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SHORT COMMUNICATION

Ultrasound-assisted one-pot cyclization for the synthesis of 2-substituted benzimidazole derivatives: A rapid access via NaOH/I_2 as an efficient oxidant system

Nasrin Alishahi*

Department of Chemistry, Catalysis Division, Faculty of Organic Chemistry, University of Isfahan, Isfahan, Iran

Abstract

Benzimidazole scaffolds represent an important class of heterocyclic compounds due to their wide range of pharmacological and industrial applications. An efficient, metal-free, and operationally simple protocol for the synthesis of 2-substituted benzimidazoles via an ultrasound-assisted one-pot cyclization of aromatic aldehydes and *o*-phenylenediamine is reported. The method employs NaOH/I_2 as an inexpensive and effective oxidant system under mild conditions at room temperature. This approach addresses the limitations of conventional methods, which often require transition-metal catalysts, hazardous oxidants, prolonged heating, and produce lower yields. In the present study, reactions were completed within 4–7 min, affording the desired products in yields up to 99% across a broad substrate scope. Control experiments demonstrated the beneficial role of ultrasonic irradiation, and a plausible mechanism was proposed. The combination of mild reaction conditions, short reaction times, and high efficiency highlights the potential of this method as a green and scalable alternative for benzimidazole synthesis.

Keywords: 2-Substituted benzimidazole; Ultrasound-assisted synthesis; NaOH/I_2 oxidant; One-pot reaction; Metal-free protocol; Green chemistry

***Corresponding author:**
Nasrin Alishahi
(alishahi.nasrin@yahoo.com)

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1. Introduction

Benzimidazole and its derivatives represent significant heterocyclic structures and key components in the synthesis of biologically active molecules.[1,2] As the benzimidazole moiety is an auxiliary isostere of natural nucleotides, it has been extensively utilized as a promising scaffold in drug design.[1,3,4] Indeed, it was considered an amide isostere in chemical compounds; consequently, the incorporation of benzimidazole moieties instead of the amide group produces novel antibiotics against several Gram-negative strains.[5]

The benzimidazole core serves as a crucial pharmacophore, widely employed as a key framework for developing selective drugs across various therapeutic fields, such as HIV reverse transcriptase inhibitors, alkylating agents, topoisomerase inhibitors, antimicrobials, antivirals, antihistamines, antihelmintics, antioxidants, antihypertensives, anticoagulants, and antiulcers.[4,6-8] It has been illustrated *in silico*

SARS-CoV-2 screening activity.[9-12] Certain benzimidazoles have been documented to exhibit antidiabetic properties, primarily through the activation of AMP-activated protein kinase and peroxisome proliferator-activated receptor, the suppression of α-amylase and α-glucosidase enzymes, and other mechanisms related to diabetes management.[13,14] Several methods have been documented for synthesizing benzimidazoles, including the condensation of *o*-phenylenediamines with aldehydes, carboxylic acids, or their derivatives, using traditional catalysts, such as polyphosphoric acid, *p*-toluenesulfonic acid, boric acid, ceric ammonium nitrate, and sodium hydrogen sulfite. In addition, the condensation of *o*-phenylenediamines with *o*-esters in the presence of various Lewis acid catalysts is also a well-established approach.[15-17]

Classical synthetic routes to 2-substituted benzimidazoles typically rely on the condensation of *o*-phenylenediamines with aldehydes under oxidative conditions. These protocols often employ transition-metal catalysts (e.g., Cu, Fe, and Pd) or stoichiometric hazardous oxidants, and require prolonged heating at elevated temperatures, which contributes to high energy consumption and raises environmental concerns. Recent improvements have included the use of microwave irradiation to reduce reaction times and increase yields,[15,18] solvent-free methodologies to minimize waste,[19] greener oxidants such as hydrogen peroxide or molecular oxygen,[20] and solid-supported catalysts for better recovery and recycling.[19] Ultrasound-assisted methods have also emerged as effective alternatives, offering improved mass transfer and significantly faster reaction rates.[21] However, many of these ultrasound-based protocols still require specialized catalysts or longer reaction times compared to the present system. The ultrasound $NaOH/I_2$ method presented herein addresses these limitations by providing a metal-free, room-temperature, one-pot approach with remarkably short reaction times (as little as 4 min) and excellent yields over a broad substrate scope, using an inexpensive and operationally simple oxidant system. Thus, there is a need to devise straightforward, practical, and highly efficient methods for synthesizing benzimidazole cores within a short timeframe.

