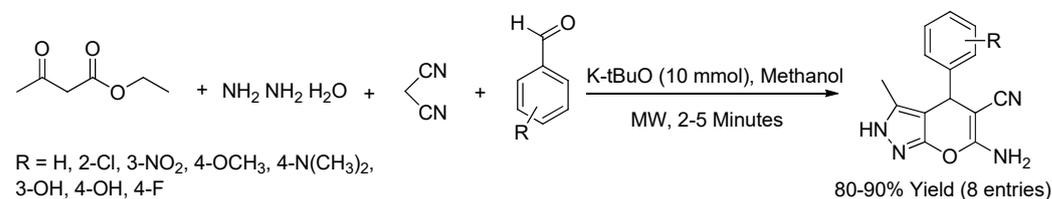
It is a heterocycle with a 5-membered ring of three carbon atoms and two nitrogen atoms next to each other. The pyrazoles are good antitumor agents [132], analgesic, antipyretics, anti-inflammatory, antioxidant and antiviral agents [133–135], tranquilisers, chemosensors [136], antimicrobial scaffolds [137], and neuroprotective and oestrogen receptor [138], the antagonist of the human CCK(1) receptor [139], pyrazole derivatives are used in agrochemical, pharmaceutical, and chemical industries [134,140].

Parikh et al. [141] have synthesized 6-amino-1,4-dihydropyrano[2,3-*c*]-pyrazole-5-carbonitriles by microwave method using zinc triflates as heterogeneous catalyst obtained 92–99% products in 15 min (Scheme 17). The mechanism involves recyclable catalysts over several cycles without the loss of efficiency.



Scheme 17. Preparation of 6-amino-1,4-dihydropyrano[2,3-*c*]-pyrazole-5-carbonitriles.

Yellappa et al. [142] introduced a new method for synthesizing 4*H*-pyrano[2,3-*c*] pyrazoles under microwave conditions using a potassium tertiary butoxide catalyst. The products were obtained in 4–5 min. with a 90% yield (Scheme 18). Most of the synthesized compounds exhibited good activity against Gram-positive (MIC range 7.8125 to 62.25 µg/mL) and Gram-negative bacteria (MIC range 7.8125 to 31.125 µg/mL), emphasizing the biological applications of synthesized compounds.



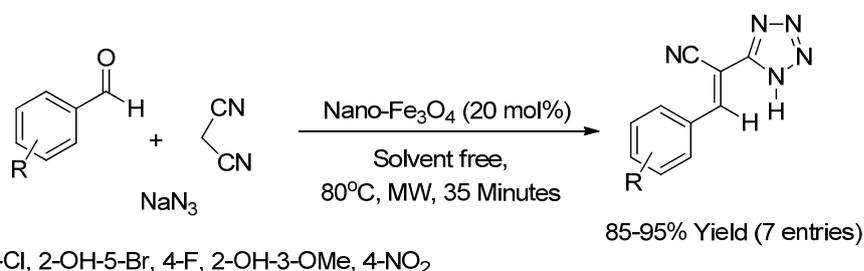
Scheme 18. Synthesis of 4*H*-pyrano[2,3-*c*] pyrazoles.

2.1.6. Preparation of Tetrazoles

Tetrazoles are a subclass of synthetic organic heterocyclic compounds, including five nitrogen and one carbon atom arranged in a ring. Tetrazole is also the name for the parent molecule, CH₂N₄, from which three isomers are possible. The wide applications of tetrazoles include antihypertensive, anti-allergic, antibiotic and anticonvulsants [143,144], investigated as rocket propellant components based on their high energy properties [145–148].

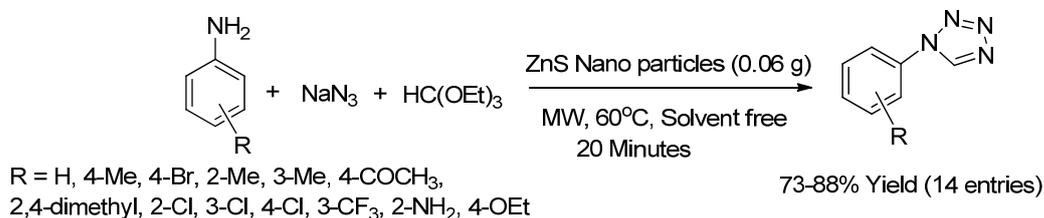
Akbarzadeh et al. [149] described the synthesis of 5-substituted-1*H*-tetrazoles via multi-component domino Knoevenagel condensation using microwave eco-friendly method

using Fe_3O_4 magnetic nanoparticles as heterogeneous catalyst in short reaction timings (35 min) with excellent yield (Scheme 19). The approach is practical because of the catalyst's low cost and nontoxicity, the removal of volatile and poisonous solvents, the rapid reaction time, the excellent yield, the straightforward methodology, and the simple workup. The magnetic facilitated the easy recovery of the catalyst, and the regenerated was used for at least five consecutive runs with minimal loss of activity.



