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RAPID AND EFFICIENT SYNTHESIS OF IMIDAZOLE [1,2-A] PYRIDINE VIA SILICA SUPPORTED Cu-Zn NPS BY GREEN CHEMISTRY ROUTE (CLICK CHEMISTRY)

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ABSTRACT

A novel and environmentally sustainable method for synthesizing imidazole[1,2-a] pyridine derivatives was developed using silica-supported Cu-Zn nanocomposites as catalysts in an ethanol medium under sonochemical conditions. This approach utilizes cost-effective and recyclable materials, which positively impact both economic growth and environmental sustainability. Silica-supported Cu-Zn nanocomposites offer several advantages over traditional catalysts, such as strong acids or bulk metal catalysts. Unlike conventional acidic or metallic systems, which often require harsh conditions and are difficult to recover, Cu-Zn nanocomposites supported on silica provide a high surface area, enhanced dispersion of active sites, and improved catalytic efficiency under milder conditions. The synergistic interaction between copper and zinc enhances both redox and Lewis acid properties.[1,2] Facilitating faster and more selective reactions. Additionally, the silica support contributes to thermal stability, ease of handling, and excellent recyclability, making these nanocomposites a more sustainable and practical alternative for green synthesis, particularly for the formation of complex heterocycles such as imidazo[1,2-a]pyridines. We report the use of silica-supported nanocomposite particles as catalysts, demonstrating excellent activity in the one-pot tricomponent condensation of imidazo[1,2-a]pyridine and achieving high yields (91–96%). An efficient green alternative was realized by performing the reaction at 60°C. Sonochemistry, which enhances the reaction rate through ultrasound waves, was employed to accelerate the catalytic process. High yields were achieved using silica-supported Zn nanocomposites. Ethanol serves as a sustainable solvent and a biodegradable alternative to toxic solvents. This sonochemical approach underscores green chemistry methodologies that promote the sustainability and efficiency of organic synthesis.[3,4,5]

Keywords: NPS: Nano Particles, Sonochemistry, Green Solvent, Click Chemistry, Green Solvent
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INTRODUCTION

With the increase in environmental awareness, the development of greener methods for the production of essential chemicals has become crucial. Green chemistry aims to minimize the use of hazardous reagents and waste. The demand for heterocyclic compounds, which are vital for pharmaceutical and crop protection, continues to rise. However, their synthesis faces challenges, such as poor selectivity, weak atom economy, and reliance on harmful chemicals. Traditional methods require harsh conditions and long reaction times, which cause environmental damage. Recent advances in catalytic strategies, such as transition-metal catalysis and multicomponent reactions, offer solutions by improving yields while reducing the environmental impact. However, challenges persist in terms of energy consumption and recyclability. Bridging these gaps is essential for a sustainable chemical synthesis.[6,7] Recent advancements in the integration of nanocatalysts with sonochemical techniques have achieved 85% energy efficiency gains while reducing the reaction time from hours to minutes, transforming the pharmaceutical precursor synthesis. This study addresses sustainable heterocyclic synthesis by developing a method for producing imidazo[1,2-a]pyridine derivatives using silica-supported Cu-Zn nanocomposites under sonochemical conditions. Our approach combined nanocatalysis and ultrasound activation to achieve superior performance under mild conditions. Sonochemical activation creates acoustic cavitation, microscopic bubbles that collapse, generating localized hotspots that accelerate reactions at low bulk temperatures. Silica-supported Cu-Zn nanocomposites provide high surface area and recyclability. This approach reduces the reaction time and operates at lower temperatures, thereby minimizing energy consumption

