Mini-Review:

Microwave-Assisted Synthesis of Bioactive Selenium Heterocycles: Recent Advances and Applications

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Abstract: *Selenium heterocycles and their fused rings are important class of compounds and they are found in many naturally occurring bioactive compounds. They have gained considerable attention as potential therapeutics for the treatment of many diseases such as diabetes, stroke, and cancer. Selenium incorporated natural products, including nucleosides, glycosides, and terpenoids, have been enhanced for synergistic pharmaceutical applications. Microwave-assisted organic synthesis (MAOS) has emerged as a powerful tool in green chemistry, offering significant advantages over conventional heating methods. By utilizing microwave irradiation, MAOS facilitates rapid and uniform heating, leading to improved reaction rates, higher product yields, and shorter reaction times. This technique aligns with the principles of atom economy, as it often allows for the complete conversion of substrates into desired products with minimal formation of undesired byproducts. Recent studies have demonstrated the efficacy of MAOS in synthesizing selenium-containing heterocycles. For instance, the microwave-assisted synthesis of 1,3-selenazole derivatives via Hantzsch-type condensation reactions has yielded high product yields and reduced reaction times compared to traditional methods. This review focuses on the development of selenium heterocycles under microwave irradiation and covers recent reports in the synthesis of these compounds.*

Keywords: *microwave; selenium heterocycles; green methods; bioactive compounds*

■ INTRODUCTION

Microwave-assisted synthesis is the use of microwave irradiation to carry out chemical reactions. Microwave is a form of electromagnetic energy falling at the lower frequency end (300 to 300,000 MHz) of electromagnetic radiation. The domestic microwave ovens work at 2.45 GHz. Microwave energy consists of an electric field and a magnetic field. The electric field transfers the energy to heat a substance. Microwaves interact with polar and ionic species. The substances try to align with the electric current created by the microwaves. As the electric field oscillates, the dipoles or ion field try to realign with the oscillating electric field and, in the process, lose energy in the form of heat due to molecular friction [1-2].

Microwave irradiation generates in-core heating, which passes through the material to couple directly with the electromagnetic component of the reacting molecules, inducing a rapid rise in temperature. Contrary to thermal heating, in the case of microwave heating in closed vessels, heating beyond the boiling point of a reaction medium can be obtained. The use of microwave technology for versatile chemical transformations in shorter reaction times and under milder reaction conditions compared to conventional heating has emerged as an important tool in contemporary organic synthesis. The effect of microwave irradiation on several

transformations and observed multifold acceleration of the reaction rates compared to the conventional methods have been reported since 1980s [3-4].

Recent advancements in microwave-assisted organic synthesis (MAOS) have enabled this technique to align closely with the core principles of green chemistry. Microwave irradiation promotes rapid and uniform heating, which leads to increased atom economy and higher product yields by accelerating reaction kinetics and minimizing the formation of side products. Moreover, it significantly enhances energy efficiency by reducing reaction times from hours to minutes, lowering overall energy consumption compared to conventional heating methods. Microwave technology also contributes to substantial waste and by-product reduction, as the optimized reaction conditions often eliminate the need for excess reagents and purification steps. In many cases, reactions can be performed under solvent-free conditions or with minimal use of benign solvents, thereby minimizing the reliance on toxic and hazardous chemicals. This aligns with the green chemistry goals of safer synthesis and reduced environmental impact. Furthermore, microwave-assisted reactions are generally characterized by operational simplicity and cleanliness, often taking place in closed systems that limit emissions and exposure risks. The overall reaction efficiency-considering time, yield, energy input, and environmental footprint is markedly improved, making microwave-assisted synthesis a powerful and sustainable tool in modern synthetic chemistry [5].

Many heterocyclic scaffolds are considered privileged structures [6-10]. Various heterocycles with different positional combinations of nitrogen, oxygen, or sulfur, in five- or six-membered rings are applied in a wide range of fields [11-15]. More than 85% of all biologically active molecules contain a heterocyclic moiety [16-17]. Selenium (Se) is an essential trace element and occurs naturally in many foods as a constituent of selenoproteins, which play an important role in DNA synthesis, thyroid hormone metabolism, reproduction, infection, and protection from oxidative damage [18]. Se is present both in organic and inorganic forms. Se derivatives reduce the risk of cancer [19]. Se-containing compounds are widely applied in different fields such as catalysis, materials chemistry, medicinal biology, organic synthesis, and polymer science [20-23].

Se powder, sodium selenide, selenium dihalides, selenoureas, isoselenocyanates, selenoamides, selenazadienes, and diorganyl diselenides are significantly used in the synthesis of Se-containing heterocycles [24-27]. Many of these find application as agrochemicals, antibiotics, anticancer agents, antioxidants, anti-inflammatory agents, antiviral agents, cardiotonics, dyes, spasmolytic agents, and organic electric conductors [28-33].