Scheme 19. Synthesis of 5-substituted-1*H*-tetrazoles.

Naeimi et al. [150] synthesized 1-substituted-1*H*-tetrazoles using zinc sulfide nanoparticles as heterogeneous catalysts under solvent-free conditions, obtaining 88% yield in 20 min at 60 °C (Scheme 20). According to the experimental findings, various 1-substituted tetrazoles were produced in good yields under MI by ZnS NPs acting as a powerful and recyclable heterogeneous catalyst. This procedure has benefits over other published techniques, including a solid recyclable catalyst, solvent-free conditions, and a greener process. The catalyst was recovered and reused several times. The authors were concluded that, the catalyst can be reused for seven times with a minimal loss of its activity.

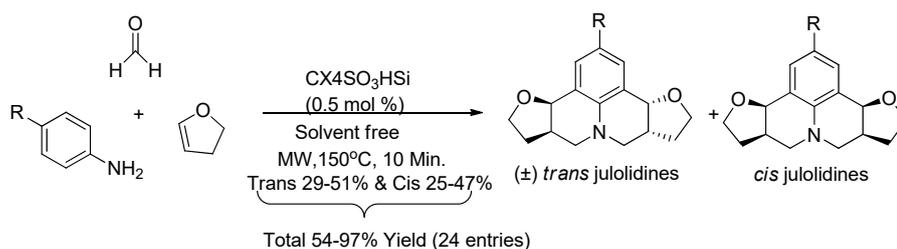


Scheme 20. Synthesis of 1-substituted-1*H*-tetrazoles using ZnS Nano particles under MI.

2.1.7. Synthesis of Trans and Cis Julolidines

Julolidine is a heterocyclic aromatic organic compound with the formula $\text{C}_{12}\text{H}_{15}\text{N}$. Julolidines have been employed in producing dye-sensitised solar cells, photoconductive substances, fluorescent detectors for bio imaging, and for identifying ions and volatile compounds in environmental and biological materials.

The Walysson Ferreira de Paiva and group [151] have described using a sol-gel approach to immobilise calixarene as an effective heterogeneous catalyst $\text{CX}_4\text{SO}_3\text{HSi}$ for multi-component Povarov reactions (Scheme 21). The catalytic activity of the $\text{CX}_4\text{SO}_3\text{HSi}(n)$ for sustainable and greener production of julolidines was investigated. Notably, the Povarov solvent-free reaction may be catalysed by the catalyst with just 0.5 mol% of the catalyst required under microwave assistance. Additionally, this methodology enables the synthesis of two C-N bonds and four additional C-C bonds in a single step. This material is the first silica support, calix[4]arene, that serves as a heterogeneous catalyst for the multi-component synthesis of julolidines. The catalyst was reused and recyclable for five successive runs without loss in its activity.



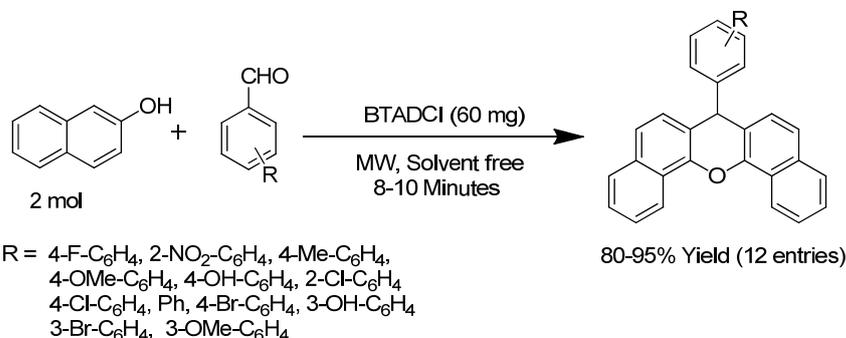
R = 4-Br, 4-Cl, 4-F, 4-I, H, 4-CO₂Bu,
4-C(CH₃)₃, 4-CH₃, 4-CN, 4-CF₃, 4-Ph, 4-OPh

Scheme 21. Synthesis of *trans* and *cis* julolidines using silica-supported calix[4]arene as heterogeneous catalyst under MI.

