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Design and Microwave-Assisted Green Synthesis of Substituted Benzimidazole Derivatives

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Abstract : *Benzimidazole is an important nitrogen-containing heterocyclic scaffold with considerable significance in medicinal and pharmaceutical chemistry because of its diverse biological properties. Benzimidazole derivatives have attracted substantial attention as potential bioactive compounds and as valuable building blocks for the development of biologically and pharmaceutically useful molecules. In the present study, benzimidazole derivatives were synthesized using microwave irradiation as a rapid and environmentally friendly synthetic approach. The microwave-assisted method offers the advantages of shorter reaction times, improved reaction efficiency, and good product yields compared with conventional synthetic procedures. The synthesized compounds were evaluated for their antimicrobial activity to investigate their potential biological applications. The structures of the newly synthesized derivatives were characterized using various spectroscopic techniques, including infrared (IR), proton nuclear magnetic resonance (^1H NMR), and UV–Visible spectroscopy. The study demonstrates the usefulness of microwave-assisted synthesis as an efficient approach for the preparation of benzimidazole derivatives with potential pharmaceutical and biological applications.*

Key Words: Benzimidazole; Heterocyclic compounds; Microwave-assisted synthesis; Green chemistry; Antimicrobial activity; Spectroscopic characterization; Pharmaceutical chemistry.

1. INTRODUCTION

Benzimidazole is an important nitrogen-containing aromatic heterocyclic compound consisting of a benzene ring fused with an imidazole ring. The presence of two nitrogen atoms within the imidazole portion gives this bicyclic scaffold distinctive electronic and chemical properties, while the fused-ring system provides considerable scope for structural modification. Because different substituents can be introduced at several positions of the benzimidazole nucleus, a wide variety of derivatives can be designed with different physicochemical and biological characteristics.

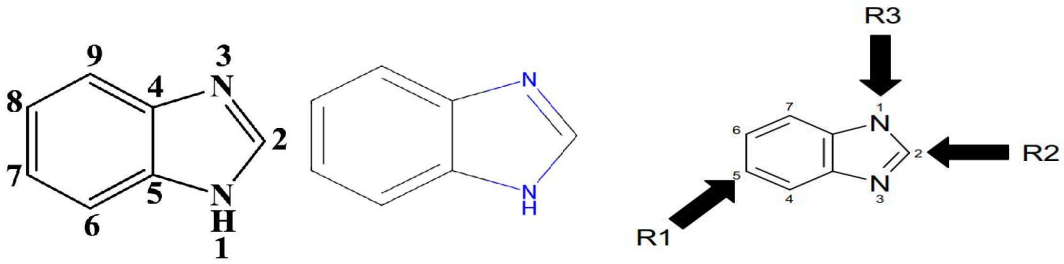
The biological significance of the benzimidazole nucleus can be traced to early observations in the mid-twentieth century. In 1944, Woolley proposed that benzimidazole possesses structural features resembling those of purine systems and suggested that this similarity could be associated with biological activity [1]. This observation encouraged further investigation into the biological role of benzimidazole-containing molecules. Subsequently, Brink and co-workers identified 5,6-dimethylbenzimidazole as a degradation product associated with vitamin B12 and investigated related compounds exhibiting vitamin B12-like biological properties [2,3]. These early findings established an important connection between the benzimidazole framework and biologically relevant molecular systems and stimulated extensive research into its chemistry.

Over subsequent decades, benzimidazole has developed into a valuable pharmacophore in medicinal chemistry. Its ability to participate in hydrogen bonding, π–π interactions, electrostatic interactions, and other molecular interactions enable appropriately substituted derivatives to interact with a broad range of biological targets. Modification of the

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benzimidazole nucleus can alter properties such as lipophilicity, solubility, metabolic stability, target affinity, and selectivity. Consequently, systematic structural modification of this scaffold has become an important strategy for developing compounds with improved pharmacological profiles.

The fundamental structure of benzimidazole is composed of a benzene moiety fused to an imidazole ring, and its conventional numbering system is shown in Figure 1. The nitrogen atoms of the imidazole ring play an important role in determining the chemical behaviour of the molecule and its interaction with biological macromolecules. Substitution at different positions of the aromatic and imidazole portions can significantly influence the biological properties of the resulting derivatives. This structural flexibility makes benzimidazole particularly useful for exploring structure–activity relationships (SAR) and designing molecules directed toward specific therapeutic targets.



