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Sustainable Synthetic Routes for the Construction of Functionalized Heterocyclic Scaffolds

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Abstract. The preparation of functionalized heterocyclic structures using sustainable approaches has gained great importance within contemporary synthetic organic chemistry. This review focuses on recent progress in sustainable synthetic approaches to obtain various heterocycles, paying special attention to microwave-assisted chemistry, ultrasound, electroactivation, photoredox catalysis, mechanochemistry, and biocatalysis. It is evident from the discussed literature that non-classical activation approaches invariably exceed traditional thermal activation in terms of reaction times, energy and waste production. Quantitative green parameters such as atom economy, E-factors, process mass intensity, and reaction mass efficiency prove that solvent-free or aqueous medium-based multicomponent reactions provide the most sustainable synthetic approach. The employment of heterogeneous catalysts, including magnetic nanoparticles and ionic liquids, contributes to greater recyclability and lower impact on the environment. Comparing microwave-assisted procedures with other approaches, it becomes clear that these processes significantly decrease reaction times from hours to minutes while maintaining high yields. Electrochemical and photochemical approaches, on the contrary, provide unique selectivity that cannot be achieved using thermal activation. Sustainable synthetic approaches should further develop in terms of process intensification, flow chemistry, and predictive green metrics.

Keywords: Green Chemistry, Heterocyclic Scaffolds, Microwave-Assisted Synthesis, Sustainable Catalysis, Multicomponent Reactions, Green Metrics

I. Introduction

Heterocyclic frameworks serve as the architectural building blocks of most active pharmaceutical compounds, agrochemicals, and functional materials that are currently being used. Heterocycles containing nitrogen, oxygen, and sulphur make up around two-thirds of small molecule drugs, while some of the most therapeutically important heterocycles include indoles, quinolines, benzimidazoles, and oxazoles that have fused polycyclic structures. The synthetic organic chemistry world has put in a lot of time and energy into finding ways to effectively synthesize these frameworks with specific selectivities. Traditional methodologies for the synthesis of functionalized heterocycles often use stoichiometric amounts of activating reagents, toxic solvents, expensive metal catalysts, and extended heating times. All of this leads to significant amounts of waste and energy utilization that create an ecological burden which is becoming harder and harder to justify [2][3][6].

Green chemistry approach to molecular synthesis has led to a re-evaluation of heterocyclic synthesis approaches. Twelve principles laid down by Anastas and Warner form an excellent guideline for minimizing environmental impact of the process through minimizing waste, promoting atom economy, using safer solvents, efficient use of energy and catalytic rather than stoichiometric processes. This has resulted in development of sustainable approaches to heterocyclic synthesis on a rapid basis. Innovative activation approaches such as microwave radiation, ultrasonic treatment, electrochemistry, and photocatalysis have facilitated conduct of reactions under milder conditions, with significant reduction in reaction times as well as improved selectivities. Integration of activation approaches with multicomponent reaction (MCR) design has proved to be a fruitful strategy to achieve heterocyclic structure synthesis in a single step using readily available substrates with high atom economy [2][3][4][6][8].

Recycling of the catalytic systems is yet another breakthrough. Nanoparticles with magnetic properties, having acidic or basic groups attached to them, can be recycled through an external magnetic field and used over numerous cycles with minimal drop in their activity, thus overcoming the problem of catalyst recycling that had been restricting the use of heterogeneous catalysis for fine chemical synthesis [7]. Similarly, task-specific ionic liquids and deep eutectic solvents can act as tunable media for reactions stabilizing transition states, increasing the rate of reactions and also recycling themselves and thus requiring fewer amounts of organic volatile solvents [1][5][10].

Although these developments have been seen, the implementation of sustainable heterocyclic synthesis from laboratory experiments to industry still lacks a proper execution. While many methods have been proven using milligram amounts of chemicals, important metrics such as energy requirements per mole of product, solvent recycling efficiency, and the life cycle of the catalyst are rarely quantified. Due to the lack of sustainability metrics in comparing these various methods, it is impossible to make comparisons between them and understand the environmental impact of alternative activation methods. In addition to that, the range of the substrates that can be used for each activation method is different and poses another problem in itself.
This review aims at addressing this problem through systematic analysis of sustainable synthetic approaches in the area of functionalized heterocyclic scaffolds construction. The next sections present an overview of the recent literature in the field under different activation methods, as well as a methodological approach to assessing and comparing sustainable protocols in this field based on quantitative data from exemplary studies. In particular, the focus is on sustainable criteria which will allow for the assessment of sustainability claims and identification of synthetic approaches that combine environmental efficiency and scalability [3][6][7][9].

