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Green Sonochemical Synthesis of Benzimidazole Derivatives Using Toluene: An Eco-Friendly and Sustainable Approach

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Abstract: *Benzimidazole derivatives are an important class of nitrogen-containing heterocyclic compounds possessing diverse biological and pharmaceutical activities such as antimicrobial, antifungal, antiviral, anticancer, anti-inflammatory, and antiparasitic properties. Due to their extensive medicinal importance, the synthesis of benzimidazole derivatives has attracted considerable attention in modern organic chemistry. Conventional synthetic methods often involve harsh reaction conditions, toxic solvents, longer reaction times, and lower product selectivity, thereby limiting their industrial and environmental applicability.*

In the present investigation, an efficient green sonochemical methodology has been developed for the synthesis of substituted benzimidazole derivatives using o-phenylenediamine and various aromatic aldehydes in toluene medium under ultrasonic irradiation. The sonochemical approach utilizes acoustic cavitation generated by ultrasonic waves, resulting in enhanced molecular interactions, accelerated reaction rates, and improved product formation under mild conditions.

The reactions were completed within 5–16 minutes under ultrasonic irradiation and afforded benzimidazole derivatives in excellent yields ranging from 75–95%. The synthesized compounds were characterized using UV–Visible spectroscopy, Fourier Transform Infrared Spectroscopy (FTIR), and Proton Nuclear Magnetic Resonance (^{1}H-NMR) spectroscopy. Spectral analyses confirmed successful cyclization and formation of the benzimidazole nucleus.

The study demonstrated that aromatic aldehydes containing electron-withdrawing substituents such as nitro, bromo, fluoro, and chloro groups exhibited faster reaction rates and higher yields compared to electron-donating substituents. The developed sonochemical method offers several advantages including eco-friendly conditions, reduced reaction time, operational simplicity, high product purity, lower energy consumption, and improved synthetic efficiency.

The present work establishes ultrasonic irradiation as a powerful green chemistry tool for the rapid and sustainable synthesis of biologically important benzimidazole derivatives with potential pharmaceutical applications.

Keywords: Benzimidazole, sonochemistry, ultrasonic irradiation, green synthesis, heterocyclic compounds, toluene, sustainable chemistry.

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I. INTRODUCTION

Heterocyclic compounds constitute one of the most significant and rapidly developing branches of organic chemistry. These compounds contain one or more heteroatoms such as nitrogen, oxygen, or sulfur within a cyclic ring system along with carbon atoms. Among various heterocyclic compounds, nitrogen-containing heterocycles occupy a prominent position because of their widespread occurrence in biologically active molecules, pharmaceuticals, agrochemicals, natural products, dyes, and polymers.

Nitrogen heterocycles are fundamental structural components of several naturally occurring biomolecules including amino acids, nucleic acids, alkaloids, enzymes, hormones, vitamins, and coenzymes. Purine and pyrimidine bases present in DNA and RNA are classic examples of nitrogen-containing heterocyclic systems responsible for genetic information storage and transmission.

Among the various nitrogen heterocycles, benzimidazole derivatives have attracted tremendous attention because of their remarkable biological and medicinal significance. Benzimidazole is a bicyclic aromatic heterocyclic compound formed by fusion of a benzene ring with an imidazole ring at the 4 and 5 positions. The benzimidazole nucleus possesses two nitrogen atoms within the imidazole ring, contributing significantly to its unique physicochemical and biological properties.

Benzimidazole derivatives exhibit a broad spectrum of pharmacological activities including:

- Antimicrobial activity
- Antifungal activity
- Antiviral activity
- Anticancer activity
- Antiulcer activity
- Antihypertensive activity
- Anthelmintic activity
- Anti-inflammatory activity
- Antioxidant activity

Several benzimidazole-based drugs such as albendazole, mebendazole, thiabendazole, omeprazole, pantoprazole, and lansoprazole are widely used in medicinal therapy for treatment of parasitic infections, gastric ulcers, microbial diseases, and acid reflux disorders.

The benzimidazole ring system also possesses significant industrial applications in corrosion inhibitors, fluorescent materials, dyes, agrochemicals, and coordination chemistry. Because of these versatile applications, the synthesis of benzimidazole derivatives remains an active area of research in synthetic organic chemistry.

Traditional synthetic methods for benzimidazole derivatives generally involve condensation of o-phenylenediamine with carboxylic acids, aldehydes, or their derivatives under strongly acidic or oxidative conditions. However, many conventional methods suffer from several drawbacks such as:

- Harsh reaction conditions
- Toxic solvents
- Longer reaction time

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- High energy consumption
- Low atom economy
- Difficult purification procedures
- Environmental pollution

Therefore, the development of efficient, sustainable, and environmentally benign synthetic methodologies has become increasingly important.