In comparison with conventional approaches to benzimidazole synthesis, the present ultrasound-assisted $NaOH/I_2$ protocol offers several distinctive advantages. First, the process is entirely metal-free, eliminating the need for expensive or environmentally problematic catalysts. Second, the reaction proceeds efficiently at room temperature under mild conditions, substantially reducing energy consumption. Third, the combination of ultrasound irradiation with the $NaOH/I_2$ oxidant system results in significantly shortened reaction times (4–7 min) without sacrificing yield. Fourth, the operational simplicity of this one-pot method makes it well-suited for scale-up, enabling practical synthesis of a diverse range of 2-substituted benzimidazole derivatives with minimal purification requirements. Collectively, these attributes demonstrate both the theoretical innovation and the engineering significance of the method, positioning it as a greener and more efficient alternative to many existing protocols.[22,23]

Accordingly, in continuation of our recent studies on the construction of vital heterocyclic compounds, this study presents a simple approach for synthesizing diverse substituted benzimidazole derivatives using aryl aldehydes and *o*-phenylenediamine, facilitated by the $NaOH/I_2$ system under ultrasonic irradiation at ambient temperature (Figure 1).[15,16,24]

2. Materials and methods

2.1. General information

All reagents, obtained in high purity from Fluka (Honeywell, United States [US]) and Merck (Germany)

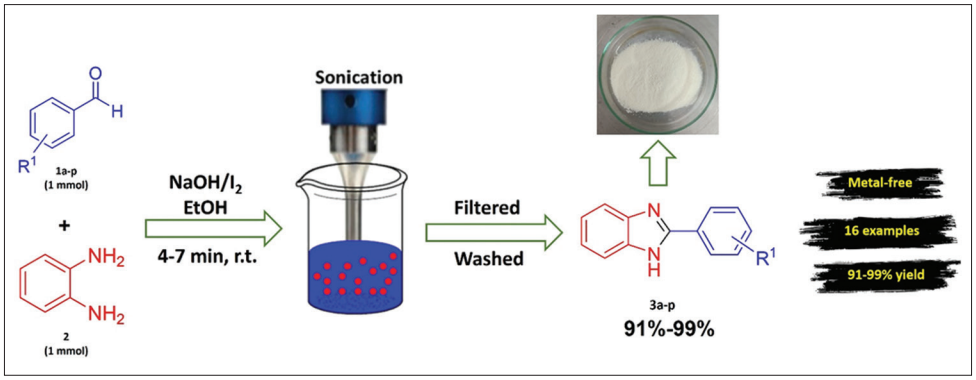

Figure 1. Synthesis of 2-substituted benzimidazole derivatives using ultrasound irradiation and $NaOH/I_2$ as an oxidant system
Notes: 1a-p represent the different aromatic aldehydes used as substrates; 2 is *o*-phenylenediamine; 3a-p correspond to the synthesized 2-substituted benzimidazole derivatives.
Abbreviation: r.t.: Room temperature.

chemical suppliers, were used as received without additional purification. Solvents were purified using standard protocols: EtOAc was washed with 5% aqueous Na_2CO_3, followed by saturated aqueous NaCl and dried over $MgSO_4$; MeOH was purified via fractional distillation; and EtOH was dried over CaO and distilled under reduced pressure.

