RESEARCH ARTICLE

BIOCHAR CATALYZED SYNTHESIS OF SUBSTITUTED TETRAZOLE DERIVATIVES

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Abstract:

The sustainable transformation of organic molecules using environmentally responsible catalysts has become a central objective in modern synthetic chemistry. Biochar, a carbon-rich material derived from pyrolysis of biomass, has emerged as an efficient, tunable, and renewable catalytic material for various organic reactions. Among the numerous biochar-enabled transformations, the synthesis of tetrazole derivatives has gained significant attention due to the biological and pharmaceutical relevance of tetrazoles and the need for green catalytic alternatives. This review provides an in-depth and focused analysis of recent developments in the use of functionalized biochar-based catalysts for the synthesis of substituted tetrazoles. The article discusses biochar production, physicochemical features, catalyst functionalization strategies, mechanistic insights, representative catalytic systems, and comparative advantages over conventional catalysts. It also summarizes the structural diversity of biochar-supported metal and non-metal catalytic systems that efficiently promote [3+2] cycloaddition between nitriles and azides. Reusability, sustainability, reaction efficiency, and operational simplicity are critically evaluated. Finally, challenges and future prospects are discussed to guide the continued development of biocharassisted tetrazole synthesis as an emerging pillar of green heterocyclic chemistry.

Keywords: Biochar, Tetrazoles, Green Catalysis, Biomass Pyrolysis, Sustainable Chemistry, [3+2] Cycloaddition.

1. Introduction:

Tetrazoles are nitrogen-rich heterocycles that hold remarkable importance in medicinal, agricultural, and materials chemistry.[1–5] They frequently act as bioisosteres of carboxylic acids, enhance metabolic stability, and exhibit diverse biological properties such as antimicrobial, anticancer, anti-inflammatory, and antiviral activities. Traditional methods for producing tetrazoles often rely on strong Brønsted acids, metal salts, toxic organic solvents, and harsh reaction conditions,

leading to sustainability concerns. Thus, the development of green, efficient, and reusable catalysts for tetrazole synthesis has become a priority.[6–9]

Parallel to this, biochar has emerged as a transformative catalyst support and catalytic material in green chemistry.[1,10–14] Biochar is produced by pyrolysis of biomass, such as agricultural residues, animal waste, wood, or plant biomass, under limited oxygen. The resulting carbon-rich structure contains functional groups (carboxyl, hydroxyl, carbonyl), diverse porosity, high stability, tunable surface chemistry, and excellent compatibility for further modification. Functionalization of biochar allows incorporation of Lewis acidic metals, magnetic particles, ionic liquids, or heterocyclic ligands, resulting in highly active catalysts suitable for organic transformations.[1,2,9–17]

Although biochar has been applied to a broad range of reactions, its use in heterocycle synthesis, especially tetrazoles, has rapidly advanced only in the past few years. Biochar-supported catalysts offer several key advantages for tetrazole synthesis. Modern catalytic strategies in heterocyclic synthesis increasingly emphasize sustainability, efficiency, and environmental responsibility.[1,2,9–21] Catalysts that operate under mild reaction conditions not only reduce energy consumption but also minimize the formation of hazardous by-products, thereby lowering the overall environmental impact. Their high catalytic activity and selectivity ensure rapid formation of the desired products with minimal waste or side reactions. Many contemporary systems, especially magnetic or mechanically recoverable catalysts, offer the additional advantage of simple separation from the reaction mixture, eliminating the need for complex purification steps.[22–24] Furthermore, minimal metal leaching enhances product purity and reduces ecological concerns, while the ability of these catalysts to be reused over multiple cycles significantly improves cost-effectiveness and operational sustainability.[25–27] Importantly, their compatibility with green solvents such as PEG-400 or water aligns strongly with the principles of green chemistry, making them ideal candidates for developing eco-friendly and high-performance synthetic methodologies in organic synthesis.

This review systematically presents all major advances in biochar-catalyzed tetrazole synthesis, offering mechanistic analysis and evaluating their placement within sustainable chemistry.