Benzimidazole-related structures are also found in biologically important natural systems. One notable example is N-ribosyl-5,6-dimethylbenzimidazole, which functions as an axial ligand for the cobalt centre of vitamin B12. This naturally occurring association further illustrates the biological relevance of the benzimidazole framework and provides an important example of how this heterocycle can participate in biologically essential molecular structures.

The medicinal importance of benzimidazole derivatives arises from their broad spectrum of biological activities. Research has demonstrated that appropriately substituted benzimidazole compounds can exhibit antimicrobial, antiparasitic, antiviral, anticancer, antidiabetic, analgesic, antihistaminic, and other pharmacological effects [4–12]. Their biological activities are generally influenced by the nature, electronic properties, and position of substituents attached to the parent nucleus. This versatility has led to the incorporation of benzimidazole-based structures into numerous pharmaceutical research programs.

II. PHARMACEUTICAL AND BIOLOGICAL SIGNIFICANCE

Benzimidazole derivatives have attracted particular interest as antimicrobial and antiparasitic agents. Structural modifications of the parent heterocycle can produce compounds capable of interfering with essential biological processes in microorganisms and parasites. This property has made the benzimidazole scaffold valuable in the search for new therapeutic agents, particularly in view of the increasing problem of antimicrobial resistance.

The scaffold has also demonstrated considerable promise in anticancer drug discovery. Different benzimidazole derivatives have been investigated for their ability to influence molecular pathways associated with cancer-cell proliferation, cell-cycle regulation, apoptosis, and other processes involved in tumour development. The possibility of modifying the heterocyclic nucleus to improve target selectivity has further increased its importance in contemporary medicinal chemistry.

In addition, benzimidazole-containing molecules have been investigated for applications in metabolic disorders, including diabetes, as well as for analgesic, antiviral, antihistaminic, neurological, endocrinological, and ophthalmological applications [4–12]. Such pharmacological diversity demonstrates that benzimidazole should not be regarded merely as a single therapeutic class but rather as a versatile chemical platform capable of generating compounds with different mechanisms of biological action.

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III. IMPORTANCE OF BENZIMIDAZOLE IN SYNTHETIC AND MEDICINAL CHEMISTRY

From a synthetic perspective, the benzimidazole nucleus provides an attractive platform for the preparation of structurally diverse derivatives. Classical approaches generally involve the cyclization of suitable o-phenylenediamine derivatives with aldehydes, carboxylic acids, or related carbonyl compounds. However, the increasing demand for rapid, economical, and environmentally responsible synthetic procedures has encouraged the development of improved methodologies.

Among these approaches, microwave-assisted synthesis has received considerable attention because it can accelerate chemical transformations and, under suitable conditions, provide good yields within significantly shorter reaction periods. The use of microwave irradiation can therefore be advantageous in the preparation of benzimidazole derivatives, particularly when rapid synthesis and efficient reaction conditions are desired. In combination with appropriate catalysts and environmentally preferable reaction media, microwave technology can contribute to the development of greener synthetic protocols.

The present work therefore focuses on the synthesis of benzimidazole derivatives using microwave irradiation, followed by evaluation of their biological potential. The synthesized compounds can be characterized using spectroscopic techniques such as infrared (IR), ^1H nuclear magnetic resonance (^1H NMR), and UV–Visible spectroscopy to establish their structural features. Their antimicrobial activity can subsequently be investigated to determine whether the synthesized molecules exhibit promising biological effects.

IV. RATIONALE OF THE PRESENT STUDY

The continuing emergence of resistant microorganisms and the demand for new biologically active molecules emphasize the importance of developing efficient synthetic routes for heterocyclic compounds. Benzimidazole is particularly attractive in this context because its molecular framework permits extensive chemical modification and has already demonstrated a broad range of pharmacological activities.

Therefore, the combination of benzimidazole chemistry, microwave-assisted synthesis, spectroscopic characterization, and antimicrobial screening provides a useful approach for investigating new derivatives with potential pharmaceutical applications. The study may contribute to the development of efficient synthetic methodology while also providing information about the relationship between the chemical structure of benzimidazole derivatives and their antimicrobial properties.