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and waste. This work supports the UN Sustainable Development Goal 12 by improving resource efficiency and establishing circular catalyst use. This methodology could revolutionize industrial synthesis of heterocyclic compounds while preserving environmental integrity. Key advantages include 85% energy savings versus conventional heating, reaction times reduced to 10-20 minutes, enhanced yields (83-99%), and recyclable nano-catalysts with maintained activity.[8,9] Nano catalysts and sonochemistry have emerged as innovative solutions for achieving efficient reactions. These sonochemical catalysts accelerate reactions under mild conditions, reducing energy consumption and environmental impacts. Nano catalysts enhance the reaction efficiency and are reusable, aligning with the green chemistry principles for creating imidazo[1,2-a] pyridines. These heterocycles exhibit diverse biological activities, including antimicrobial, antiviral, anti-inflammatory, anticancer, and CNS-modulating properties, making them valuable for drug development. Traditional methods rely on harsh conditions and toxic solvents, raising environmental concerns. Silica-supported nano catalysts provide a high surface area, thermal stability, and reusability under mild conditions. Although metal-catalyzed and microwave-assisted reactions have advanced, they lack recyclability. The combination of nano catalysts and sonochemistry enhances the reaction speed through improved mass transfer.[10,11] In this study, a green method was developed for the synthesis of imidazo[1,2-a]pyridine derivatives using silica-supported Cu-Zn nanocomposites under sonocatalytic conditions. Traditional heating methods for heterocyclic compounds involve long reaction times and harsh conditions, which pose environmental challenges. With nano catalysts and sonochemistry, ultrasonic waves create intense heat and pressure pockets, thereby reducing the reaction time. The high surface area and reactivity of nano catalysts offer efficiency and reusability. Unlike conventional methods, which require harsh conditions and corrosive solvents, this approach improves yields under mild conditions while reducing energy consumption. This combination provides an eco-friendly path for synthesizing heterocyclic scaffolds, such as imidazo[1,2-a]pyridines, which exhibit various biological activities but are traditionally synthesized under harsh conditions.[12]

EXPERIMENTAL

Material and Methods

Copper nitrate ($Cu(NO_3)_2$), Zinc nitrate ($Zn(NO_3)_2$), Sodium hydroxide (NaOH), Ethanol (for washing and dispersion), Distilled water, all materials bought from the local market.

Preparation of Cu-Zn Nanocomposite

Copper nitrate and zinc nitrate were separately dissolved in distilled water to prepare their respective solutions, and the zinc nitrate solution was slowly added to the copper nitrate solution under continuous stirring. This ensured the thorough mixing of the metal ions in the solution. To initiate the precipitation of Cu-Zn nanoparticles, a base aqueous solution is used. NaoH was added dropwise to the mixed solution to adjust the pH to approximately 10.5. At this pH, copper and zinc hydroxides precipitated out of the solution. This solution was then subjected to an ultrasonic treatment at 40 kHz for 60 min. Filter the precipitate, wash it thoroughly with distilled water to remove impurities and excess ions, and then dry the collected material in an oven at 90–100°C to reduce the Cu-Zn hydroxide to metallic copper and zinc nanoparticles. Alternatively, heat the dried precursor material in an air atmosphere at temperatures of approximately 300–350°C to reduce it to nanoparticles. Binding with Silica: The Cu-Zn nanocomposite was dispersed in ethanol or another suitable solvent to form a stable dispersion. Sonicate the porous silica in the Cu-Zn dispersion for 2 hours (40kHz), during which the nanocomposite adsorbs onto the surface of silica particles. The mixture was periodically stirred to ensure uniform impregnation. To further enhance the stability and distribution of the Cu-Zn nanoparticles on silica, the composite material can be calcined at 500–600°C for 4 h in an air atmosphere. This step removes any residual organic solvent and ensures that the nanocomposite is securely attached to silica. After the synthesis, the Cu-Zn/SiO_2 composite was characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD) to confirm the successful incorporation of the nanocomposite into silica support.