Selenazoles, selenophenes, selenazines, benzo[*b*]selenophenes, tetraselenafulvalene, selena crown ethers, selenopyrans are some examples of Se-containing heterocycles. Se chemistry has attracted the attention of researchers across the world due to the wide range of its applications. Fig. 1 includes some significant Se-containing heterocyclic compounds. Even though many reviews appeared on MAOS [34-44], there is still scope to provide a decadal update regarding the recent research reports on heterocyclic scaffolds such as Se-containing heterocycles. The present review aims to discuss the current advances in the microwave-assisted syntheses of Se-containing heterocyclic scaffolds.

■ ADVANCES IN MAOS OF Se HETEROCYCLES

Grozav Ignat and colleagues [45] developed a series of eight novel 1,3-selenazole derivatives through Hantzsch-type condensation reactions involving α-halogenocarbonyls and selenosemicarbazides (Fig. 2). These reactions were conducted under both classical heating and microwave irradiation conditions. Notably, the MAOS approach significantly enhanced reaction efficiency, yielding higher product quantities (up to 95%) within shorter reaction times compared to conventional methods.

The synthesized selenazole compounds were evaluated for their antiproliferative activity against two leukemia cell lines (CCRF-CEM and HL60) and three carcinoma cell lines (MDA-MB231, HCT116, and U87MG). Several derivatives exhibited promising cytotoxic effects, particularly against the CCRF-CEM