Green chemistry aims to design chemical processes that minimize hazardous substances, reduce waste generation, conserve energy, and improve environmental compatibility. One of the important modern approaches in green chemistry is sonochemical synthesis.

Sonochemistry involves the use of ultrasonic irradiation to accelerate chemical reactions through acoustic cavitation. Acoustic cavitation refers to the formation, growth, and implosive collapse of microscopic bubbles in liquids under ultrasonic waves. This collapse generates localized high temperature and pressure zones, leading to enhanced reaction kinetics and molecular interactions.

Sonochemical methods provide several advantages:

- Shorter reaction time
- Eco-friendly conditions
- Improved product yield
- Lower energy consumption
- Cleaner reaction profile
- Enhanced selectivity
- Simple work-up procedure
- Reduced solvent usage

Because of these advantages, ultrasonic irradiation has emerged as a highly efficient green chemistry technique for heterocyclic synthesis.

The present work therefore focuses on the green sonochemical synthesis of benzimidazole derivatives using substituted aromatic aldehydes in toluene medium under ultrasonic irradiation.

II. OBJECTIVES OF THE STUDY

The main objectives of the present investigation are:

1. To synthesize substituted benzimidazole derivatives using sonochemical methodology.
2. To develop a rapid and eco-friendly synthetic route.
3. To study the influence of different aromatic substituents on reaction efficiency.
4. To characterize synthesized compounds using spectroscopic techniques.
5. To evaluate the effectiveness of ultrasonic irradiation in heterocyclic synthesis.
6. To establish a sustainable alternative to conventional synthetic methods.

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III. LITERATURE REVIEW

Several researchers have reported various synthetic methods for benzimidazole derivatives. Phillips (1928) first synthesized benzimidazoles by condensation of o-phenylenediamine with carboxylic acids under acidic conditions.

Grimmett and Katritzky extensively studied the chemistry and biological importance of benzimidazole compounds. Recent developments have focused on environmentally sustainable methodologies such as:

- Microwave-assisted synthesis
- Solvent-free synthesis
- Nanocatalyst-mediated synthesis
- Ultrasound-assisted synthesis
- Green catalytic approaches

Sharma and Joshi (2012) reported that ultrasonic irradiation significantly accelerates heterocyclic synthesis through acoustic cavitation effects. Deshmukh et al. (2015) demonstrated efficient sonochemical synthesis of heterocyclic compounds with reduced reaction times and improved yields.

Recent investigations have shown that green sonochemical approaches provide sustainable alternatives to conventional heating methods and align effectively with modern principles of green chemistry.

IV. MATERIALS AND METHODS

4.1 Chemicals Used

The chemicals used in the present study included *o*-phenylenediamine, various aromatic aldehydes, toluene, glacial acetic acid, ethanol, and distilled water. All chemicals used were of analytical reagent grade and were used without further purification throughout the experimental work.

4.2 Apparatus and Instruments

The experimental work was carried out using various laboratory instruments and apparatus. An ultrasonic bath operating at 35–40 kHz was used to facilitate ultrasonic-assisted synthesis. Round-bottom flasks and magnetic stirrers were used for carrying out the reaction under controlled conditions. Thin layer chromatography (TLC) plates and a UV chamber were used to monitor the progress of the reaction. Product isolation was performed using a Buchner funnel and vacuum filtration assembly. The synthesized compounds were characterized using FTIR spectroscopy, UV–Visible spectroscopy, and ^{1}H-NMR spectroscopy for structural confirmation and analysis.

V. EXPERIMENTAL PROCEDURE

General Procedure for Sonochemical Synthesis

A mixture of o-phenylenediamine (1 mmol) and substituted aromatic aldehyde (1 mmol) was taken in a clean 50 mL round-bottom flask containing 15–20 mL of toluene. Two to three drops of glacial acetic acid were added as catalyst.

The reaction flask was placed in an ultrasonic bath operating at 35–40 kHz frequency and maintained at 50–60°C. The reaction mixture was subjected to ultrasonic irradiation for 5–16 minutes depending on the nature of the aromatic aldehyde.

The progress of the reaction was monitored using Thin Layer Chromatography (TLC) with hexane:ethyl acetate (7:3) as solvent system.

After completion of the reaction, the mixture was cooled to room temperature and poured into crushed ice or cold distilled water. The precipitated benzimidazole derivative was filtered under vacuum, washed with water, dried, and recrystallized from ethanol.

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VI. . REACTION MECHANISM

The reaction proceeds through the following steps:

1. Condensation of aromatic aldehyde with o-phenylenediamine.
2. Formation of Schiff base intermediate.
3. Intramolecular cyclization under ultrasonic irradiation.
4. Oxidative aromatization yielding benzimidazole derivative.

Ultrasonic irradiation accelerates the reaction by generating localized high temperature and pressure conditions through acoustic cavitation.