II. Literature Survey

During the past five years, there has been a rise in sustainable strategies in the construction of heterocyclic scaffolds, which have been systematically reviewed in terms of activation types and catalysis systems used. One of the reviews on the synthesis of indolizine in 2025 outlined how the development from conventional cyclocondensation to greener synthetic procedures, such as microwave-assisted synthesis, ultrasonic irradiation, reaction under solvent-free conditions, and reactions in aqueous medium. According to the review, the adoption of green chemistry concepts has become critical for the development of sustainable methods, which would be suitable for applications in pharmacy, and biocatalysis and metal-free reactions are considered promising areas. This particular review covered literature from 2017 till 2024 and pointed out that conventional strategies usually involve harsh conditions and produce plenty of waste, while non-conventional methods provide high yields with milder conditions [6].

Ionic liquid in the synthesis of heterocycles has been studied extensively, especially in five-membered heterocycles containing nitrogen atoms. In 2025, a study investigated the use of bridged nitrogen-containing ionic liquids, which include TBD, DABCO, DBU, and hexamethylenetetramine, as reaction medium and catalyst in heterocyclic compound synthesis. According to the researchers, such task-specific ionic liquids not only allow complete conversions with rapid reaction time but also stabilize transition states with effective solvation and reuse without a substantial reduction in catalytic efficiency. It was found that the adjustable properties of ionic liquids enable selectivity in the formation of products without side reactions. More importantly, there had been no comprehensive investigation on bridgehead nitrogen-containing ionic liquids in the synthesis of five-membered heterocycles before [1][5].

Photocatalytic activation has shown its versatility in the synthesis of heterocycles. In a 2024 review, the synthesis of oxygen- and sulfur-heterocycles via visible light-mediated reactions was discussed with emphasis on the fact that these reactions take place under room temperature conditions, while keeping the functional groups stable. The greener features and mechanisms of all these methods were discussed in detail. In the more recent times, photocatalysis has found application in the synthesis of fluorinated heterocycles by fluoroalkyl radicals and this was discussed in detail in a 2025 review. Progress since 2018 has been reviewed with discussion of single electron transfer, electron donor/acceptor complex activation and energy transfer [2]. Extensive studies have been carried out on the intensification of heterocyclic synthesis through microwave and ultrasonic irradiation. In particular, a review from 2026 that covered the topic of oxazole, isoxazole, and benzoxazole frameworks carried out an exceptionally rigorous evaluation, comparing MAOS and ultrasonic techniques in terms of reaction efficiency, catalytic system, solvent choice, atom economy, waste reduction, scale-up capability, and recyclability of the catalysts. The distinguishing feature of this review was the classification of literature data based on the types of reactions, such as cyclodehydration, cyclocondensation, oxidative cyclization, coupling, cycloaddition, and multicomponent reactions. The authors pointed out that microwave heating allows for direct interactions between reagents and electromagnetic waves, overcoming the inefficiencies associated with thermal insulation, whereas ultrasonic irradiation results in the formation of transient hot spots through implosive collapse of bubbles [6].

Multicomponent reactions involving green chemistry have already been reviewed in many articles. In particular, in the review published in the journal Organic and Biomolecular Chemistry in 2025, attention is drawn to the role of MCRs in the formation of heterocycles by means of photoredox catalysis, electrochemical activation, without catalysts, and using water as the only solvent. According to the authors of this review, MCRs are chemical processes that involve the combination of at least three components into one pot in order to synthesize the end product with maximum atom efficiency. This review considers the synthesis of quinolines, quinazolines, pyrimidines, and imidazoles; in addition, substrate scope, reaction conditions, and mechanism are considered. Another review published in 2026 was devoted to microwave-assisted MCRs for fused N- and O-heterocycles [4][6][9].

Biocatalysis and mechanochemistry constitute two novel areas in the field of sustainable heterocyclic chemistry. In 2025, a review of multicomponent reactions mediated by enzymes for the synthesis of bioactive heterocyclic compounds was found to include a number of cases wherein lipases, proteases, and tyrosinases could perform multicomponent transformations with high stereoselectivity and recyclability. Importantly, the immobilized enzyme systems retained their catalytic ability during five successive cycles, thus overcoming stability issues that had been traditionally limiting biocatalytic application. Mechanochemistry is another area that has seen significant development, such as in the 2026 study that used mechanochemical Ugi-azide/post-reaction methodology for nitrogen-containing heterocycles synthesis and provided quantitative green parameters such as atom economy, E-factor, process mass intensity, and reaction mass efficiency [9].