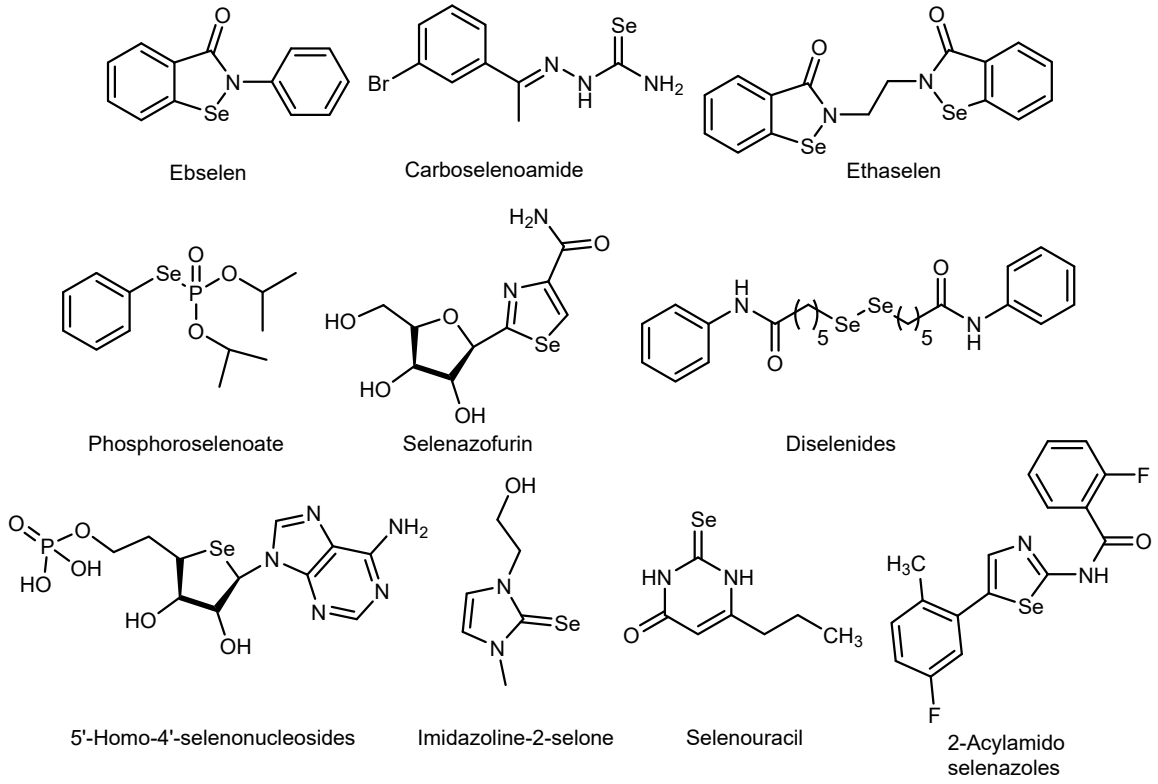


Fig 1. Some bioactive organo-selenium compounds

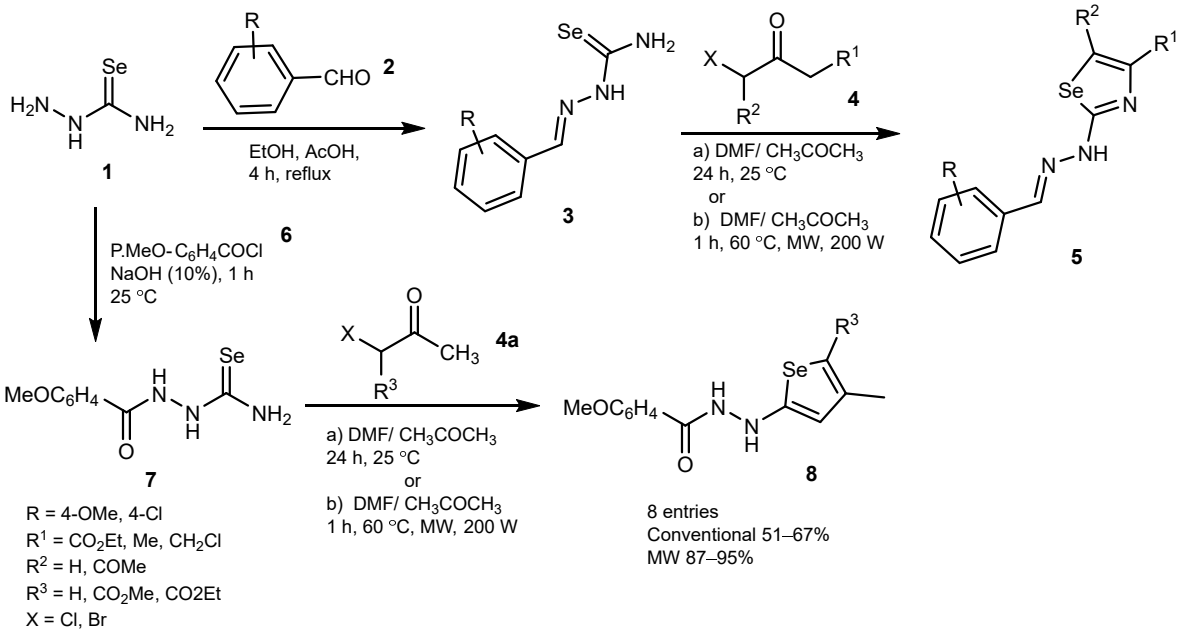


Fig 2. Synthesis of 1,3-selenazole compounds

leukemia cell line, with IC_{50} values below 10 μM, indicating potential as anticancer agents.

This study underscores the efficacy of MAOS in efficiently producing biologically active Se-containing

heterocycles. The findings suggest that such compounds hold promise for further development in anticancer therapeutics.

Raghavendra et al. [46] described the synthesis of some novel selenopyrano[2,3-*b*]quinolines by the reaction of 2-seleno-3-formyl-quinolines with diethylmalonate, ethyl acetoacetate and ethyl cyanoacetate in the presence of piperidine under microwave irradiation in 7–9 min (Fig. 3). Liang and colleagues [47] developed a series of benzimidazole-containing selenadiazole derivatives through a MAOS Hantzsch-type condensation reaction. This method involved the reaction of α-halogenocarbonyls with selenosemicarbazides in the presence of *N,N*-diisopropylethylamine (DIPEA) and the peptide coupling reagent HBTU, resulting in the formation of selenadiazole rings fused with benzimidazole cores (Fig. 4). The microwave irradiation significantly reduced reaction times and improved yields compared to conventional heating methods.

The synthesized compounds were evaluated for their antiproliferative effects against human breast cancer cell lines, including MDA-MB-231. Notably, several derivatives induced cell cycle arrest and apoptosis, mechanisms associated with the ROS activation and the AKT signaling pathway. These findings suggest that the incorporation of Se into the benzimidazole scaffold enhances the biological activity of the compounds, potentially through the modulation of redox-sensitive pathways. This study highlights the efficacy of microwave-assisted synthesis in the rapid and efficient preparation of Se-containing heterocycles with promising anticancer properties. The incorporation of Se into the benzimidazole framework offers a strategic approach to developing novel therapeutic agents targeting breast cancer.