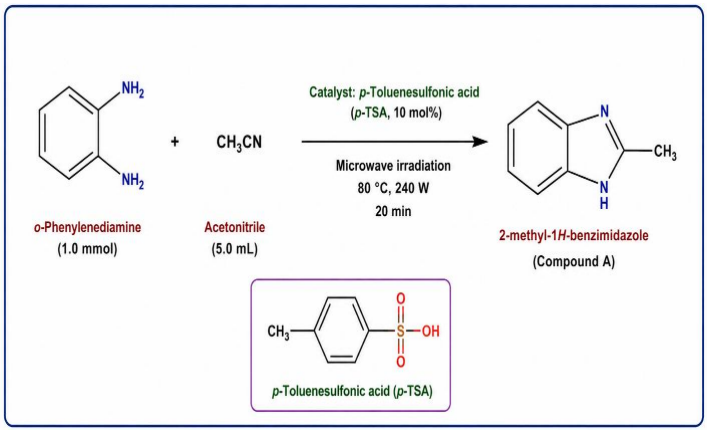
V. SYNTHESIS OF 2-METHYL-1H-BENZIMIDAZOLE (COMPOUND A)

A mixture of o-phenylenediamine (1,2-diaminobenzene) and acetonitrile was taken in a suitable microwave-compatible reaction vessel. A small quantity of p-toluenesulfonic acid (p-TSA) was added as an acid catalyst, and the reaction mixture was thoroughly mixed. The reaction vessel was subjected to microwave irradiation at 100 °C for 10–15 min under controlled conditions. The use of p-TSA as a catalyst was selected to facilitate the cyclization of o-phenylenediamine with acetonitrile and to avoid the use of a larger quantity of strong mineral acid.

The progress of the reaction was monitored periodically by thin-layer chromatography (TLC) using an appropriate solvent system. After completion of the reaction, the mixture was allowed to cool to room temperature and was subsequently poured slowly into approximately 50 mL of ice-cold water with continuous stirring. The resulting solid product was separated by filtration and washed thoroughly with distilled water to remove the catalyst and water-soluble impurities.

The crude product was dried and purified by recrystallization from a suitable solvent or solvent mixture. The purified compound was obtained as 2-methyl-1H-benzimidazole (Compound A). The synthesized product was subjected to spectroscopic characterization using IR, ^1H NMR, and UV–Visible spectroscopy.

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VI. EXPERIMENTAL PROCEDURE

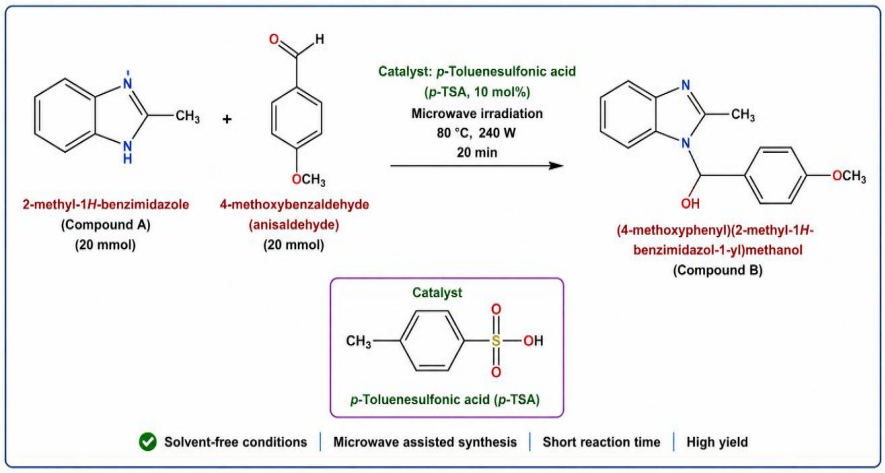
A mixture of 2-methyl-1H-benzimidazole (Compound A, 20 mmol) and 4-methoxybenzaldehyde (anisaldehyde, 20 mmol) was placed in a suitable microwave reaction vessel. p-Toluenesulfonic acid (p-TSA) was added as a catalyst, and the reactants were thoroughly mixed. The reaction mixture was subjected to microwave irradiation at 80 °C for approximately 20 min under controlled conditions.The progress of the reaction was monitored by thin-layer chromatography (TLC) using an appropriate solvent system. After completion of the reaction, the reaction mixture was allowed to cool to room temperature. It was then slowly poured into approximately 50 mL of ice-cold water with continuous stirring. The precipitated solid was separated by filtration and washed with distilled water to remove the water-soluble impurities and residual catalyst.