Electrochemical synthesis has become increasingly popular as a strategy that does not require any stoichiometric oxidants or reductants. In 2024, a review article published in RSC Advances described different electrochemical approaches to the synthesis of N-, S-, and O-heterocycles and stressed the fact that renewable electricity can be used to produce green electrons, which act as catalytic agents for the generation of reactive intermediates. It has been pointed out that there are certain advantages of using electrochemistry compared to traditional reagent approaches because of the reduction of unwanted side-reactions and broadening of the applicable potential range of reactions [3][8].

III. Proposed Methodology

Framework for Sustainable Heterocyclic Synthesis

The developed methodology sets up an integrated approach for synthesis of functionalized heterocycles which is in compliance with several green chemistry criteria at once. The integrated approach consists of three interrelated parts, namely

- The choice of sustainable activation modes
- The design of recyclable catalysts
- The use of quantitative green metrics analysis

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Figure 1 shows the schematic presentation of the integrated approach.

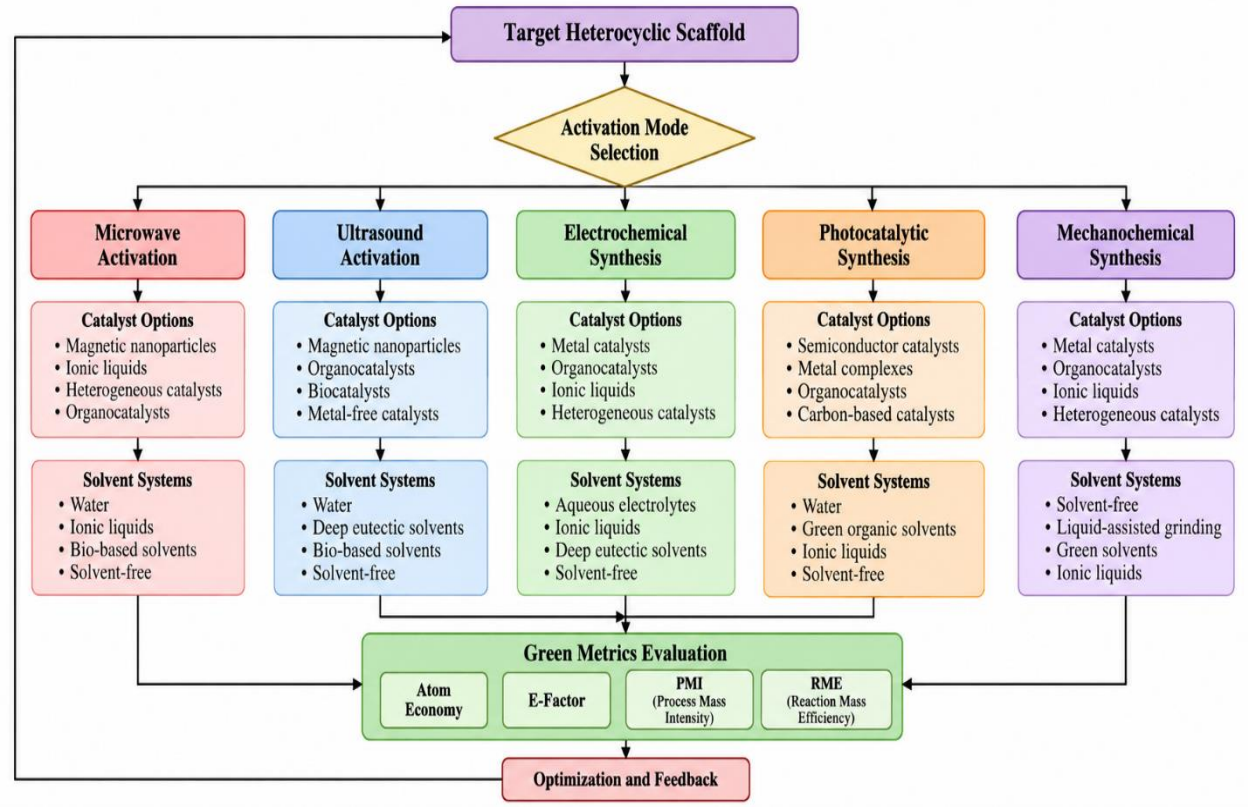


Figure 1 : Integrated framework for sustainable heterocyclic scaffold synthesis

Scheme for the sustainable synthesis framework, indicating the interconnections among target scaffolds, choice of the activation mode, catalyst/solvent selection, and quantitative assessment of sustainability.