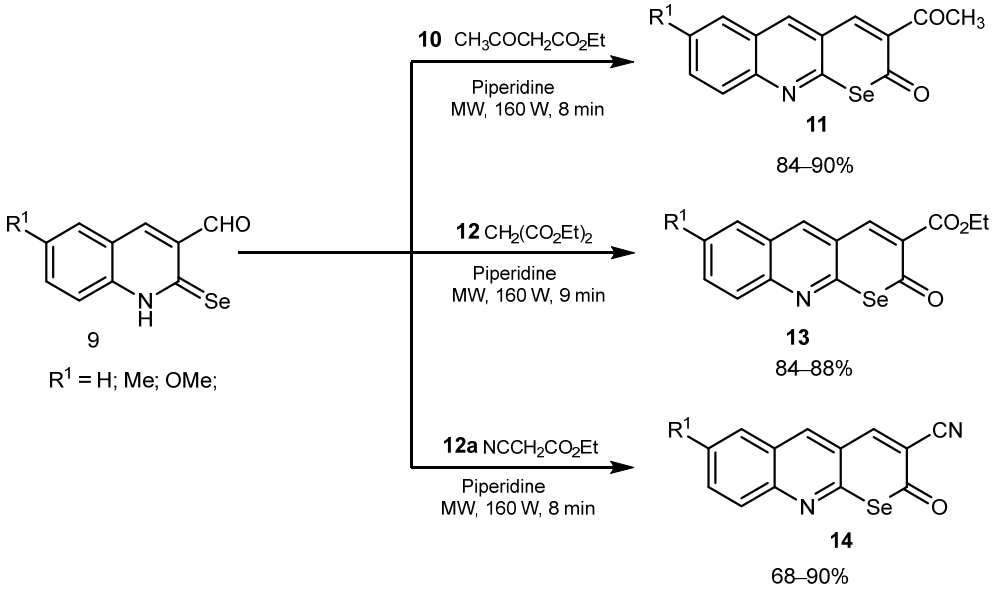


Fig 3. Synthesis of selenopyrano[2,3-*b*]quinoline derivatives

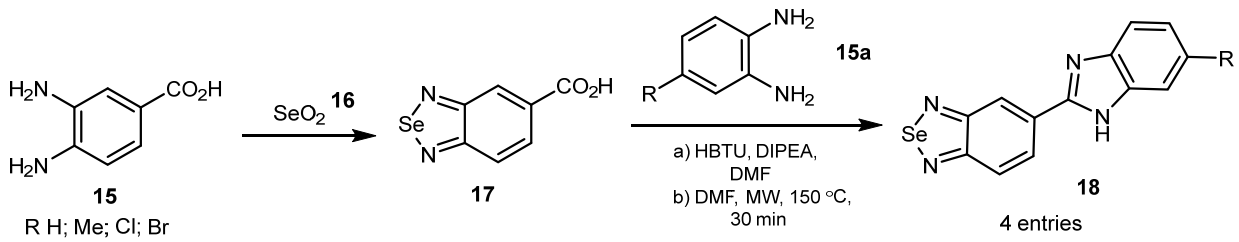


Fig 4. Synthetic approach to benzimidazole containing selenadiazole derivatives

A novel one-pot protocol for 2-(alkyl)aryl benzoselenazoles from *N*-(acetyl)benzoyl-2-iodo anilines was developed by Redon et al. [48] using Woollin's reagent-(2,4-diphenyl-1,3,2λ^5,4λ^5-diselena-diphosphetane-2,4-diselone) as a selenating agent under microwave irradiation (Fig. 5(a–b)). The authors examined various conditions of the model reaction by using different bases and solvents as well as reaction temperatures. The combination of microwave irradiation and ionic liquid was effectively employed by Chandramouli [49] for the synthesis of a series of 2-amino-1,3-selenazoles. The reaction involved the condensation of α-bromo-β-keto esters with acyl or phenacyl halides, affording the desired products in excellent yields within remarkably short reaction times (Fig. 6). The process utilized 1-butyl-3-methylimidazolium tetrafluoroborate ([Bmim][BF_4]) as the ionic liquid, which played a dual role as both solvent and catalyst. Notably, the ionic liquid could be recovered and reused up to 10 times without any significant loss in catalytic efficiency, highlighting its sustainability and economic advantages.

A key feature of this methodology is the application of microwave irradiation, which significantly enhances the reaction rate and yield compared to conventional thermal heating methods. Under microwave conditions, the uniform and rapid internal heating reduces reaction times from hours to minutes, minimizes side reactions, and often leads to cleaner product profiles. In contrast, traditional heating methods typically require longer durations and higher energy input, which may result in lower yields and reduced selectivity. The synergistic effect of microwave irradiation with the ionic liquid medium provides a green and efficient alternative for the synthesis of heterocyclic compounds such as selenazoles.

Shinde et al. [50] described the synthesis of novel thiadiazol, selenadiazole, and spirocyclic benzopyrans by both conventional and non-conventional methods involving semicarbazides and thiosemicarbazides of 2-ethyl-2-methyl-4*H*-chromen-4-ones (Fig. 7). The products were screened for both anti-bacterial and anti-fungal activities.

The authors compared the results obtained by conventional, microwave, and ultrasound methods. Solvent-free, microwave assisted one-pot synthesis of selenolo[2,3-*b*]quinoline derivatives was developed by Raghavendra et al. [51] by the reaction of 2-seleno-3-formyl quinolines with 2-chloroacetamide and phenacyl bromide in the presence of potassium carbonate (Fig. 8).

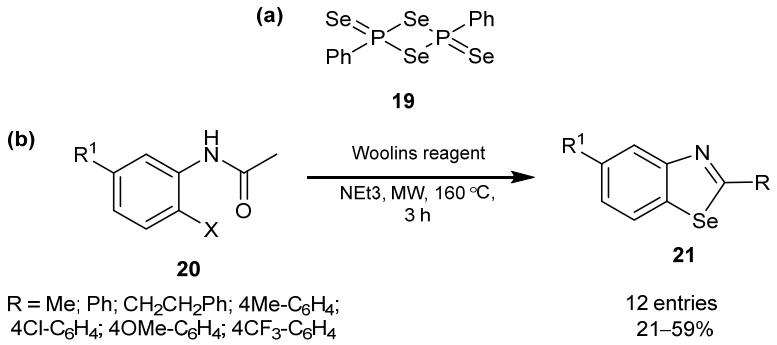


Fig 5. (a) Woollin's reagent and (b) one-pot cyclization of *N*-acetyl-2-halo anilines with Woollin's reagent