SEM images provide visual insights into the surface morphology, particle size, shape, and distribution of the catalyst. The description should convey these characteristics clearly and relate them to the catalyst's performance or synthesis conditions. The above image represents a Figure SEM image of Cu-Zn doped

Silica supported catalyst prepared via the common ion effect rather than the ultrasonication method, recorded at 8.9 mm* 5.00 k magnification (scale bar: 200 nm). The image shows uniformly distributed spherical nanoparticles with an average size of ~8.9 nm and a porous surface structure.

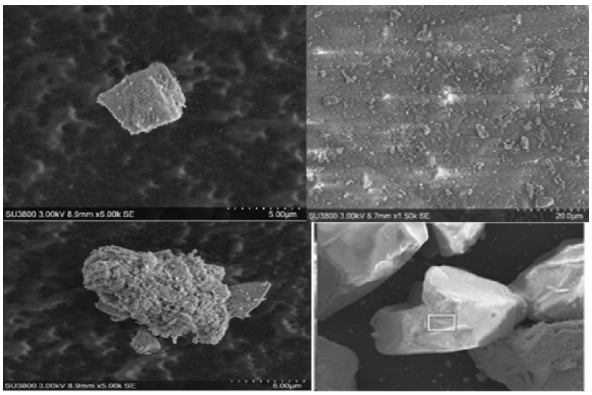


Fig.-1: SEM Images

The elemental composition and distribution of the Cu-Zn-doped silica-supported catalyst were analyzed using SEM-EDX. The EDX spectrum (Fig.-2) confirms the presence of Zn, Cu, and Silica, with atomic percentages of silica, Zn, and Cu, respectively, closely matching the nominal Cl doping level Elemental mapping (Fig.-2) reveals a homogeneous distribution of Cu-Zn across the Silica, consistent with Cu and Zn phases in the XRD pattern (Fig.-3). The uniform near incorporation, combined with the porous morphology observed in SEM images (Fig.-1), likely enhances the catalyst's redox properties, contributing to its superior performance in the reaction.

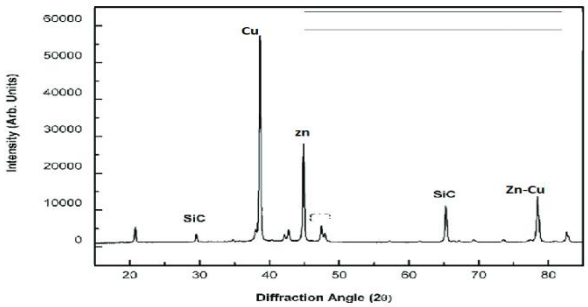


Fig.-3: XRD Images

XRD Data

The XRD pattern of the Cu/Zn catalyst (Fig.-3) confirms the presence of both monoclinic Cu and hexagonal Zn phases, with characteristic diffraction peaks matching JCPDS cards 45-0937 and 36-1451, respectively. The sharp peaks at 2θ = 39.5 indicate a highly crystalline Cu phase, while peaks at 2θ = 46.0 ° confirm the Zn phase. The average crystallite size, calculated using the Scherrer equation for the (111) peak of Cu, is approximately 25 nm. Secondary phases 79.9 °were observed, confirming the sandwich structure Zn-Cu, suggesting high phase purity and minimal lattice strain. The well-defined crystallinity likely contributes to the catalyst's stability during the method of synthesis. The Cu-Zn silica-supported catalyst was characterized using SEM, EDX, and XRD to investigate its morphology, composition, and crystallinity (Fig.-3). The SEM image (Fig.-1) reveals spherical nanoparticles with an average size of 8.9 nm and a porous surface, suggesting a high surface area suitable for photocatalysis. SEM-EDX mapping (Fig.-1,2) shows a uniform distribution of Cu, Zn, and silica with a Cl content consistent with the nominal at doping. The absence of impurities confirms the purity of the catalyst. The XRD pattern (Fig.-3) identifies the anatase Cu-Zn silica-supported phase, with major peaks at 2θ = 39° and 45.0°. A slight peak shift indicates Cu-Zn incorporation into the lattice, and the crystallite size, calculated via the Scherrer equation, is ~8.9 nm, corroborating SEM observations. The combined results highlight a well-dispersed, phase-pure catalyst with uniform doping, which likely contributes to its enhanced visible-light photocatalytic performance.