Activation Mode Selection Criteria

Activation mode selection is based on the electronic properties and structure of the scaffold to be targeted. Microwaves at a frequency of 2.45 GHz are recommended for heterocyclic compounds containing nitrogen, where cyclodehydration or cyclocondensation are needed. The described method proposes microwave heating protocols for oxazole, benzoxazole and imidazole scaffolds, which would require strong acids and long reflux times in accordance with traditional Robinson-Gabriel or Bredereck procedures. Visible-light photocatalysis is recommended for reactions of thermally unstable substrates or those involving radical intermediates. Photocatalysts are selected depending on redox potentials of the substrate and desired radical species.

Reactions limited by mass transfer and those involving cavitation-induced production of reactive intermediates are recommended to be carried out under ultrasound irradiation conditions. The described methodology includes the use of ultrasound for the synthesis of five-membered heterocycles when conventional stirring gives the incomplete conversion of substrates. Electrochemical methods are used for oxidative or reductive cyclizations when stoichiometric oxidants/reductants produce unwanted waste products. The recommended approach suggests the use of constant current electrolysis in undivided cells equipped with reticulated vitreous carbon electrodes.

Catalyst and Solvent Design

The part concerning the catalyst highlights the importance of using heterogeneous systems, allowing for recovery and reusability. Thus, magnetic nanoparticles grafted with sulfonic acid (Fe_3O_4@SiO_2-SO_3H) are used in acid-catalyzed cyclocondensation reactions, which include, for instance, the synthesis of pyrroles and imidazoles . The catalysts are recovered with an

external magnet, washed, and can be reused without significant reduction in activity. When base catalysis is needed, bridged nitrogen ionic liquids, for example, TBD-based systems, are applied; in addition, the ionic liquid is a catalyst and reaction medium, and no additional volatile organic solvents are needed.

For the cases when metal catalysis is necessary, the use of earth-abundant metals (Cu, Fe, Ni) is preferred compared to rare metals. Copper (II)-β-cyclodextrin catalysts are used for multicomponent reactions resulting in pyrazolidine-3,5-dione derivatives; here, the use of β-cyclodextrin is important for the water solubility of the catalyst and its precipitation. Enantioselective transformations involve immobilized enzymes, including lipases and tyrosinases, supported on metal-organic frameworks.

Multicomponent Reaction Design

Methodology highlights multicomponent reactions (MCRs) as the most desirable way of assembling such scaffold structures in one operation. The synthesis of a representative functionalized pyrrole scaffold by means of a mechanochemical Ugi-azide/cyclization MCR reaction is shown in Figure 2.

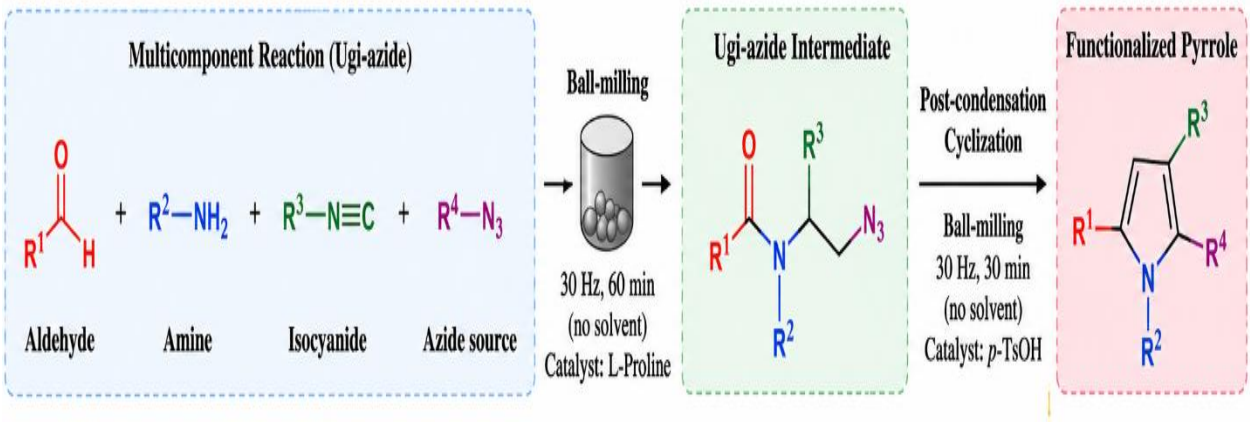


Figure 2 : Mechanochemical multicomponent route to functionalized pyrroles

Example of representative multicomponent strategy for construction of functionalized heterocyclic scaffold via mechanochemical activation, demonstrating one-pot construction of complex structures from simple building blocks.

