IJARSCT
International Journal of Advanced Research in Science, Communication and Technology
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
ISSN: 2581-9429
Volume 6, Issue 5, June 2026
Impact Factor: 8.2

Green Sonochemical Synthesis of Benzimidazole Derivatives Using Toluene

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Abstract: *Benzimidazole derivatives represent an important class of nitrogen-containing heterocyclic compounds possessing a wide range of biological and pharmaceutical activities such as antimicrobial, antiviral, antifungal, anticancer, anti-inflammatory, and antiparasitic properties. Due to their remarkable medicinal significance, the synthesis of benzimidazole derivatives has become an important area of research in organic and medicinal chemistry. Conventional synthetic methods often require harsh reaction conditions, longer reaction times, toxic solvents, and expensive catalysts, thereby creating environmental and economic concerns.*

The present investigation focuses on the green sonochemical synthesis of benzimidazole derivatives using substituted aromatic aldehydes and o-phenylenediamine in toluene medium under ultrasonic irradiation. Sonochemical synthesis is considered an environmentally benign and energy-efficient approach that utilizes ultrasonic waves to accelerate chemical reactions through acoustic cavitation. The developed methodology offers several advantages including reduced reaction time, improved product yield, cleaner reaction profile, operational simplicity, and eco-friendly reaction conditions.

Different substituted aromatic aldehydes containing electron-donating and electron-withdrawing groups were successfully employed in the synthesis. 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, FTIR spectroscopy, and ^{1}H-NMR analysis.

The results demonstrated that electron-withdrawing substituents favored higher yields and faster reaction rates compared to electron-donating substituents. Spectral analysis confirmed the successful formation of the benzimidazole nucleus and the presence of characteristic functional groups. The study establishes sonochemical synthesis as an efficient, rapid, environmentally sustainable, and economically viable method for the preparation of biologically important benzimidazole derivatives.

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

I. INTRODUCTION

Heterocyclic compounds constitute one of the most important and rapidly developing branches of organic chemistry. These compounds contain at least one heteroatom such as nitrogen, oxygen, or sulfur within a cyclic ring structure. Among the various heterocyclic compounds, nitrogen-containing heterocycles occupy a central position due to their widespread occurrence in natural products, pharmaceuticals, agrochemicals, and biologically active molecules.

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Nitrogen-containing heterocyclic compounds are essential components of numerous biological systems. Purine and pyrimidine bases present in DNA and RNA, amino acids such as histidine and proline, vitamins, alkaloids, and enzymes all contain heterocyclic structures. Due to their structural diversity and broad biological activities, heterocyclic compounds have attracted immense attention in medicinal and synthetic chemistry.

Among nitrogen-containing heterocycles, benzimidazole derivatives are particularly important because of their remarkable pharmacological properties. Benzimidazole is a bicyclic aromatic heterocyclic compound formed by the fusion of benzene and imidazole rings. The benzimidazole nucleus exhibits amphoteric behavior due to the presence of nitrogen atoms within the imidazole ring.

Benzimidazole derivatives possess a broad spectrum of biological activities including:

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

Several benzimidazole-containing drugs such as albendazole, mebendazole, omeprazole, pantoprazole, and thiabendazole are widely used in medicinal therapy.

Conventional synthetic methods for benzimidazole derivatives often involve:

- Harsh acidic conditions
- Toxic solvents
- Longer reaction times
- High energy consumption
- Low selectivity

Therefore, the development of environmentally friendly and efficient synthetic methodologies has become highly important.

Green chemistry emphasizes sustainable chemical processes that reduce hazardous substances and environmental pollution. Sonochemical synthesis has emerged as an important green chemistry technique utilizing ultrasonic irradiation to accelerate organic reactions.

Ultrasonic irradiation generates acoustic cavitation, involving formation and collapse of microscopic bubbles in liquids. This process produces localized high temperature and pressure conditions that enhance reaction rates and improve yields.

Advantages of sonochemical synthesis include:

- Reduced reaction time

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- Mild reaction conditions
- Higher product yield
- Lower energy consumption
- Cleaner reactions
- Eco-friendly methodology
- Improved selectivity

The present work therefore focuses on the green sonochemical synthesis of benzimidazole derivatives using toluene as solvent under ultrasonic irradiation conditions.