The obtained crude product was dried and subsequently purified by recrystallization from a suitable solvent or solvent system. The purified product was obtained as (4-methoxyphenyl) (2-methyl-1H-benzoimidazol-1-yl) methanol (Compound B). The synthesized compound was further subjected to characterization using IR, ^1H NMR and UV–Visible spectroscopic techniques.

Synthesis of (4-Methoxyphenyl) (2-methyl-1H-benzoimidazol-1-yl) methanol (Compound B): Compound A (2-methyl-1H-benzimidazole, 20 mmol) and 4-methoxybenzaldehyde (20 mmol) were mixed in the presence of p-toluenesulfonic acid (p-TSA) as a catalyst. The reaction mixture was irradiated under microwave conditions at 80 °C for 20 min. Reaction completion was monitored by TLC. After completion, the mixture was cooled to room temperature and poured into ice-cold water (50 mL). The separated solid was collected by filtration, washed with water (10 mL), and dried. The crude product was purified by recrystallization using a suitable solvent to obtain Compound B. The purified compound was characterized by IR, ^1H NMR and UV–Visible spectroscopy.

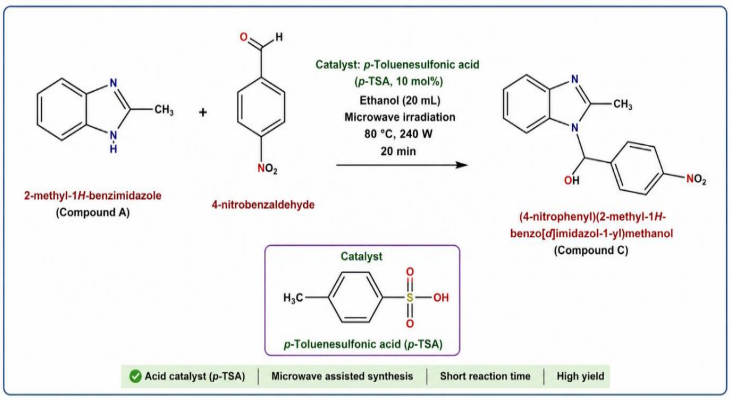
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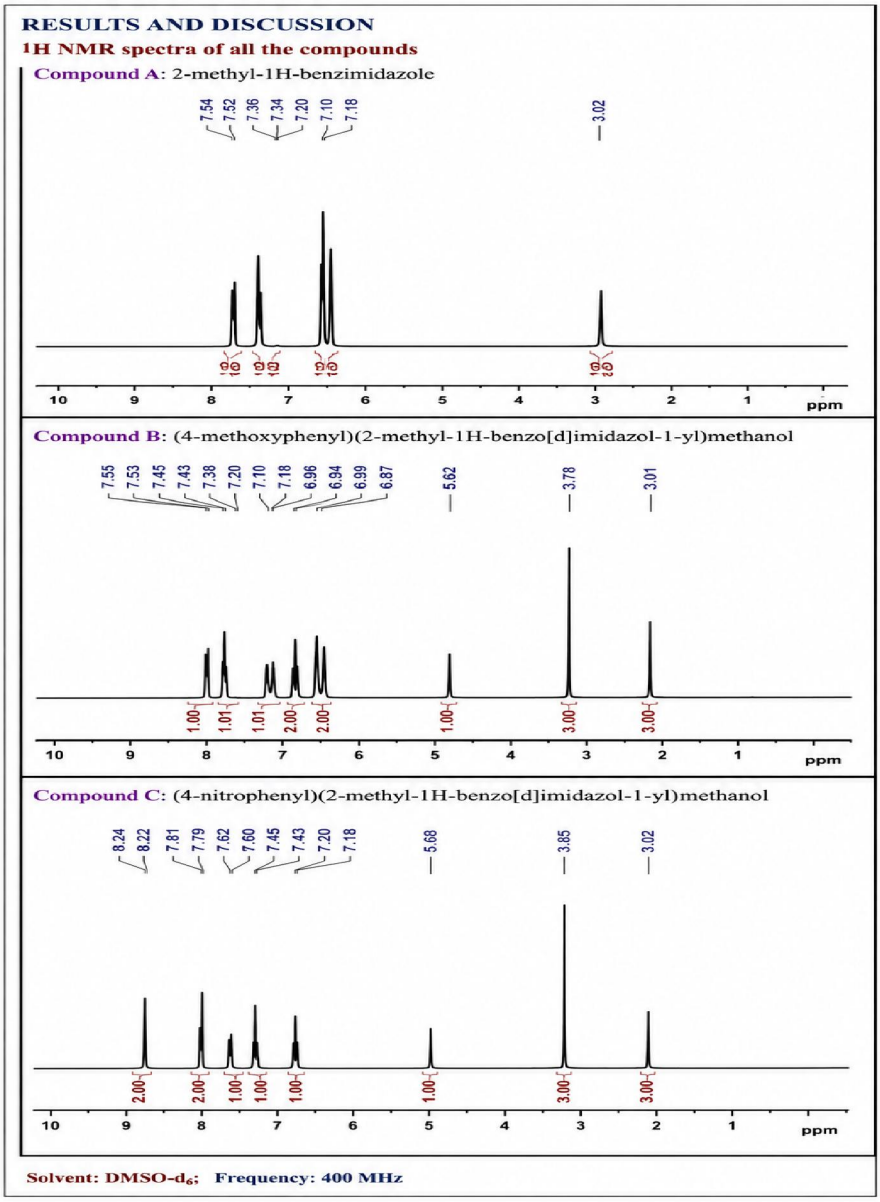
VII. SYNTHESIS OF COMPOUND C
(4-NITROPHENYL) (2-METHYL-1H-BENZO[D]IMIDAZOL-1-YL) METHANOL