2.2. Apparatus

Melting points were measured using the IA9200 instrument (Thermo Fisher Scientific, US). Fourier-transform infrared (FTIR) spectra were obtained from the FTIR-8300 spectrometer (Shimadzu, Japan) with KBr pellets. ^{1}H-nuclear magnetic resonance (NMR) and ^{13}C-NMR spectra (at 400 MHz and 100 MHz, respectively) were acquired from the DRX-400 spectrometer (Bruker, US) in $CDCl_3$ or DMSO-d_6, using tetramethylsilane as the internal reference. The ultrasonic homogenizer HD 3200 (Bandelin, Germany) equipped with a KE 76 probe (6 mm diameter; Bandelin, Germany) was used to generate ultrasonic irradiation and ensure homogenization of the reaction mixture. The piezoelectric crystal in this probe typically operates at 700 kHz, but through the use of appropriate clamps, the output frequency was adjusted and lowered to 20 kHz. The probe imparted a frequency of 20 kHz to the reaction mixture. Modifying the tip's power influences the cavitation rate, while the frequency remains fixed at 20 kHz across varying power levels. A thermal approach was used for calibrating the ultrasonic power.

2.3. Typical procedure for the synthesis of 2-substituted benzimidazole derivatives using I_2/NaOH oxidant system

A mixture of aryl aldehyde (1 mmol) and *o*-phenylenediamine (1 mmol), combined with NaOH/I_2 (4 mmol/2 mmol) as an effective oxidant system, was dissolved in EtOH and subjected to ultrasonic irradiation at 45 W and sonicated at room temperature (25 ± 2°C) until completion as monitored by thin-layer chromatography (eluent: petroleum ether/EtOAc, 4:1). Upon completion, the mixture was diluted with a H_2O: EtOAc solution (1:1; total 10 ml) and stirred at room temperature for 20 min. The organic phase was separated, dried over sodium sulfate, and evaporated under vacuum conditions to remove the solvent and residual materials. The crude product was purified by recrystallization in an EtOAc: MeOH mixture (2:1), yielding a pure compound. The resulting benzimidazoles were characterized through melting point determination and spectroscopic analysis.

3. Results and discussion

3.1. Optimization of the reaction conditions

This research explored the influence of ultrasonic irradiation on the model reaction involving 4-nitrobenzaldehyde (1 mmol) and *o*-phenylenediamine (1 mmol) in the presence of a NaOH/I_2 system at ambient temperature (Table 1). The influence of the molar ratios of the oxidant system was first examined in the reaction (Table 1, entries 1–7), and a 4:2 molar ratio of NaOH: I_2 was found to be optimal to provide a maximum yield of the desired product (Table 1, entry 6).

In addition, the impacts of various solvents, such as CH_3CN, EtOH, $CHCl_3$, CH_2Cl_2, n-hexane, and H_2O, on the model reaction were examined (Table 1, entries 8–12). EtOH was identified as the optimal solvent for this reaction. Building on this approach, the influence of ultrasonic irradiation and the effects of varying ultrasonic power levels on the reaction were explored. The reaction was conducted using the NaOH/I_2 system as an oxidant in EtOH, under both silent conditions and ultrasonic irradiation (Table 1, entries 13–16).

The findings demonstrated that ultrasonic irradiation significantly enhanced the reaction system, leading

Table 1. Optimization of reaction conditions[a]

Entry	Power (W)	NaOH: I_2 ratio	Solvent	Time (min)	Yield (%)[b]
1	45	2:1	EtOH	30	58
2	45	1:1	EtOH	30	25
3	45	1:2	EtOH	60	<10
4	45	2.2	EtOH	30	49
5	45	3:2	EtOH	30	81
6[c]	45	4:2	EtOH	4	99
7	45	5:2	EtOH	30	99
8	45	4:2	CH_3CN	30	72
9	45	4:2	$CHCl_3$	30	58
10	45	4:2	CH_2Cl_2	30	67
11	45	4:2	n-Hexane	30	42
12	45	4:2	H_2O	30	85
13[d]	None	4:2	EtOH	60	-
14	35	4:2	EtOH	30	54
15	40	4:2	EtOH	30	87
16	50	4:2	EtOH	5	98