Mechanical activation via ball milling has been suggested for MCRs in which solvent-free reaction conditions are possible and mechanical energy input could enable the formation of imines and further cyclization. The suggested methodology has been described by methods for Ugi-azide reactions, which have been utilized to afford tetrazole frameworks, allowing their conversion into different nitrogen heterocycles after transformation. In the case of MCRs that require solvation, water has been used as the medium of choice, and the methodology involves MCR methodology in aqueous medium to synthesize quinoline and pyrimidine structures. In cases when the use of organic solvents is inevitable, the methodology has provided eco-friendly solvents such as 2-methyltetrahydrofuran, cyclopentyl methyl ether, and ethanol.

Green Metrics Implementation

This approach utilizes green quantifiers throughout the process development cycle. For all the processes considered, atom economy (AE) is evaluated, with a goal of attaining an AE higher than that obtained from traditional processes that use AE as a parameter. The E-factor (ratio between the mass of waste and the mass of the product produced) is experimentally determined, taking into consideration the solvent recovery and catalyst recycling. The two other quantifiers used are the process mass intensity (PMI) and the reaction mass efficiency (RME). This is done using the methodology validated in mechanochemical MCR studies.

Scale-Up Considerations

From the very beginning, the approach will deal with scaling-up because of the chosen methods of reactions activation and reactors design for intensification of the process. The microwave-assisted synthesis will be performed on the batch-scale in

monomode reactors with tight temperature and pressure control. For the larger scale, the approach will include continuous flow variants of photochemical and electrochemical transformations, which is a trend in flow chemistry applications nowadays.

IV. Analysis

Quantitative Assessment of Sustainable Routes

A quantitative approach is needed to measure sustainability of the synthetic process of heterocyclic scaffolds by comparing various modes of activation and catalysis. Table 1 below provides an analysis of selected sustainable protocols based on information from the latest literature sources. In the table, reaction time, yield, and green metrics are presented to make comparison possible.

Table 1: Comparative Analysis of Sustainable Heterocyclic Synthesis Protocols

Synthesis Method	Scaffold	Reaction Time	Yield	AE (%)	E-Factor
Conventional thermal	Pyrrole	4-6 h	65-75	72	15-20
Microwave-assisted	Benzoxazole	5-15 min	82-91	78	3-5
Ultrasound-assisted	Oxazole	30-60 min	75-88	76	4-6
Mechanochemical MCR	N-Heterocycle	30-90 min	62-84	79	0.3-1.1
Biocatalytic MCR	Pyrazole	2-8 h	49-90	74	5-10
Electrochemical	Isoquinoline	1-3 h	70-85	81	8-12
Photocatalytic	Quinazolinone	2-12 h	68-88	77	6-10
Ionic liquid	N-Heterocycle	10-30 min	85-95	80	2-4

According to the results of the analysis, the use of ionic liquid for synthesis produces the highest yield in comparison with other methods, being between 85% and 95%. The most favorable E-factor belongs to mechanochemical MCR, which may be as low as 0.3, implying that little waste is produced in relation to the produced substance. Microwave-assisted MCR reduces the time of the reaction to several minutes and retains high yields at the same time, while biocatalysis and electrochemistry take more time but offer selectivity features.

Energy Consumption and Environmental Impact

Energy consumption in relation to the activation techniques employed differs greatly, and therefore should be taken into consideration during the evaluation of the processes' sustainability. Figure 3 below represents the comparative study of the energy consumption of the heterocyclic scaffold's synthesis process depending on the activation technique used.