2. Biochar: Sources, Preparation, and Functionalization for Tetrazole Catalysis

Biochar used in tetrazole catalysis derives its catalytic efficiency largely from the type of biomass selected as the feedstock, since different natural sources provide distinct elemental compositions and structural features. Commonly employed materials include plant-based residues such as neem bark, wheat straw, bamboo, woody biomass, and various agricultural wastes, all of which generate carbon-rich, high-surface-area char with stable aromatic or graphitic frameworks suitable for catalyst support. However, nutrient-rich feedstocks like chicken manure have gained particular prominence in recent tetrazole synthesis studies because they contain abundant minerals, nitrogen, and inorganic components that enhance metal anchoring, increase surface basicity, and facilitate thermal stability during catalytic operations. This inherent compositional advantage enables chicken-manure-derived biochar to form robust, functionalizable carbon matrices, making it an especially effective and versatile platform for developing biochar-supported catalytic systems used in tetrazole formation.[25–31]

Pyrolysis plays a central role in preparing biochar-based catalysts for tetrazole synthesis, with slow pyrolysis at 300–800 °C being the most widely used method because it yields mechanically robust carbon frameworks with desirable porosity and abundant surface functionalities. The pyrolysis parameters, temperature, residence time, and heating rate, directly influence the surface area, pore-size distribution, ash content, mineral composition, and degree of aromatization, all of which determine the catalytic performance of the resulting biochar.[7,32] Although advanced techniques like microwave-assisted pyrolysis offer advantages such as uniform heating and shorter processing times, their use in tetrazole catalysis remains limited. Following pyrolysis, the raw biochar typically requires functionalization to become catalytically active. Common modification strategies include silane functionalization to create anchoring sites, grafting organic ligands such as Schiff bases, azoles, or pyridyl groups, and immobilizing metal ions (Cu^{2+}, Ni^{2+}, Fe^{2+}, Nd^{3+}) to provide Lewis acidic centers essential for activating nitriles and azides. Additional enhancements such as magnetization with Fe_3O_4 nanoparticles allow easy recovery of the catalyst, while ionic liquid modification improves solubility, stability, and catalytic efficiency. Together, these structural refinements transform biochar into a powerful and versatile platform for promoting tetrazole formation.[6,33–38]

3. Biochar-Catalyzed Synthesis of Tetrazoles

Moradi and co-workers explored the use of biochar as a renewable and resilient support for developing functionalized nanocatalysts, contributing to sustainable catalyst design.[2] Biochar, a carbon-rich solid with surface functionalities such as carbonyl, carboxyl, and hydroxyl groups, provides an excellent platform for heterogeneous catalysis. In their study, biochar nanoparticles (BNPs) were first prepared via the pyrolysis of chicken manure. These BNPs were then treated with 3-chloropropyltrimethoxysilane (CPTMS) to generate CPTMS@biochar. Subsequent anchoring of 2-(thiophen-2-yl)-1H-benzo[d]imidazole onto this modified surface produced TBA@biochar, which was finally loaded with copper to yield the active catalytic material, CuTBA@biochar. Comprehensive characterization using TGA, XRD, SEM, EDS, nitrogen adsorption–desorption, AAS, and FT-IR confirmed nanoparticle sizes below 100 nm. Remarkably, the catalyst required only 0.78 mol% loading to deliver tetrazole derivatives (3) in high yield through a [3+2] cycloaddition between nitriles and sodium azide (Scheme 1a). High TOF and TON values demonstrated the system's efficiency. The synthesized tetrazoles displayed notable antibacterial activity against both Gram-positive and Gram-negative strains. Importantly, the catalyst maintained its performance over several cycles without copper leaching, highlighting its long-term durability and green chemistry relevance.

Norouzi et al. introduced a magnetically responsive Fe_3O_4-biochar catalyst for organic transformations. In their approach, BNPs were prepared through slow pyrolysis of *Laurnatus europaeus* biomass at 700 °C with a controlled heating rate of 5 °C/min under nitrogen for 15 hours. Magnetization was achieved by embedding Fe_3O_4 nanoparticles, followed by functionalization with (3-aminopropyl)triethoxysilane (APTES). The modified biochar was further derivatized with cyanuric acid and thiourea, creating an appropriate platform for immobilizing a copper complex, ultimately producing MBC@BTT-Cu(II). This multifunctional nanocatalyst efficiently promoted the synthesis of 5-substituted 1H-tetrazoles (3) (Scheme 1b) from benzonitrile derivatives and sodium azide. The