Notes: [a]Reaction conditions: 4-nitrobenzaldehyde and o-phenylenediamine in the presence of NaOH/I_2 as an oxidant system; [b]Isolated yield. [c]Optimal reaction conditions; [d]The reaction without ultrasonic irradiation.

to shorter reaction times and higher product yields. In contrast, the reaction conducted with the NaOH/I_2 system without sonication produced lower yields and extended reaction times (Table 1, entry 13). Furthermore, it was found that employing the NaOH/I_2 system with ultrasonic irradiation at a power of 45 W yielded optimal results, producing a 98% isolated yield in just 4 min (Table 1, entry 6).

3.2. Proposed role of ultrasonic irradiation

The beneficial effect of ultrasonic irradiation is attributed primarily to acoustic cavitation – the rapid formation, growth, and collapse of microscopic bubbles within the liquid medium. This phenomenon generates transient localized hotspots with extremely high temperatures and pressures, as well as intense micro-streaming and turbulence. These effects enhance mass transfer between phases, improve molecular mixing, and can accelerate both condensation and oxidative aromatization steps. In the present NaOH/I_2 system, ultrasonic irradiation potentially promotes more efficient generation and activation of reactive iodine species in proximity to the reacting intermediates, thereby increasing the overall reaction rate. Control experiments revealed that performing the reaction without ultrasound extended the reaction time from 4 min to 60 min and reduced the yield from 99% to 40%. These observations are consistent with literature reports on the role of ultrasonic cavitation in organic synthesis.[25,26]

A range of substrates was subsequently investigated using the optimal reaction conditions. As shown in Figure 2, aromatic aldehydes with electron-donating (methyl, methoxy, and alkyl) or electron-withdrawing (nitro, chloro, and bromo) groups on the aromatic ring reacted efficiently with *o*-phenylenediamine in the presence of NaOH/I_2 to afford the desired 2-substituted benzimidazoles in excellent yields (Figure 2, 3a–3p).