II. OBJECTIVES OF THE STUDY

1. To synthesize benzimidazole derivatives using sonochemical methodology.
2. To develop an environmentally friendly synthetic route.
3. To study the effect of different aromatic substituents on reaction efficiency.
4. To characterize synthesized compounds using spectroscopic techniques.
5. To evaluate the advantages of ultrasonic irradiation in heterocyclic synthesis.

III. LITERATURE REVIEW

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

Recent research has focused on green and sustainable methods including:

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

Sonochemical methods have gained significant attention because they dramatically reduce reaction time and improve reaction efficiency.

Researchers have demonstrated that ultrasonic irradiation enhances organic transformations through acoustic cavitation effects. Green sonochemical synthesis has therefore become an important tool in sustainable organic chemistry.

IV. EXPERIMENTAL SECTION

4.1 Materials Used

- o-Phenylenediamine
- Aromatic aldehydes
- Toluene
- Glacial acetic acid
- Ethanol

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- Distilled water

4.2 Apparatus

- Ultrasonic bath
- Round-bottom flask
- Magnetic stirrer
- TLC plates
- UV chamber
- Buchner funnel
- Vacuum filtration setup

V. GENERAL SYNTHETIC PROCEDURE

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

The reaction mixture was subjected to ultrasonic irradiation in an ultrasonic bath operating at 35–40 kHz frequency at 50–60°C.

The progress of reaction was monitored by Thin Layer Chromatography (TLC). After completion of the reaction, the mixture was cooled and poured into ice-cold water. The precipitated product was filtered, washed, dried, and recrystallized using ethanol.

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. Cyclization under ultrasonic irradiation.
4. Oxidative aromatization leading to benzimidazole formation.

Ultrasonic irradiation accelerates the reaction by enhancing molecular collisions and energy transfer 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

DOI: 10.48175/IJARSCT-35658

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VIII. SPECTRAL ANALYSIS

8.1 UV–Visible Spectroscopy

The UV–Visible spectrum showed characteristic absorption bands due to π→π* and n→π* transitions.

λmax (nm)	Transition	Assignment
230–250	π→π*	Aromatic ring transitions
270–290	π→π*	Extended conjugation
320–360	n→π*	Nitro group transitions

The absorption peaks confirmed successful formation of conjugated benzimidazole structures.

8.2 FTIR Spectroscopy

Important FTIR peaks observed:

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

The FTIR spectrum confirmed formation of benzimidazole ring and nitro substituent incorporation.

8.3 ^{1}H-NMR Spectroscopy

Characteristic 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 protons

The downfield NH signal confirmed successful cyclization to benzimidazole nucleus.

IX. RESULTS AND DISCUSSION

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

The sonochemical method significantly reduced reaction time compared to conventional heating methods. Most reactions were completed within 5–16 minutes.

Electron-withdrawing substituents such as:

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

produced higher yields and faster reaction rates due to increased electrophilicity of aldehydic carbon.

Electron-donating substituents such as methoxy and methyl groups required slightly longer reaction times.

Cinnamaldehyde derivative showed the highest yield (95%) due to enhanced conjugation and stabilization of reaction intermediates.

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Spectroscopic characterization confirmed successful formation of benzimidazole derivatives.
Advantages of developed methodology:

- Eco-friendly conditions
- Reduced reaction time
- High product yield
- Cleaner reaction profile
- Easy work-up
- Lower energy consumption
- Minimal solvent usage

The study confirms that sonochemical synthesis is an efficient and sustainable approach for heterocyclic synthesis.

X. CONCLUSION

The present study successfully developed a green sonochemical method for the synthesis of substituted benzimidazole derivatives using toluene under ultrasonic irradiation.
The method proved highly efficient, environmentally friendly, rapid, and economical. Ultrasonic irradiation significantly enhanced reaction efficiency and reduced reaction time while affording excellent yields.
The synthesized compounds were successfully characterized using UV–Visible spectroscopy, FTIR spectroscopy, and ^{1}H-NMR analysis.
The developed methodology aligns well with green chemistry principles and offers a sustainable alternative to conventional synthetic methods.
The synthesized benzimidazole derivatives may serve as valuable candidates for future biological and pharmaceutical investigations.

XI. FUTURE SCOPE

Future studies may focus on:

1. Biological evaluation of synthesized derivatives
2. Anticancer and antimicrobial screening
3. Use of greener solvents
4. Nanocatalyst-assisted sonochemical synthesis
5. Industrial-scale sonochemical synthesis
6. Computational and molecular docking studies

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