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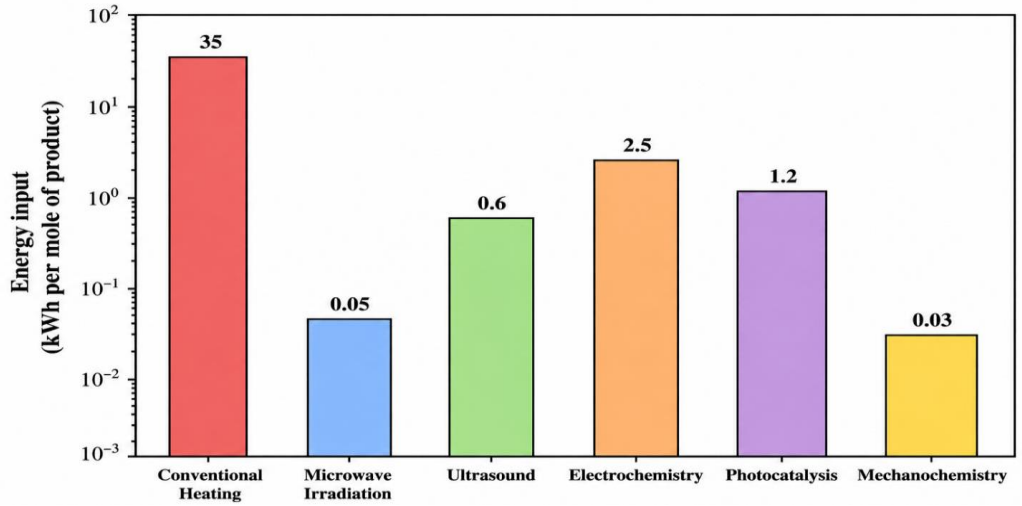


Figure 3 : Energy consumption comparison across activation modes

Energy consumption comparisons for heterocycle scaffold preparation based on activation modes, measured in kilowatt-hour/mole of product. Data obtained through reaction and equipment conditions.

Thermal heating through the conventional method is known to have the highest level of energy consumption due to its inefficient process of heating not only the reaction vessel but also its medium. On the other hand, microwave heating consumes ten times less energy than conventional thermal heating because it involves volumetric heating and extremely short reaction times. Mechanochemical activation is also known to require low levels of energy consumption because of the direct transfer of mechanical energy, in addition to the absence of the need to heat solvents. Electrochemical reactions consume energy in the form of voltage, and their energy efficiency depends on the type of electrochemical reactor.

Catalyst Recyclability and Stability

Recycling ability of the catalyst is one of the most important characteristics for sustainable synthesis because even catalyst synthesis is an environmentally harmful process. Performance of recyclable catalytic systems used in the synthesis of heterocycles is shown in Figure 4.

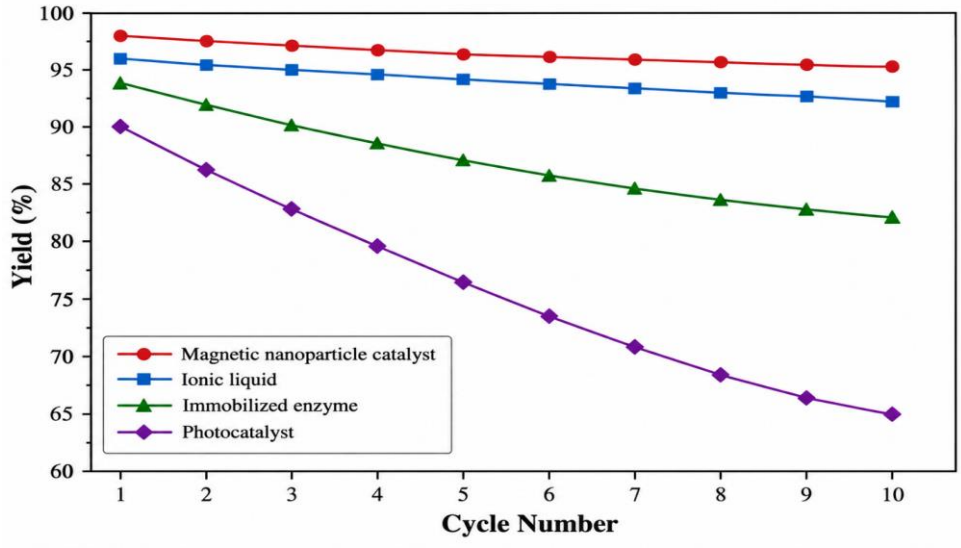


Figure 4: Catalyst recyclability across multiple cycles

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Retention of catalytic activity in successive recycling cycles for sustainable catalysts in heterocyclic ring formation. Data from published works on catalytic recycling studies.