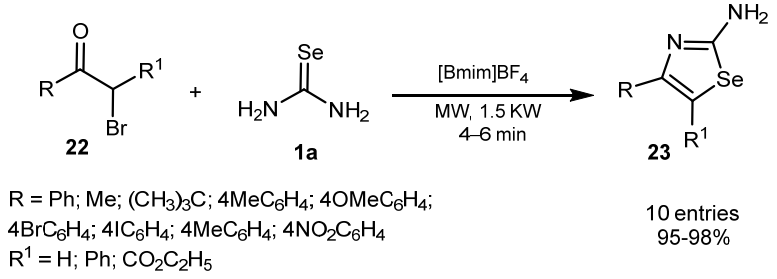


Fig 6. [Bmim]BF_4 catalyzed synthesis of 1,3-selenazole derivatives

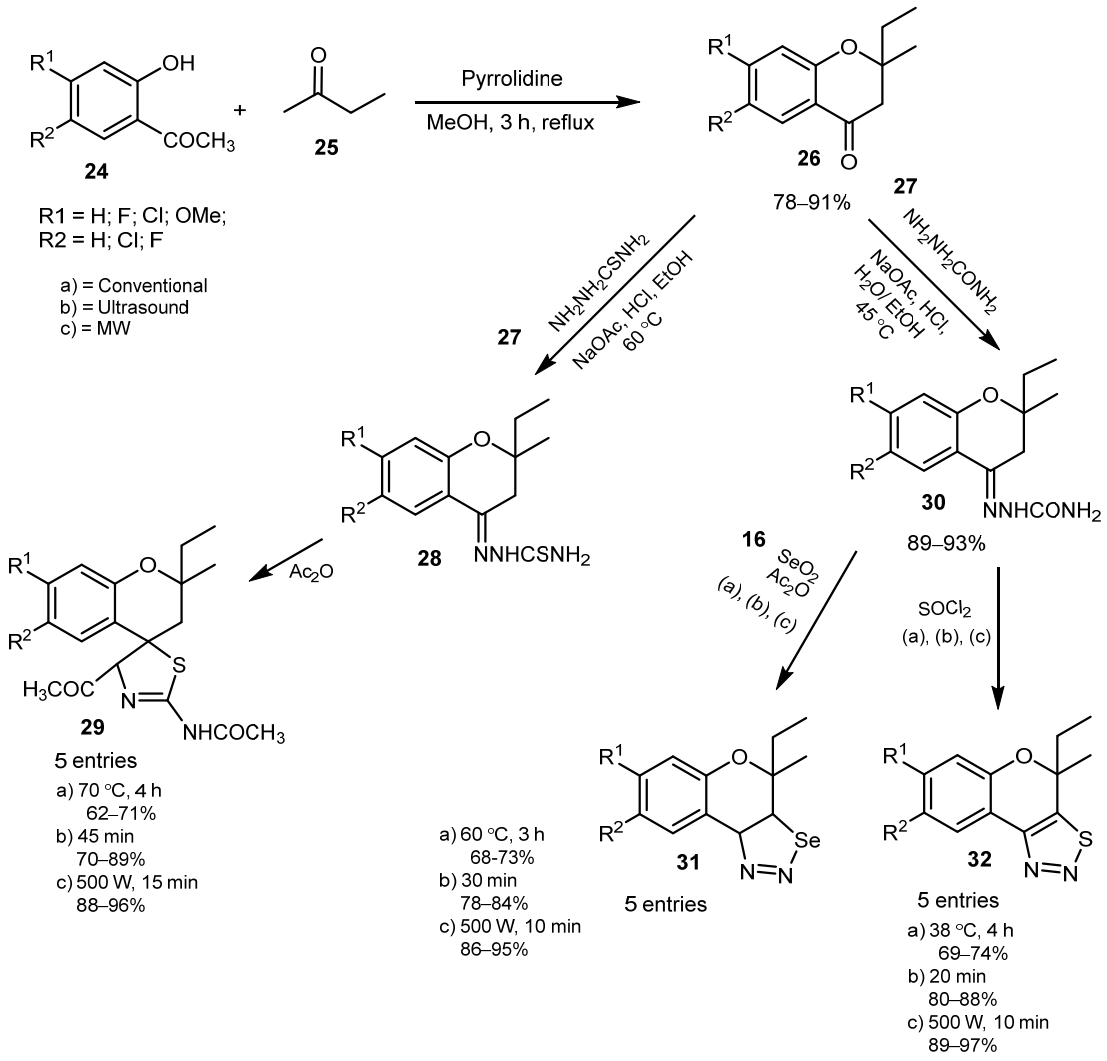


Fig 7. Synthesis of novel thiadiazoles, selenadiazoles, spirocyclic benzopyran derivatives by conventional, microwave and ultrasound methods

Several substituted (*Z*)-4-methylene-3-selenaquinolines were synthesized by Sashida et al. [52] through an efficient and environmentally friendly approach involving the reaction of *o*-ethynylanilines with isoselenocyanates. The key transformation proceeds via a 6-*exo*-*dig* cyclization mechanism under solvent-free and catalyst-free conditions, facilitated by microwave irradiation (Fig. 9(a–b)). This protocol enabled the formation of selenated quinoline derivatives in good to excellent yields within remarkably short reaction times. In their preliminary efforts to optimize the synthesis of 2-imino-3-selenaquinolines, the authors explored a variety of reaction conditions, including different heating methods, reaction times, and reagent ratios. Microwave irradiation proved to be the most effective, significantly outperforming conventional thermal methods. Reactions carried out under traditional heating typically required extended durations (often several hours) and produced lower yields, sometimes accompanied by the formation of undesired by-products. In contrast, the microwave-assisted protocol enabled rapid and selective ring closure within minutes, offering a cleaner reaction profile and improved overall efficiency. This study highlights the substantial advantages of microwave technology in heterocyclic synthesis, particularly for Se-containing frameworks where it reduces energy consumption and