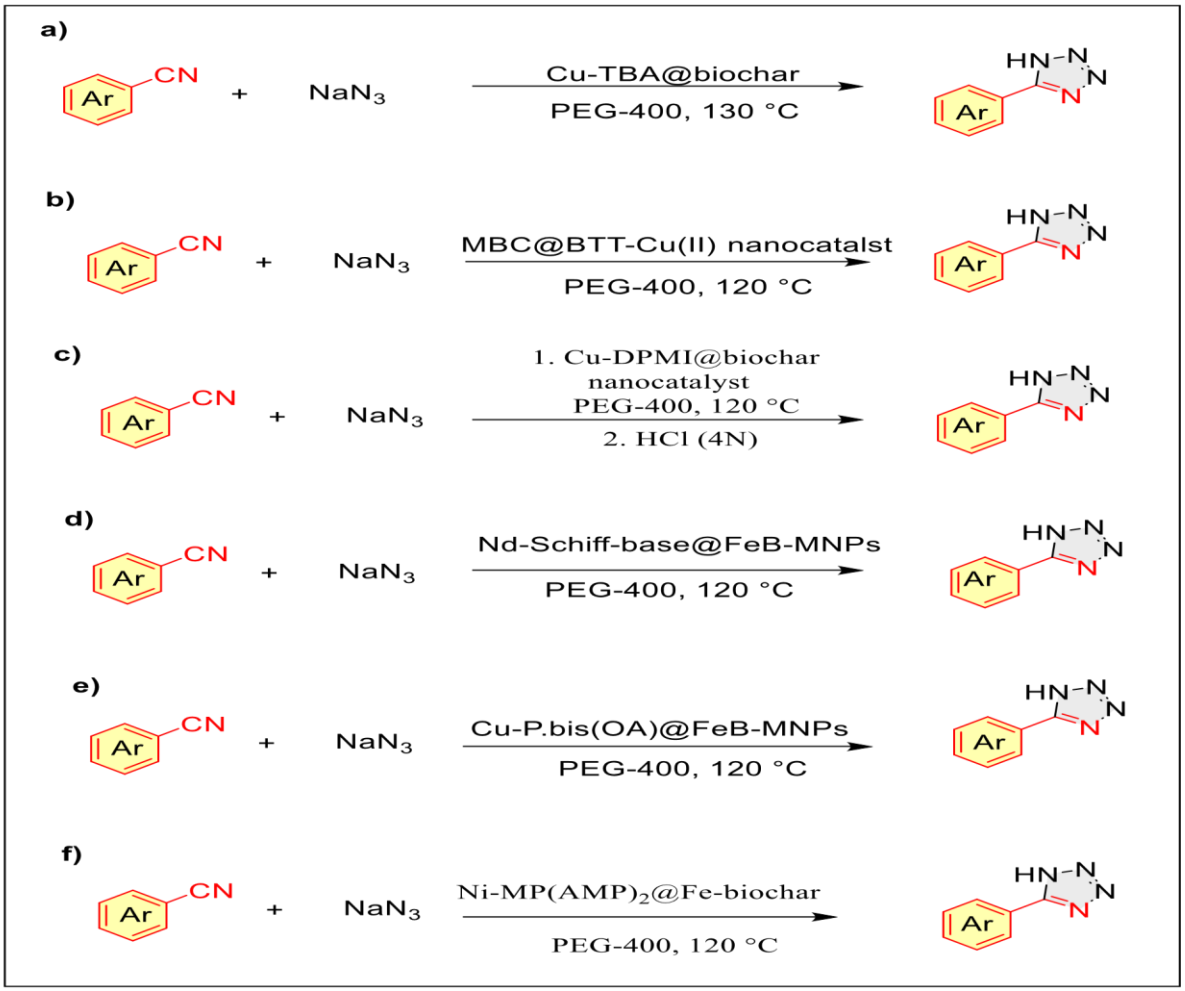
method is characterized by low catalyst loading, simple operation, excellent yields, and recyclability for at least five successive cycles with minimal loss of catalytic strength. Mechanistically, the nitrile (1) first coordinates to the copper species, forming intermediate I (R-C≡N-Cu). This adduct undergoes a [3+2] cycloaddition with azide ion to form intermediate II, which upon protonation in acidic medium produces the final tetrazole. The study highlights the advantages of magnetic biochar-supported metal catalysts, which exhibit high activity, facile separation, reduced toxicity, and compatibility with green solvents.