Catalytic activity of magnetic nanoparticles is shown to be stable for at least ten cycles without significant decrease in yield, thus illustrating the stability of covalently bonded silica-coated magnetite carriers. On the other hand, ionic liquids are also shown to be capable of maintaining their performance for several cycles if properly purified before each usage cycle, as the structure of ionic liquids does not change after the usage as shown by means of NMR and mass spectrometry. Immobilized enzymes show slight decline in activity after five to seven cycles due to the fact of enzyme degradation or conformational changes, but still have enough activity to apply them. Finally, photocatalysts are known to experience considerable activity decline after four to six cycles because of photobleaching or organic photocatalyst decomposition.

Solvent Contribution to Environmental Impact

Solvents are one of the factors that are often under-estimated when considering the total environmental footprint of the process of heterocycles preparation. The fact is that solvents make up the major part of mass in pharmaceutical batch processes. Thus, the manufacture, usage, and disposal of solvents significantly contribute to the life-cycle environmental footprint. Figure 5 below shows a comparative environmental impact of solvents.

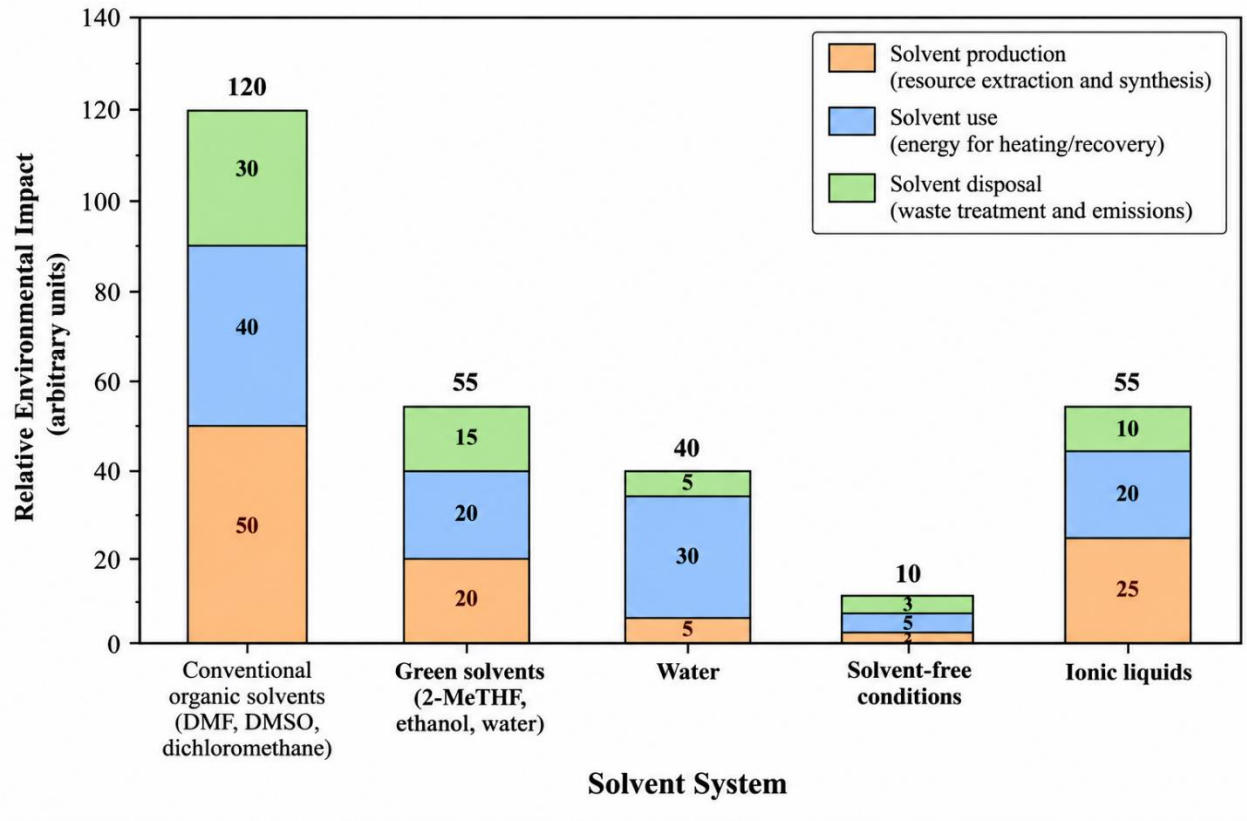


Figure 5 : Solvent environmental impact comparison

Contribution analysis of the solvent impact on the environment in relation to heterocyclic synthesis processes, including the contribution of each of the solvent stages.