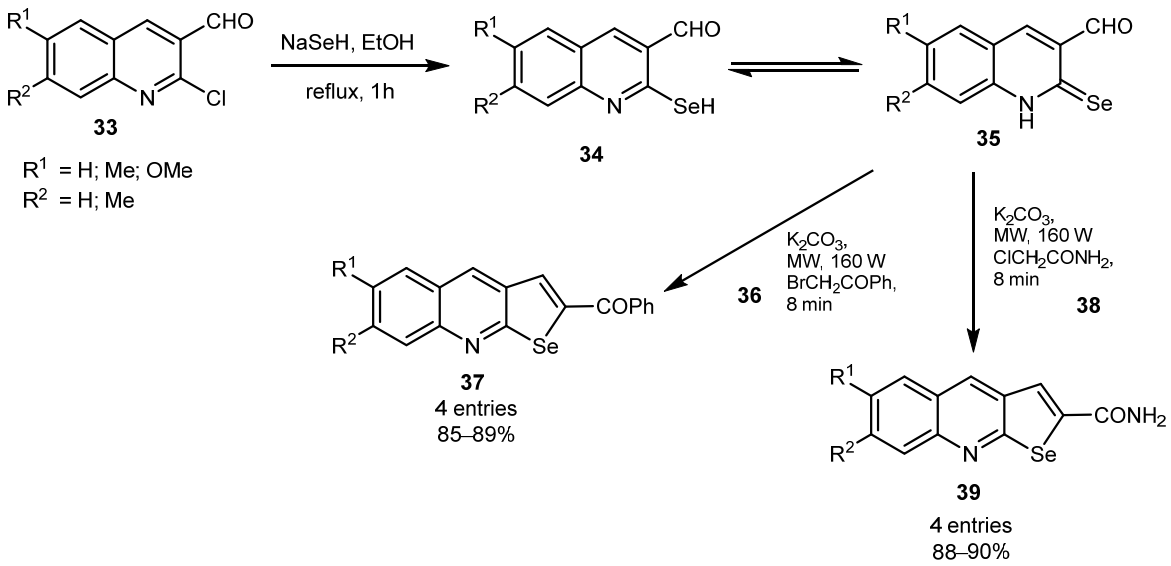


Fig 8. Synthesis of selenolo[2,3-*b*] quinolines

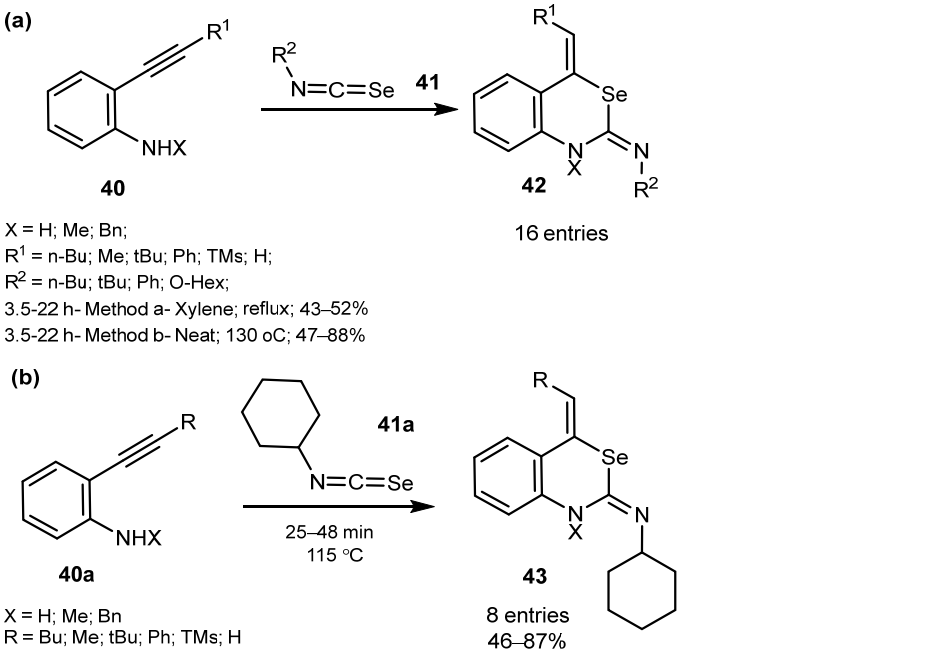
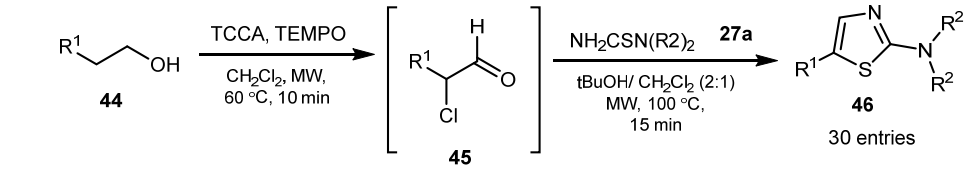


Fig 9. (a) Synthesis and (b) microwave irradiated synthesis of 4-methylene-3-selena quinolines

reaction time and eliminates the need for solvents and catalysts. These features make the method highly compatible with green chemistry principles, distinguishing it as a superior alternative to classical synthetic routes.