Preparation of Imidazole [1,2-a]pyridine

Initially, 2-amino pyridine (0.50 mmol), substituted acetophenones (0.60 mmol), silica-supported Cu-Zn (5 mg), and ethanol (2 mL) was taken in a 25-mL RBF and kept and sonicated at 60 °C at 40kHz for 5 minutes under these conditions. Subsequently, the reaction mixture was diluted with 10 mL of distilled water, extracted with ethyl acetate. The organic layer was dried over Na2SO4 and concentrated under reduced pressure, ethyl acetate was removed, and the product was confirmed by melting point,1HNMR, 13C NMR, and Mass spectra.

Recovery of Catalyst Ethanol is used as a Cleaning Solvent for the Catalyst

2-phenylimidazo[1,2-a] pyridine: 1H NMR: δ 7.09 (1H), 7.52-7.55 (3H), 7.66-7.85 (4H),8.69-8.92 (2H). 13C NMR: δ 110 (1C), 116(1C), 124 (1C),125(2C), 125(1C), 128 (2C),128.8 (2C), 134.4 (1C), 145.2 (1C), 145.5 (1C).MS:195.20(M+1)

2-(4-methoxyphenyl)imidazo[1,2-a] pyridine: 1H NMR: δ 3.72 (3H), 7.13-7.27 (3H), 7.66-7.89 (4H),8.64-8.77 (2H),. 13C NMR: δ 55.3 (1C), 110.5 (1C), 113.5 (2C), 116.3 (1C), 124.7 (1C), 125.4 (1C), 128.4 (1C), 129.0 (2C), 132.2 (1C), 145.2 (1C), 145.5 (1C), 160.3 (1C). MS:225.10(M+1)

2-(4-Chlorophenyl) imidazo[1,2-a] pyridine: 1H NMR: δ 7.08 (1H), 7.37 (2H)7.58-7.82 (4H),8.68-8.87 (2H), 13C NMR: δ 110.5 (1C), 116.3 (1C), 124.7 (1C), 125.4 (1C), 127.6 (2C), 128.4 (1C), 129.1 (2C), 132.2 (1C), 134.6 (1C), 145.2 (1C), 145.5 (1C). MS:230.26(M+1)(Provide NMR data with original file and integration)

2-(4-Bromophenyl)imidazo[1,2-a]pyridine: 1H NMR: δ 7.08 (1H), 7.30 (2H), 7.61-7.83 (4H),8.78-8.84 (2H).13C NMR: δ 110.5 (1C), 116.3 (1C), 123.0 (1C), 124.7 (1C), 125.4 (1C), 127.1 (2C), 128.4 (1C), 131.8 (2C), 132.2 (1C), 145.2 (1C), 145.5 (1C).MS:274.01(M+1))(Provide NMR data with original file and integration)

2-(4-ethyl phenyl)imidazo[1,2-a]pyridine: 1H NMR: δ 1.21 (3H), 2.67 (2H), 6.97 (1H), 7.32 (2H), 7.47-7.52 (3H), 7.81 (1H), 8.75-8.86 (2H)). 13C NMR: δ 15.3 (1C), 28.6 (1C), 110.5 (1C), 116.3 (1C), 124.7 (1C), 125.4 (1C), 127.7 (2C), 128.4 (1C), 129.1 (2C), 132.2 (1C), 142.7 (1C), 145.2 (1C), 145.5 (1C). MS:224.12(M+2).

This approach eliminates the need for an anaerobic process and a substantial quantity of metal catalyst, base, and ligands. It presents several practical benefits, including the use of ultrasonic conditions, achieving very brief reaction times akin to click chemistry with a nano-sandwich catalyst, and compatibility with a range of functional groups, all while functioning efficiently in a normal atmosphere.