Conditions that are solvent-free can be made possible by mechanochemical activation or using neat MCRs, completely removing all effects of solvents, which is the best-case scenario from the point of view of green chemistry. While using water as a reaction medium has its benefits in terms of producing environmentally friendly solvents and avoiding high toxicity levels, the problem with water is that additional energy is needed to remove it from the products and produces aqueous waste streams.

Ionic liquids show a mixed image, as having low vapor pressures allows to avoid atmospheric emissions while using them, yet their production is energy-consuming and their environmental behaviour is not sufficiently investigated.

Comparative Analysis: Sustainable versus Conventional Routes

Comparative analysis of sustainable and conventional pathways for heterocyclic scaffold synthesis, extracted from literature data, illustrates some common features. First of all, reaction times achieved using sustainable activation modes are significantly lower – from five to fifty times than those observed in conventional reflux conditions. Microwave activation proves to be the most effective method in terms of decreasing reaction time. Moreover, such a decrease in time automatically leads to a decrease in energy consumed for reactions since the whole energy consumed is directly proportional to the power and time. The yields obtained during sustainable methods prove to be equivalent to or better than those obtained through conventional methods, thus contradicting the myth that sustainable chemistry is associated with decreased efficiency of synthesis.

Sustainable methods' functional group tolerance depends on the activation mode used. Thus, photocatalytic and electrochemical activations are especially good in terms of selective activation of certain bonds through a one-electron transfer pathway and are capable of conducting reactions which cannot be conducted by means of conventional thermal activation. However, selectivity may turn out to be a drawback when wide functional group tolerance is needed as reactive intermediates produced may interact with functional groups.

Scalability is the most poorly developed issue. Despite the fact that commercial reactors operating in microwave irradiation in a range from single tubes to several liters are already available, the use of microwave reactors in industry is rather limited. There are many developments in electrochemical and photochemical flow reactors, which allow producing them in industrial scale; however, this type of reactor demands much capital expenditure and process development that is why their usage is not widespread.

V. Conclusion

The sustainable synthesis of heterocycles decorated with functional groups has evolved from scattered cases of successful application of green chemistry principles to a systematic methodology field characterized by established principles and performance metrics. Based on the survey conducted within the context of the present review, it is clear that alternative activation strategies such as microwave irradiation, mechanochemistry, and photocatalysis not only bring about significant environmental advantages but do not compromise synthesis efficiency either. Orders of magnitude reduction in reaction times are easily attained with an equal amount of energy savings. Optimized sustainable synthetic procedures yield results comparable or superior to conventional synthetic procedures in terms of yields and selectivity profiles.

The results of quantitative analysis presented in this review show that green metrics help to differentiate between various approaches to synthesis. Mechanochemical MCRs exhibit the best E-factor values due to the absence of waste from solvents that is the major source of the environmental footprint in traditional syntheses. Reactions carried out using ionic liquids yield the highest values, but the environmental cost of production of ionic liquids should be taken into account in life-cycle analysis. Microwave activation allows one to combine relatively fast reaction time, high yields, and wide applicability of the approach, making it the most readily available approach to achieving sustainability of heterocycle formation.

Great progress has been made in developing catalysts that could be reused in many cycles. Magnetic nanoparticles and ionic liquids are examples of such catalysts that solve the old problem associated with heterogeneously activated reactions, which add an extra level of complexity to synthesis without providing benefits. Immobilized enzymes allow for obtaining high stereoselectivity, which cannot be obtained via chemical catalysis; however, catalyst instability still poses a challenge. Development of more stable photocatalysts is the direction for future research.

A number of issues arise from the above analysis. Firstly, reporting of sustainable metrics should be standardized. Most publications on sustainable synthesis lack some of the sustainable metrics such as E-Factor, PMI, or energy consumption, thus making the comparative analysis difficult. The reporting of sustainable metrics should be made compulsory for all methodology papers. Secondly, the scaling up of sustainable synthesis should be investigated further as academic proof at milligram

level does not necessarily result in successful scaling up to kilogram or tonne level and process intensification may be required through continuous flow process implementation. Thirdly, the use of different sustainable techniques in combination, for instance, mechanochemical activation and biocatalysis or photonic catalysis, is not fully studied yet.

The history of the field would seem to indicate that sustainable heterocyclic chemistry will gradually become the norm as opposed to being an option. As greener metrics become integrated into everyday method development and as regulation of pharmaceutical production becomes more stringent, sustainable methodology will be embraced more and more. The methods presented in this review paper serve as proof that greener chemistry is not only possible but also synergistic with synthetic success.

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