A versatile, easily accessible, and cost-effective protocol has been developed by Macías-Benítez et al. [53] for synthesizing a wide range of heterocyclic compounds, including 2-aminoimidazoles, 1,3-selenazoles, hydrazinothiazoles, imidazo[1,2-a]pyridines, and quinoxalines (Fig. 10–15). This method utilizes a variety of primary and secondary alcohols as starting materials, employing trichloroisocyanuric acid (TCCA) as a dual oxidant and chlorine source, TEMPO as a co-oxidant, and thiourea or appropriate surrogates. In preliminary investigations, various chlorinating agents such as TsCl,



R^1 = Me; Et; -$(CH_2)_5$-CH_3; -$(CH_2)_5$-CH_3; -$(CH_2)_9$-CH_3; -CH_2-$CH(CH_3)_2$; -$CH(CH_3)CH_2CH_3$; Ph

R^2 = H, H; H, Ph; H, CH_2Ph; H, cyclohexyl; H, $(CH_2)_5$-CH_3; H, $CH(CH_3)$Ph; H, $(CF_3)_2C_6H_3$; H, $(iPr)_2$ C_6H_3

Fig 10. Preparation of substituted 2-amino thiazoles

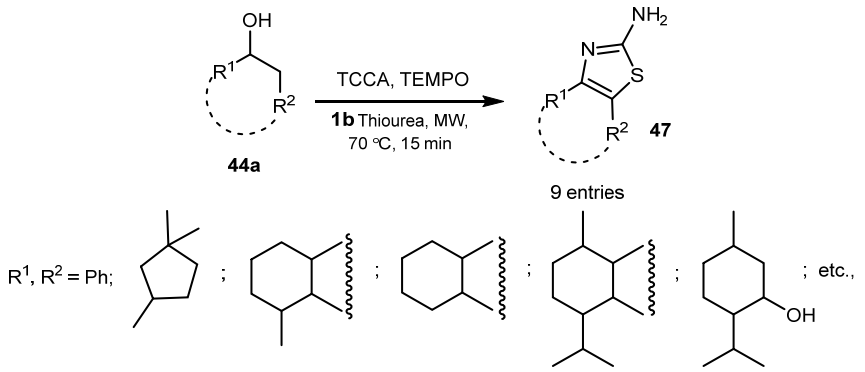
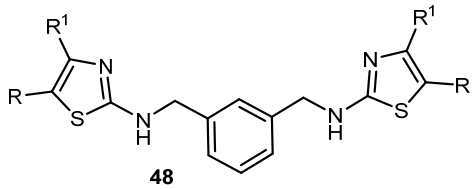
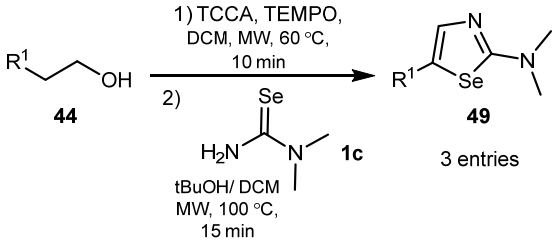


Fig 11. Synthesis of substituted 2-amino thiazoles from secondary alcohols



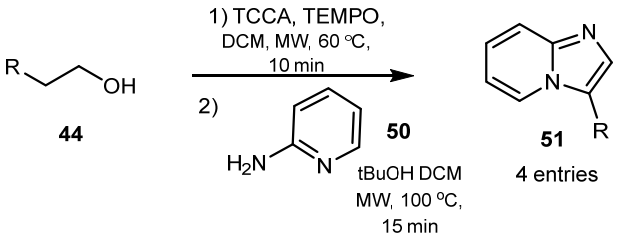
R = Et; $(CH_2)_5CH_3$; Ph; $(CH_2)_9CH_3$; R^1 = Ph

Fig 12. Preparation of thiazole containing dimers



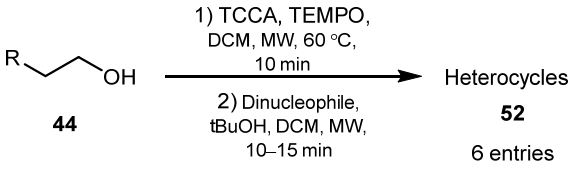
R^1 = Ph; $(CH_2)_5$-CH_3; $(CH_2)_9$-CH_3