Compound A (2-methyl-1H-benzimidazole, 0.435 g) and 4-nitrobenzaldehyde (0.523 g) were taken in a suitable microwave reaction vessel, followed by the addition of 20 mL ethanol. p-Toluenesulfonic acid (p-TSA) was added as an acid catalyst, and the reaction mixture was thoroughly mixed. The reaction mixture was then subjected to microwave irradiation at 80 °C and 240 W for 20 min. The progress of the reaction was monitored by thin-layer chromatography (TLC) using an appropriate solvent system. After completion of the reaction, the mixture was allowed to cool to room temperature. It was subsequently poured slowly into 50 mL of ice-cold water with continuous stirring. The solid product formed was separated by filtration and washed with 10 mL of distilled water to remove soluble impurities and residual catalyst. The obtained solid was dried and purified by recrystallization using a suitable solvent to afford the desired benzimidazole derivative, Compound C.



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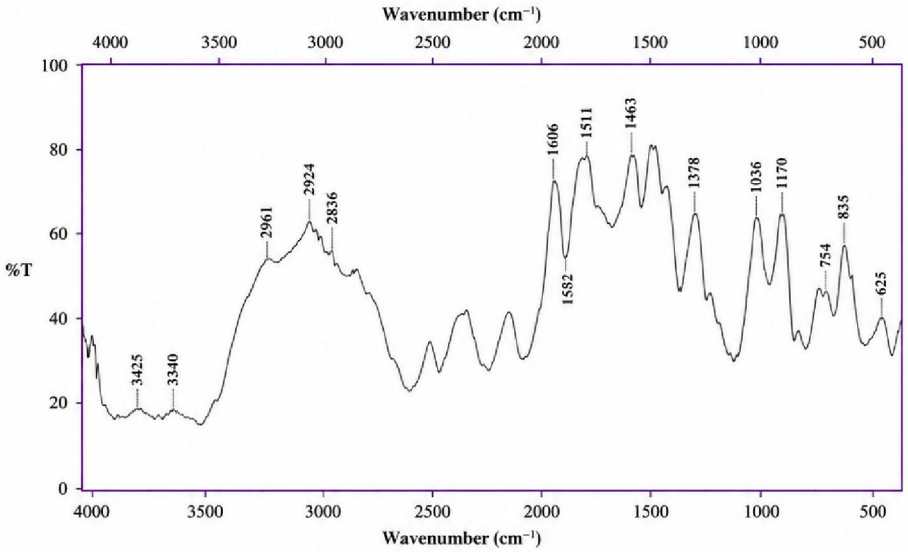
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RESULTS AND DISCUSSION