2.1.8. Synthesis of Xanthenes

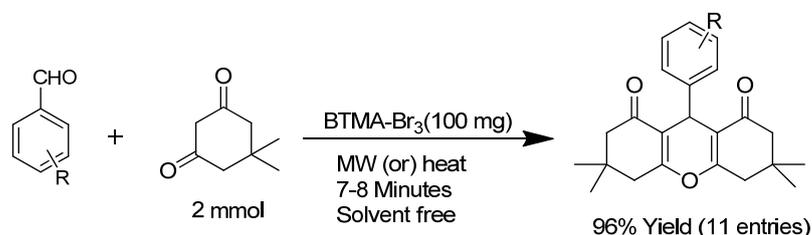
Xanthenes encompass an important class of heterocyclic synthons because of the inherent reactivity of the pyran ring with many industrial, biological, and pharmaceutical applications [152–154]. These include antimalarials, anti-bacterial, antivirals, anti-depressants, anti-inflammatories, as well as in laser technologies [155,156].

Pagadala et al. in 2018 reported [157] for the synthesis of 14-aryl-14*H*-dibenzo [a, j] xanthenes using aromatic aldehydes and β -naphthol with BTADCI (Benzyltrimethylammonium dichloriodate) as a Lewis acid catalyst in MI under solvent free conditions (Scheme 22). They established a practice with more advantages like, good yields and at a low cost of the catalyst. Authors also reported in 2020 that 1,8-dioxo-octahydroxanthenes [158] can be prepared through one pot synthesis under MW eco-friendly conditions using BTMA-Br₃ (Benzyltrimethylammonium tribromide) as a metal-free Lewis acid catalyst and benzaldehyde & dimedone as reactants (Scheme 23). The products were obtained in 7-8 min with a good yield.



R = 4-F-C₆H₄, 2-NO₂-C₆H₄, 4-Me-C₆H₄,
4-OMe-C₆H₄, 4-OH-C₆H₄, 2-Cl-C₆H₄,
4-Cl-C₆H₄, Ph, 4-Br-C₆H₄, 3-OH-C₆H₄,
3-Br-C₆H₄, 3-OMe-C₆H₄

Scheme 22. Synthesis of 14-aryl-14*H*-dibenzo [a, j] xanthenes.



R = H, 2-Cl, 4-CN, 3,4,5 (MeO)₃,
4-NO₂, 3-MeO, 4-Br, 3-OH,
4-OH, 4-MeO, 3-NO₂

Scheme 23. Synthesis 1,8-dioxo-octahydroxanthenes.

Corroborating the above findings, we predict that the heterogeneous catalyst in microwave-assisted reactions will boost the reaction rate in a minimal time and produce products with excellent yields due to the decrease in activation energy of reactants on

physical contact with the catalyst under MI. First, the heterogeneous catalyst might provide a large surface area for the adsorption of reactants over it. Then this adsorption encourages the collision between the reactants by decreasing their activation energy. Besides, MI quickly stimulates vast collisions between the reactants, increasing the product yield with greater thermodynamic feasibility.

3. Conclusions

The microwave technique has numerous applications in most of the ongoing research areas. This method predominates the other instrumental practices because of its easy workup and more efficient processes. The present research scenario uses greenery and eco-friendly instrumental setups to generate novel reactions. This microwave instrument's availability and arrangement motivate most research scholars to develop new reaction mechanisms. It also reinforces many of the entrepreneurs in terms of the abundance of microwave machinery. Generally, instrumental reactions have more advantages over traditional reflux synthetic methodologies because of their accuracy, lower reaction time, and excellent yields. The instrumental methods reduce workforce and risk parameters. The reactions with acceptable results are vital. The catalysts always decrease the reactants' energy barrier to come across it and form products. The kinetically and thermodynamically stable products are created when the reactants undergo fruitful collisions facilitated by the catalyst. The heterogeneous catalysts are easy to recover after the successful completion of a reaction, and many such materials are recyclable without any efficiency loss. The combination of microwave and heterogeneous catalysts facilitates reactions with more excellent selectivity towards product formation in less time.

The vital parameters for the most efficient methods are cost effectiveness, eco- and user-friendliness with high product selectivity and yield. The techniques involving MI and heterogeneous catalyst satisfy all these characteristics. Hence, microwave-assisted heterogeneous catalytic reactions are the backbone of understanding the eco-friendly principles of a chemical reaction.

Finally, the microwave-assisted multi-component reactions offer simple access to constructing biologically active heterocycles ranging from modestly fused rings to complex structures. This proficient technique perfectly energises the reactant molecules by microwave heating by decreasing activation energy and improving the selectivity and yield. These user-friendly, one-pot, and eco-friendly protocols using different heterogeneous catalysts offer various inventions for designing and enhancing the generation of novel heterocycles. While microwaves help speed up the process, overcome complex purification procedures, and minimise the time duration from hours to minutes. There is much scope for investigation in this area to design efficient catalysts and protocols. As a result, this review might help the researchers in the direction and inspire them to create new chemical entities through microwave-assisted heterogeneous catalysis of the multi-component reactions.

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