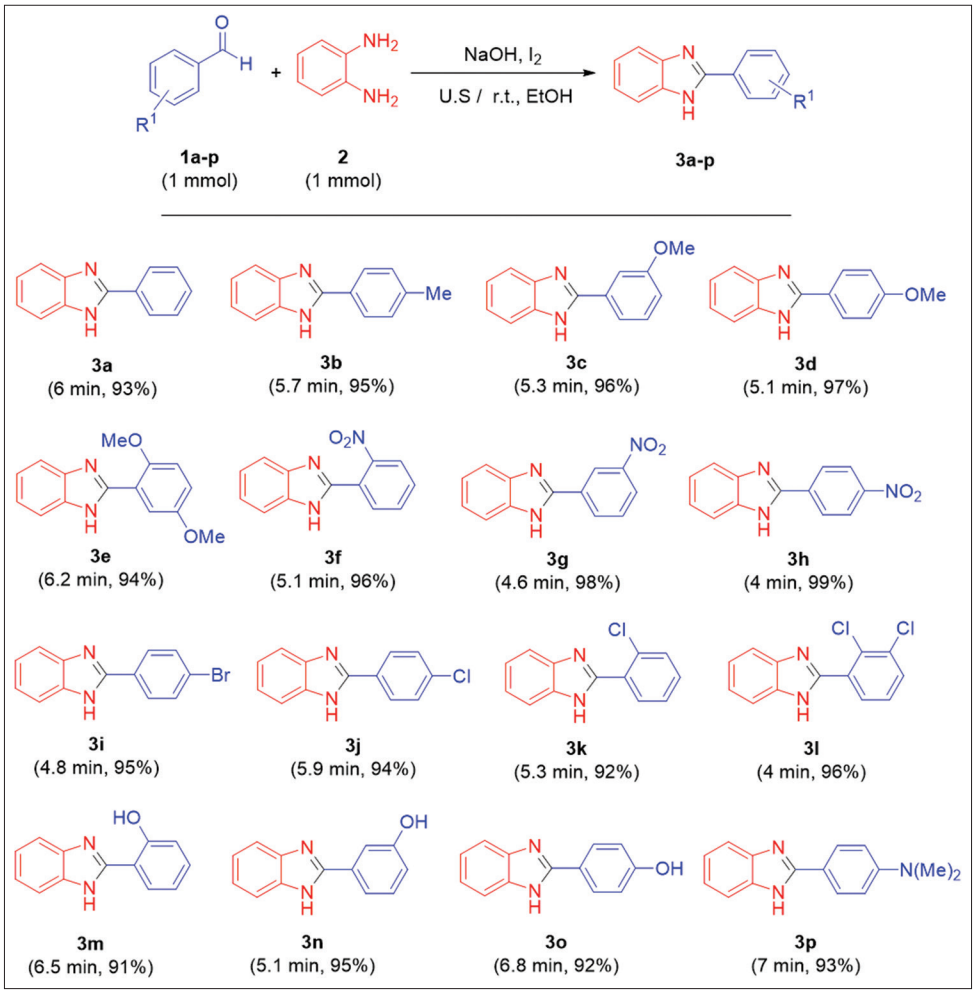

Figure 2. Reaction scopes of aromatic aldehydes and *o*-phenylenediamine in the presence of NaOH/I_2. Refer to the Supplementary Files 1 and 2 for their characterization.

The comparison between ultrasound irradiation and thermal conditions revealed that the reaction of various aromatic aldehydes with *o*-phenylenediamine and the NaOH/I_2 system was significantly enhanced by ultrasonic irradiation. As depicted in Figure 2, the use of NaOH/I_2 under ultrasonic conditions resulted in high product yields and reduced reaction times. In a previously reported work, the reaction of 4-nitrobenzaldehyde and *o*-phenylenediamine in a NaOH/I_2 system with thermal conditions for 33 min afforded 2-(4-nitrophenyl)-1H-benzo[*d*]imidazole in 97% yield.[16] In contrast, the present procedure needed only 4 min to produce 2-(4-nitrophenyl)-1*H*-benzo[*d*]imidazole in 99% yield (Figure 2 3h).

This method offers multiple benefits, including straightforward reaction conditions, high product purity, rapid reaction times, excellent yields, and simplified work-up procedures. These findings confirm that ultrasonic irradiation is an effective approach for the one-pot synthesis of 2-substituted benzimidazoles, which are valuable and significant heterocyclic compounds.

To highlight the efficiency of the present ultrasound–NaOH/I_2 system, a comparison with representative literature methods for the synthesis of 2-substituted benzimidazoles is provided in Table 2.[27-29] In addition to eliminating the need for transition-metal catalysts, the current protocol completes reactions within only 4 min at

Table 2. Comparison of the present ultrasound–NaOH/I_2 protocol with representative literature methods for 2-substituted benzimidazole synthesis

Entry	Method/system	Energy input	Solvent/ conditions	Temperature(°C)	Time (min)	Representative yield (%)	Metal catalyst	References
1	NaOH/I_2	Ultrasonic bath	EtOH	r.t.	4	99	No	This work
2	Amberlite IR-120	Ultrasonic bath	EtOH/H_2O	50	30	96	No	27
3	Natural dolomitic limestone	Ultrasonic probe	EtOH/H_2O (1:1)	45–50	30	85	Contains Ca/Mg (no transition metal)	28
4	Cu-complex-bipy/ phen-MCM-41	Ultrasonic bath	EtOH	60	90	95	Cu	29

Abbreviation: r.t.: Room temperature.