Biochar nanoparticles have become prominent precursors for carbon-based catalytic materials due to their tunable structure and porosity. Tahmasbi and colleagues developed BNPs from chicken manure through pyrolysis, providing an environmentally sound route for waste valorization. These BNPs were modified with (3-aminopropyl)trimethoxysilane in *n-hexane* at 60 °C for 24 hours to yield nPr-NH_2@biochar.[7] Further functionalization with di(pyridin-2yl)methanone in ethanol at 80 °C for 24 hours produced DPMI@biochar. Immobilization of a copper Schiff-base complex on this substrate afforded the catalyst Cu-DPMI@biochar. This catalyst displayed excellent activity and reusability in the homoselective synthesis of 5substituted tetrazoles (Scheme 1c) via [3+2] cycloaddition between nitriles and sodium azide. Work-up involved dilution with water and ethyl acetate followed by simple filtration to recover the catalyst. Based on literature-supported mechanisms, the copper center serves as the active site, initially binding the nitrile to generate intermediate I, which then reacts with azide to form the sodium tetrazolate intermediate II. Subsequent acid treatment with HCl converts II into the neutral tetrazole. Cu-DPMI@biochar exhibits high functional stability, minimal metal leaching, and strong selectivity, demonstrating its promise as an efficient nanostructured catalyst.

Tahmasbi et al. further reported an environmentally friendly neodymium-based catalyst (NdSchiff-base@BMNPs) immobilized on magnetized biochar nanoparticles. The process began with the preparation of BNPs from chicken manure, which were then magnetized using nickel nanoparticles to generate BMNPs.[29] Modification with 3-CPTMS produced an activated surface suitable for anchoring a neodymium Schiff-base complex. Structural analysis confirmed the successful functionalization, though the surface area (16.081 m^2/g) and pore volume (0.058 cm^3/g) were reduced due to the immobilized catalytic species. This catalyst efficiently facilitated the homoselective synthesis of tetrazoles (Scheme 1d) through a [3+2] cycloaddition of nitriles and sodium azide in benign solvents such as PEG-400. The catalytic sequence involves activation of the nitrile by Nd-complexation, enabling nucleophilic attack by azide to form the sodium salt intermediate (II), which is subsequently protonated to yield the tetrazole derivative. The catalyst can be readily separated magnetically and reused several times, maintaining its selectivity and structural stability. This study underscores the potential of renewable, magnetically recoverable biochar-based materials in sustainable catalysis.

In another recent contribution, Tahmasbi and co-workers described the synthesis of tetrazoles using a multidentate copper complex immobilized on BMNPs (Cu-P.bis(OA)@FeB-MNPs).[1] The biochar platform originated from chicken-manure-derived BNPs, aligning with waste-toresource

strategies. Magnetic Fe(0) nanoparticles enhanced catalyst recovery, while further surface engineering improved catalytic efficiency.