VII. OBSERVATION TABLE

Entry	Aromatic Aldehyde	Substituent	Time (min)	Yield (%)	Melting Point (°C)
1	Benzaldehyde	H	16	78	289–291
2	Anisaldehyde	4-OCH_3	16	75	232–234
3	4-Methylbenzaldehyde	4-CH_3	5	75	222–224
4	4-Chlorobenzaldehyde	4-Cl	10	78	289–291
5	4-Fluorobenzaldehyde	4-F	5	80	243–245
6	3-Bromobenzaldehyde	3-Br	5	85	263–265
7	Furan-2-carbaldehyde	Furan	5	90	283–285
8	Cinnamaldehyde	α,β-Unsaturated	8	95	199–201
9	3-Nitrobenzaldehyde	3-NO_2	7	90	309–310

VIII. SPECTRAL CHARACTERIZATION

8.1 UV–Visible Spectral Analysis

The synthesized benzimidazole derivatives showed characteristic absorption bands corresponding to π→π* and n→π* electronic transitions.

λmax (nm)	Transition	Assignment
230–250	π→π*	Aromatic ring transitions
270–290	π→π*	Conjugated benzimidazole system
320–360	n→π*	Nitro group and nitrogen transitions

Nitro-substituted derivatives exhibited bathochromic shift due to enhanced conjugation and electron-withdrawing effects.

8.2 FTIR Spectral Analysis

Characteristic FTIR absorption peaks:

Wavenumber (cm^{-1})	Functional Group
3400–3200	N–H stretching
3100–3000	Aromatic C–H stretching
1620–1580	C=N stretching
1510–1490	Aromatic C=C stretching
1530–1515	NO_2 asymmetric stretching
1350–1330	NO_2 symmetric stretching

The disappearance of aldehydic carbonyl peak and appearance of C=N stretching band confirmed successful cyclization.

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8.3 ^{1}H-NMR Spectral Analysis

Characteristic proton signals observed:

Chemical Shift δ (ppm)	Assignment
12.40–12.80	NH proton
8.20–8.50	Aromatic proton near NO_2
7.20–8.10	Aromatic ring protons

The downfield NH signal confirmed formation of benzimidazole nucleus.

IX. RESULTS AND DISCUSSION

The present study successfully demonstrated efficient green sonochemical synthesis of benzimidazole derivatives using substituted aromatic aldehydes under ultrasonic irradiation conditions.

The sonochemical approach significantly reduced reaction time compared to conventional methods. Most reactions were completed within 5–16 minutes while providing excellent yields ranging from 75–95%.

Electron-withdrawing substituents such as:

- Nitro (–NO_2)
- Bromo (–Br)
- Fluoro (–F)
- Chloro (–Cl)

enhanced electrophilicity of aldehydic carbon and facilitated rapid cyclization, resulting in higher yields and shorter reaction times.

Electron-donating substituents such as methoxy and methyl groups reduced electrophilic character and therefore required comparatively longer reaction times.

The cinnamaldehyde derivative exhibited the highest yield (95%) due to stabilization of reaction intermediates through extended conjugation.

Spectral characterization using UV–Visible, FTIR, and ^{1}H-NMR spectroscopy confirmed successful synthesis of benzimidazole derivatives.

The developed methodology offers several important advantages:

- Eco-friendly conditions
- Reduced reaction time
- High product yield
- Cleaner reaction profile
- Minimal solvent usage
- Lower energy consumption
- Operational simplicity
- Improved synthetic efficiency

The study therefore establishes sonochemical synthesis as an efficient and sustainable green chemistry approach for heterocyclic synthesis.

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X. CONCLUSION

The present investigation successfully developed a green sonochemical methodology for the synthesis of substituted benzimidazole derivatives using aromatic aldehydes in toluene medium under ultrasonic irradiation.

The method proved highly efficient, environmentally sustainable, rapid, and economically feasible. Ultrasonic irradiation significantly accelerated the condensation and cyclization reactions, reducing reaction time while improving product yield and purity.

The synthesized compounds were successfully characterized using UV–Visible spectroscopy, FTIR spectroscopy, and ^{1}H-NMR analysis, confirming formation of the benzimidazole nucleus.

The developed methodology aligns effectively with the principles of green chemistry and provides a sustainable alternative to conventional synthetic methods.

The synthesized benzimidazole derivatives may serve as valuable candidates for future medicinal chemistry investigations and biological screening studies.

XI. FUTURE SCOPE

Future research may focus on:

1. Biological evaluation of synthesized derivatives.
2. Anticancer and antimicrobial screening studies.
3. Nanocatalyst-assisted sonochemical synthesis.
4. Solvent-free sonochemical methodologies.
5. Industrial-scale sonochemical synthesis.
6. Molecular docking and computational studies.
7. Green solvent optimization for sustainable synthesis.

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