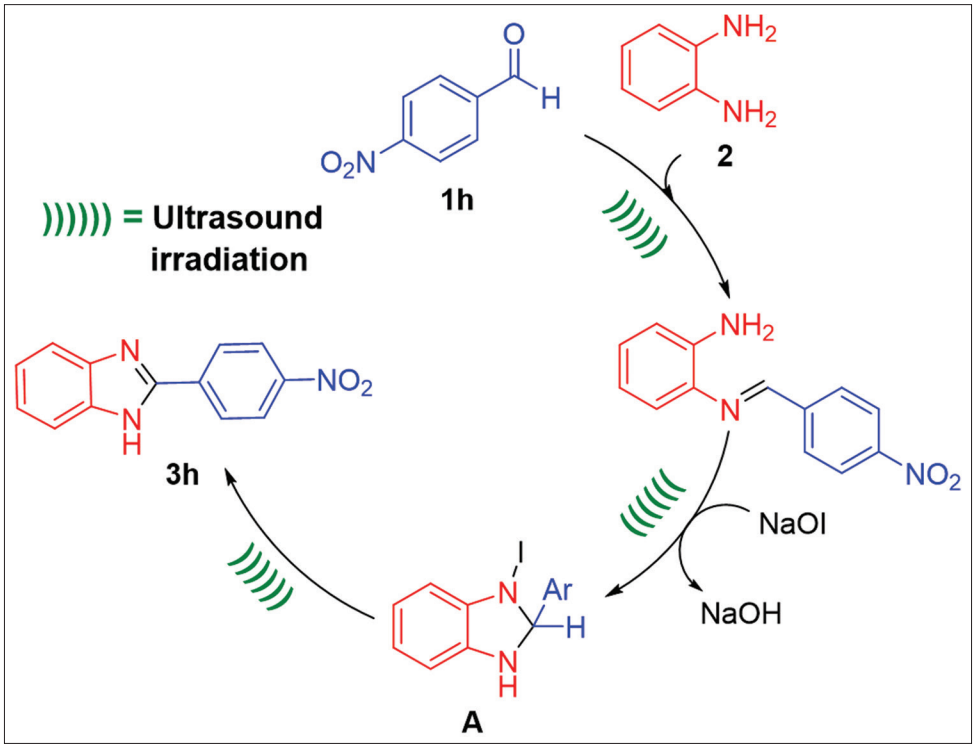

Figure 3. Proposed mechanism for the synthesis of 2-substituted benzimidazoles

room temperature, delivering yields up to 99%. By contrast, many conventional methods require prolonged reaction times (hours), elevated temperatures, or transition-metal catalysts, such as Cu-complex-bipy/phen-MCM-41. Ultrasound-assisted literature protocols using Amberlite IR-120 or natural dolomitic limestone also show advantages over purely thermal methods; however, they still require longer reaction times or special solid supports. These comparisons underscore both the synthetic efficiency and the green chemistry potential of the present approach.

3.3. The proposed reaction mechanism

The proposed mechanism for synthesizing 2-substituted benzimidazoles via cyclization of aromatic aldehydes and *o*-phenylenediamine under ultrasonic irradiation with the $NaOH/I_2$ oxidant system is illustrated in Figure 3. The mechanism potentially involves the generation of NaOI through the reaction of sodium hydroxide with iodine. The NaOI subsequently interacts with the initially formed hydrobenzimidazoles to produce intermediate A, followed by the elimination of hydrogen iodide to form the corresponding benzimidazoles.[16]

4. Conclusion

A simple and efficient ultrasound-assisted one-pot protocol for the synthesis of 2-substituted benzimidazoles has been developed using $NaOH/I_2$ as an inexpensive and effective oxidant system. The method proceeds under mild, metal-free conditions at room temperature. It enables the completion of reactions within 4–7 min, affording the desired product yields up to 99% across a broad substrate scope. Control experiments confirmed the crucial role of ultrasonic irradiation in accelerating the transformation. Compared with conventional protocols, which often require transition-metal catalysts, hazardous oxidants, elevated temperatures, or long reaction times, the present method offers a greener and more practical alternative. Together with the comparative analysis presented in Table 2, these results highlight the novelty and potential scalability of this strategy for the rapid preparation of benzimidazole derivatives.

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Conflict of interest

The author declares no conflict of interest.

Author contributions

This is a single-authored article.

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Availability of data

The data that support the findings of this study are available in the supplementary material of this article.

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