Fig 13. Synthesis of selenazoles



R = Ph; $(CH_2)_3$-CH_3; $(CH_2)_5$-CH_3; $(CH_2)_9$-CH_3

Fig 14. Synthesis of imidazo[1,2-*a*]pyridines



Dinucleophile: OPDA, PhNH-C(=NH)NHPh; Ph-$C(CH_3)$=NNHC(=S)NH_2

Fig 15. Preparation of other heterocycles

$Ca(OCl)_2$, NaOCl, and TCCA were assessed for their efficacy in the process. A range of solvents, including water, dichloromethane (CH_2Cl_2), acetonitrile (CH_3CN), *tert*-butanol (^{t}BuOH), and combinations thereof, were screened using octan-1-ol as the model substrate for thiazole derivative synthesis. Different heating methods, including conventional heating, one-pot concurrent approaches, and one-pot sequential addition approaches, were evaluated. Additionally, the effect of varying TEMPO concentrations on the reaction outcome was studied. This protocol demonstrates significant versatility and efficiency in constructing diverse heterocyclic frameworks from readily available alcohols, offering a valuable tool for synthetic organic chemistry.

Arsenyan and co-researchers [54] carried out microwave-assisted, palladium-catalyzed cyanation of 3-bromo-2-(1-hydroxyalkyl)benzo[*b*]selenophene,

producing 3-cyano compounds, using $Zn(CN)_2$ as cyanide source in DMF (Fig. 16). Attia and Abdel-Hafez [55] developed an operationally simple, microwave-assisted, one-pot synthesis of seleno[2,3-*b*]pyridine and seleno[2,3-*b*]quinoline derivatives, employing recyclable cobalt oxide nanoparticles (Co_3O_4 NPs) as an efficient heterogeneous catalyst (Fig. 17-18). The Co_3O_4 NPS were synthesized via a co-precipitation method using $CoCl_2{\cdot}6H_2O$ and NaOH. The resulting precipitate was filtered, washed, dried, and calcined to obtain the desired Co_3O_4 NPs.

Under microwave irradiation, the catalytic system facilitated the formation of the target seleno-heterocycles with higher yields (> 90%) and shorter reaction times under moderate conditions. The catalyst demonstrated excellent reusability, maintaining its activity over five consecutive cycles without significant loss in efficiency. This methodology offers a green and efficient approach for synthesizing biologically significant organoselenium

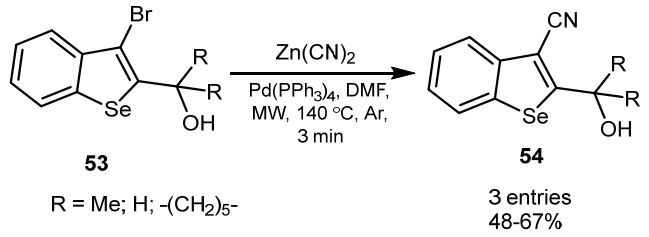


Fig 16. Synthesis of 3-cyano-benzo[*b*]selenophenes

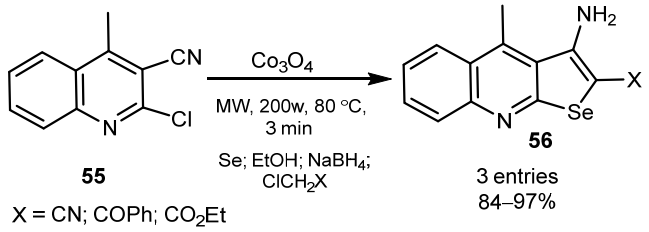


Fig 17. Co_3O_4 NPs catalyzed the synthesis of a seleno[2,3-*b*]pyridine/quinolines

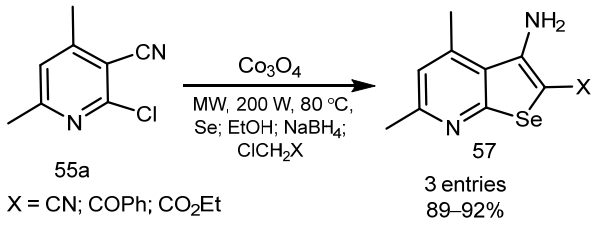


Fig 18. Synthesis of seleno[2,3-*b*]pyridine/quinolines

compounds, highlighting the potential of Co_3O_4 NPs in sustainable organic synthesis. The results of the reactions under thermal conditions were compared to those obtained by microwave irradiation.

■ CONCLUSION

This review underscores the pivotal role of microwave irradiation in the synthesis of Se-containing heterocycles, highlighting its alignment with the principles of green chemistry. The innovative and eco-friendly microwave irradiation strategies discussed herein offer significant advantages, including enhanced reaction rates, higher product yields, and reduced reaction times, all under milder conditions compared to conventional methods. Such operationally convenient approaches not only facilitate the efficient construction of diverse heterocyclic scaffolds but also contribute to sustainable practices in chemical synthesis.

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■ CONFLICT OF INTEREST

The authors declare no conflicts of interest regarding this review.

■ AUTHOR CONTRIBUTIONS

Yadavalli Venkata Durga Nageswar served as the corresponding author, wrote the manuscript, and performed the revisions. Ramesh Katla and Rakhi Katla served as co-authors; they contributed to writing some reports, designed ChemDraw files, and reviewed the revision results.

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