Table-1: Previously Reported Method for Synthesis of Imidazole [1,2-a]pyridine (Top Research Papers of the Last 5 Years, Collected from Science Direct Data for Derivative)

S. No.	Catalyst/Solvent	Method	Time (Min)/Yield(%)	Reference(Year)
1	K_2CO_3/ACN	MW	20/80	12(2024)
2	$NaHCO_3$/Chauffage/Ethanol	Convectional	70/90	13(2024)
3	Uv+CuCl/MeOH	UVlight	15/79	14(2024)
4	Zinc+iodine/Chlrobenzene	Conventional	1440/74	15(2023)
5	Cu+SiO2/Toluene	Conventional	2880/64-81	16(2019)
6	NBS/PEG-400	Ultrasonic	60/98	17(2023)
7	Molecular iodine/Graphene oxide	RT under starring	720/79-85	18(2022)
8	I2	Electric grinding	720/72-82	19(2020)

Additionally, this method is straightforward to implement, requires no additives, and is perfect for the efficient synthesis of structurally significant motifs like 2,3-diarylimidazo[1,2-a]pyridines.

RESULTS AND DISCUSSION

The synthesis of imidazole [1,2-a]pyridine using silica-supported Cu-Zn Nps catalysts has gained attention

because of its efficient reaction mechanism, improved catalytic activity, and recyclability. In this reaction, nanoparticles act as a support for the active metal species that drive the reaction. First, any reaction solvent plays a crucial role in the yield in terms of reaction time and environmental impact; therefore, we first determined the solvent suitability for this catalyst and condition when comparing ethanol to other solvents like DMF dimethylformamide (DMF), dioxane, hexane, acetonitrile (ACN), toluene, and DES (Deep Eutectic Solvent). Ethanol can be considered a greener option in many respects. Here, we analyze why ethanol may be considered a better green solvent, including the results and discussion based on various environmental, economic, and safety factors. Table-1 indicates that ethanol gave a 90% yield at 60 °C within 5 min in the ultrasonic cavitation method; thus, we optimized the catalyst quality and time to obtain a good yield.

Table-2: Optimization of the Reaction Conditions

Ultrasonic cavitation					
Entry	Catalyst (mol%)	Solvent	Temp (°C)	Time (min)	Yield[b] (%)
1	Silica supp. Cu-Zn (5)	DMSO	40	15	NR
2	Silica supp. Cu-Zn (5)	DMF	50	15	NR
3	Silica supp. Cu-Zn (5)	CH_3CN	50	15	NR
4	Silica supp. Cu-Zn (5)	Ethanol	50	05	92
5	Silica supp. Cu-Zn (5)	DCE	50	15	NR
6	Silica supp. Cu-Zn (5)	Toluene	50	15	44
7	Silica supp. Cu-Zn (5)	THF	50	15	43
8	Silica supp. Cu-Zn (5)	1,4-Dioxane	50	15	45