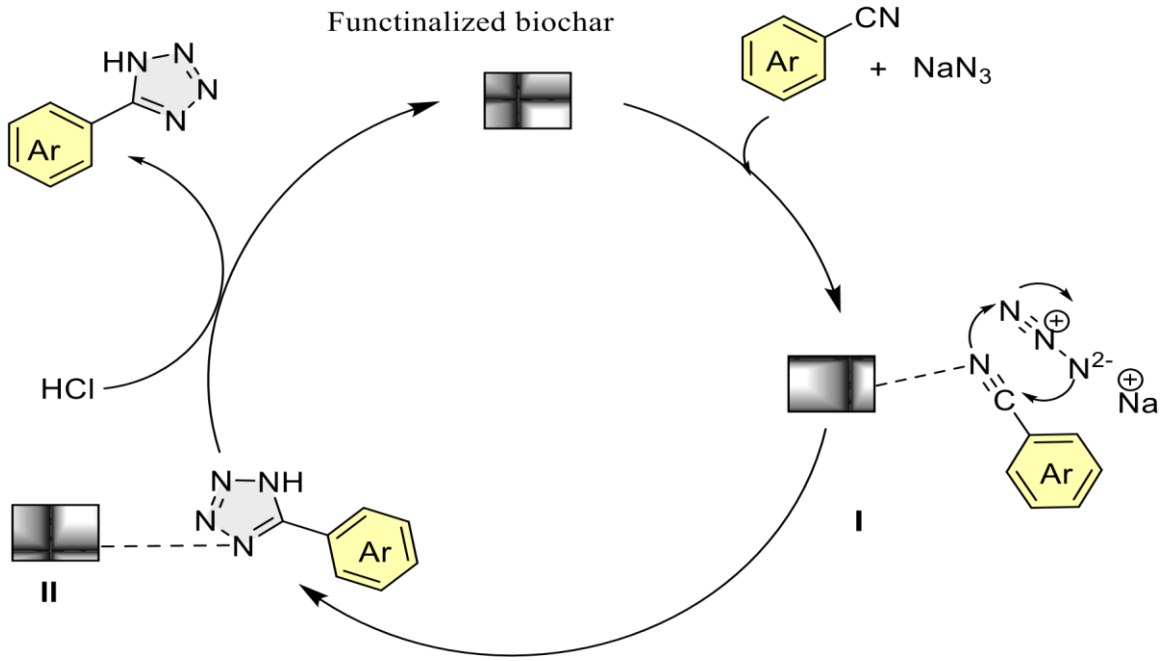


Scheme 1: Biochar-catalyzed synthesis of tetrazole derivatives

This catalyst promoted the formation of 5substituted tetrazoles (3) (Scheme 1f) via [3+2] cycloaddition between nitriles and sodium azide in PEG-400, providing high yields (up to 97%) within only 2 hours. Characterization by SEM, TGA, and VSM confirmed its stability and recyclability over six cycles. Importantly, the catalyst exhibited homoselectivity, favoring monoaddition even in substrates containing two cyano groups. Its superior yield, selectivity, and eco-compatibility positioned it as an improved lternative to existing catalytic systemsHajjami and colleagues introduced a sustainable route for producing BNPs from waste chicken manure. These BNPs underwent magnetization through a green procedure and subsequent modification to create a suitable matrix for immobilizing nickel complexes. The final material, Ni-MP(AMP)$_2$@Fe-biochar, acted as a homoselective and reusable catalyst for various organic reactions.[9] It efficiently catalyzed the synthesis of tetrazoles (Scheme 1g) from nitriles and sodium azide.

Moreover, the catalyst exhibited strong performance in forming pyranopyrazoles through a fourcomponent condensation involving benzaldehyde, ethyl acetoacetate, hydrazine hydrate, and malononitrile. Proposed mechanistic pathways for both product types were consistent with catalytic activation of nitriles and stepwise cycloaddition processes. The magnetic nature of the catalyst allowed

easy recovery with an external magnet, and it retained activity over repeated cycles. Overall, this work introduced a novel method for developing magnetized biochar as a support for nickel complexes, offering an efficient and sustainable catalyst for heterocyclic synthesis.



Scheme 2: General mechanism for the biochar-catalyzed synthesis of tetrazole derivatives

Conclusion:

This review compiles and compares a wide range of biochar-derived catalysts, each produced from different biomass sources and modified using various chemical treatments, demonstrating their broad applicability in the synthesis of heterocyclic compounds. Biochar-based catalysts provide a powerful, sustainable platform for the synthesis of substituted tetrazole derivatives. Their tunable structure, high surface area, and compatibility with metal complex immobilization make them ideal candidates for promoting the [3+2] cycloaddition between nitriles and sodium azide. Recent advancements have demonstrated excellent catalytic efficiency, high yields, and recyclability, positioning biochar as a transformative material in green tetrazole synthesis. Continued innovation in functionalization strategies, feedstock selection, and hybrid material design is expected to further enhance biochar's utility in modern organic chemistry.

Conflict of Interest:

The author declares that there is no conflict of interest regarding the publication of this chapter.

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