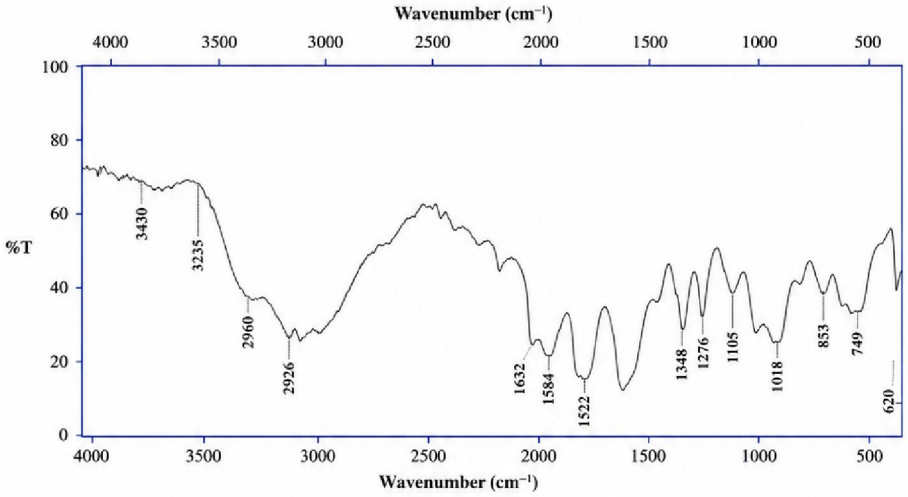
IR Characterization of compound B and C

a) IR spectrum of compound B : (4-methoxyphenyl)(2-methyl-1*H*-benzo[d]imidazol-1-yl)methanol



Interpretation: Broad band at ~3425 cm^{-1} (O–H), bands at ~2961–2836 cm^{-1} (C–H stretching), strong bands at ~1606 and ~1511 cm^{-1} (C=N and aromatic C=C), band at ~1170 cm^{-1} (C–O stretching of methoxy), and band at ~1036 cm^{-1} (C–O).

b) IR spectrum of compound C : (4-nitrophenyl)(2-methyl-1*H*-benzo[d]imidazol-1-yl)methanol



Interpretation: Broad band at ~3430 cm^{-1} (O–H), bands at ~2960–2926 cm^{-1} (C–H stretching), strong asymmetric and symmetric NO_2 bands at ~1522 cm^{-1} and ~1348 cm^{-1}, band at ~1276 cm^{-1} (C–N stretching), and band at ~620 cm^{-1} (aromatic C–H out-of-plane bending).

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VII. CONCLUSION

The present study highlights the importance of the benzimidazole nucleus as a versatile and pharmacologically significant heterocyclic scaffold with considerable potential in medicinal chemistry. The structural features of benzimidazole permit the introduction of different substituents, thereby providing opportunities to develop derivatives with diverse biological properties. In particular, the antimicrobial potential of substituted benzimidazoles makes this class of compounds relevant to the continuing search for new bioactive molecules.

In the present work, substituted benzimidazole derivatives were synthesized using microwave-assisted methodology. The use of microwave irradiation provided a convenient approach for carrying out the reactions in relatively short reaction times and under controlled conditions. Incorporation of an appropriate catalyst further supported the development of an efficient synthetic protocol. The synthesized compounds were isolated, purified by recrystallization, and characterized using IR, ^1H NMR, and UV–Visible spectroscopic techniques.

The synthesized derivatives demonstrated the feasibility of functionalizing the benzimidazole nucleus with different aromatic substituents. The spectroscopic findings supported the formation of the expected structural framework and provided useful information regarding the characteristic functional groups present in the synthesized compounds. The antimicrobial screening further indicated that structural modification of the benzimidazole ring can influence its biological behaviour.

Overall, the study demonstrates that microwave-assisted synthesis combined with suitable catalytic conditions represents a useful approach for the preparation of substituted benzimidazole derivatives. The combination of rapid synthesis, structural diversification, and biological evaluation provides a promising platform for further investigation. Future studies involving a larger series of derivatives, detailed structure–activity relationship investigations, optimization of reaction conditions, and broader biological screening may help identify benzimidazole compounds with improved antimicrobial activity and potential pharmaceutical applications.

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