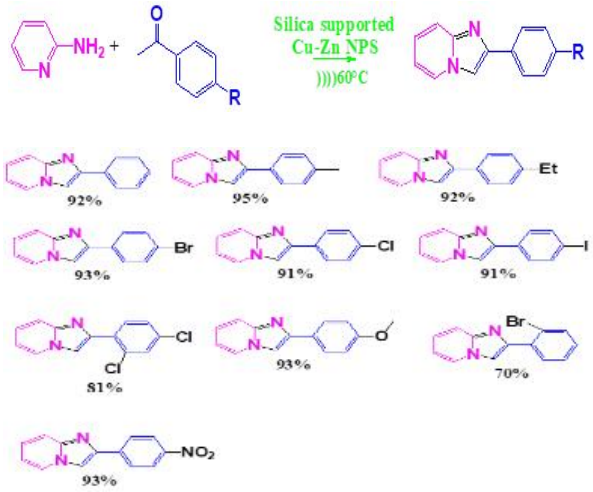
Reaction conditions: 2-aminopyridine, acetophenone, solvent (2-10 mL). Isolated yields by recrystallization. Note: Bold indicates the optimal condition. However, there are also situations where other solvents, such as DMF, Dioxane, and ACN, might be preferred in certain applications because of their stronger solvency power or compatibility with these specific reactions. Ethanol, while versatile, might not always be the best choice for all industrial or chemical processes, particularly where highly nonpolar or strong solvents are needed. Overall, ethanol stands out as a greener solvent than DMF, Dioxane, Hexane, ACN, Toluene, and DES because of its renewability, low toxicity, biodegradability, and ease of recycling. However, their performance in specific applications may not always match that of more specialized solvents. The use of ethanol as a green solvent depends on the balance between environmental sustainability and performance needs for a given chemical process, resulting in a good yield in a very short time and a good yield with easy workup. In Table-2, we apply various types of catalysts under various conditions to determine the comparison between the conventional method and the ultrasonic cavitation method, and we applied various parameters for optimization.

Table-3: Optimization of Catalyst for the Reaction

S. No.	Catalyst amount (mg)	Time (min) /temp(°C)	Conventional method	Ultrasonic method
1	1	60/70	52	65
2	1	90/90	50	63
3	2	60/70	59	67
4	2	90/90	59	65
5	3	90/90	67	72
6	4	90/90	69	73
7	5	90/90	72	94
8	5	60/70	75	93
9	5	15/60	75	94
10	5	05/60	73	95
11	10	05/60	72	92
12	15	05/60	76	92
13	20	05/60	75	90
14	25	05/60	72	89

Stoichiometric ratio of all the reactants with catalyst in Ethanol at Various temperatures and both conventional and Ultrasonic methods Table-3 indicates Sr number 10, catalyst amount 5 mg for 5 min, and 60°C temperature

conventional method gave 73% yield, and the ultrasonic method gave 95% yield. We observed that the entire reaction optimization module required more comparison with the ultrasonic method. After solvent selection and catalyst quality optimization, the same conditions were applied to all acetophenones to determine the acceptability of the reaction conditions.



[a]Reaction conditions: 2-amino pyridine (0.25 mmol), various acetophenones (0.25 mmol), and Silica supported CU-Zn Nps (5-10mol%) in Ethanol (5-10 mL) at 60 °C for 5-min stirring. Isolated yields by recrystallization.

Scheme-1

Table-4: Recovery of Catalyst

Entry	Cycle Num	Yield
01	1	98
02	2	98
03	3	97
04	4	95
05	5	94
06	6	89
07	7	75
08	8	60

As observed during the recovery of the catalyst, a catalyst of up to 3-4 cycles gave a constant yield after each cycle catalyst was washed with ethanol and hexane once, which reduced the efficiency of the catalyst after cycle 4.

CONCLUSION

The developed methodology utilizes silica-supported Cu-Zn nanocomposites as catalysts under sonochemical conditions in ethanol, presenting a highly efficient, sustainable, and environmentally benign approach for the synthesis of imidazole[1,2-a]pyridine derivatives as per SDG-12 (Responsible Consumption and Production). This catalytic system offers several advantages, including mild reaction conditions, high catalytic activity, recyclability, and environmental compatibility. The synergistic interaction between Cu and Zn, coupled with the thermal stability and recyclability of the silica support, renders this catalyst a practical and eco-friendly alternative to conventional harsh catalytic systems. The employment of ethanol as a green solvent and the use of ultrasound to enhance reaction rates further underscore the method's alignment with green chemistry principles, achieving high yields of 91-96% with minimal environmental impact. Overall, this approach represents a significant advancement in the sustainable organic synthesis of complex heterocycles, contributing positively to both economic and environmental objectives.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related *to SDG-12:Responsible Consumption and Production.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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