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ONE-POT SYNTHESIS OF 2-SUBSTITUTED BENZOXAZOLE/BENZIMIDAZOLE/BENZOTHIAZOLES USING (DIACETOXYIODO)BENZENE (DIB)AS AN EFFICIENT CATALYST

A. Venkateswarlu[1], M. Hari Krishna[1], P. Thriveni*[1].

[1]*Department of Chemistry, Vikrama Simhapuri University, Nellore-524320, A.P., India.*
***Corresponding Author E-mail**: pthriveni@vsu.ac.in*

ABSTRACT: Using DIB as a catalyst in CH_3CN, we reported the synthesis of 2-substituted benzoxazole/benzimidazole/benzothiazoles. This approach greatly increases its utility as a special and potent tool in chemical synthesis. This technique offers a novel approach to the synthesis of diverse benzoxazole, benzimidazole, and benzothiazole derivatives using various aldehydes and o-substituted amino aromatics. This technique gives good to exceptional yields of products and is relatively simple to build up. Following the reaction, the products were examined by mass spectroscopy, infrared spectroscopy, and NMR.

KEYWORDS: DIB, Benzoxazoles, Benzothiazoles, Benzimidazole, Trifluoroacetic acid, aromatic aldehydes, room temperature.

INTRODUCTION

Benzoxazole/Benzimidazole/Benzothiazoles are fused membered rings, which contain the heterocycles bearing Oxygen/Nitrogen/Sulphur atoms that constitute the core structure of oxazole/imidazole/thiazole and many pharmacologically and biologically active compounds. The numbering in oxazole/imidazole/thiazole starts from the Oxygen/Nitrogen/Sulphur atom. The basic structure of benzo oxazole/imidazole/thiazole consists of a benzene ring fused with 4, 5 positions of oxazole/imidazole/thiazole. The two rings together constitute the basic nucleus 1,3-benzoxazole/imidazole/thiazole. An important class of heterocyclic compounds, benzoxazole/benzimidazole/benzothiazoles[i] are employed in research as building blocks to synthesize larger, typically bioactive molecules. Despite being a heterocyclic with reactive sites that permit functionalization, its aromaticity gives it a relative sense of stability. A wide range of biological activities, including antihistaminic[ii], antifungal[iii], cyclooxygenase inhibiting[iv], antitumor[v], antiulcer[vi], anticonvulsant[vii], hypoglycaemic[viii], anti-inflammatory[ix,x], and cytotoxic activity[xi,xii], are exhibited by benzoxazole and its derivatives. They also have potential activity with lower toxicities in the human chemotherapeutic approach[xiii,xiv]. In addition to being employed in various industrial applications like insecticides, dyes, and fluorescent brightening agents, oxazole and its derivatives are utilized as building blocks for biochemicals and pharmaceuticals. The biological activities of the benzimidazole moiety include anticancer[xv-xx], anti-hypertensive[xxi], anthelminthic[xxii], anti-protozoal[xxiii],

antimicrobial[xxiv-xxvii], antioxidant[xxviii], anti-inflammatory[xxix-xxx], analgesic[xxxi], and anti-hepatitis-B-virus [xxxii] properties. Benzimidazole moiety possesses various biological activities such as anticancer [xxxii], anti-hypertensive [xxxiv], anthelminthic [xxxiv-xxxvii], anti-protozoal [xxxviii-xxxix], antimicrobialx[XL-XLI], antioxidant[XLII], anti-inflammatory [XLIII-XLV], analgesic [XLVI] and anti-hepatitis-B-virus [XLVII]. Benzothiazole moiety shows antimicrobial [XLVIII-XLIX] anticancer [L-LI], anthelmintic[LII], and anti-diabetic [LIII] activities. They have also found applications in industry as anti-oxidants, and vulcanization accelerators. Synthesizing bioactive molecules such as benzoxazole, benzimidazole, and benzothiazoles efficiently and in a way that is both economically and environmentally beneficial is a major issue in modern chemistry. These biological actions make the synthesis of benzoxazole/benzimidazole/benzothiazoles a considerable discussion nowadays.

Benzoxazole, benzimidazole, and benzothiazole derivatives synthesis are of great interest due to their intriguing chemical characteristics and wide range of pharmacological applications. Two major techniques were used to manufacture benzoxazoles. One is the use of strong acids or microwave conditions to couple carboxylic acid derivatives with *o*-substituted amino aromatics. The other is the oxidative cyclization of aldehydes and phenolic Schiff bases. It has been reported that various catalysts were used in the synthesis of benzoxazole-like $Pd(OAc)_2$ [LIV], $ZrOCl_2.8H_2O$ [LV], silica sulfuric acid [LVI], Silica supported sodium hydrogen sulfate [LVII], Indion 190 resin [LVIII], ($[Hbim]BF_4$) [LIX], ethane sulphonic acid [LX], $Cu(OTf)_2$ [LXI], copper(II) oxide nanoparticles [LXII], PCC-supported silica gel [LXIII], $In(OTf)_3$ [LXIV], $SnCl_2$ [LXV], DDQ [LXVI], $BF_3.OEt_2$ [LXVII], $Mn(OAc)_3$ [LXVIII], $PhI(OAc)_2$ [LXIX], $Th^+.ClO_4^-$ [LXX], $BaMnO_4$ [LXXI], NiO_2 [LXXII], and $Pb(OAc)_4$ [LXXIII].

In the case of the synthesis of Benzimidazole & benzothiazole, it involves condensation of *o*-amino thiophenol with substituted aldehydes [LXXIV-LXXX], acyl chlorides, carboxylic acids [LXXXI], or esters and nitriles the traditional method. Pd/Cu/Mn/chloranil catalyzed cyclization of *o*-halo thioformanilides is another frequently employed method [LXXXII]. A review of the literature on benzothiazoles reveals that this bicyclic ring system is present in a variety of amine or terrestrial natural compounds, which have significant biological properties. Some other preparation methods involved the reaction of 2-amino thiophenol with carboxylic acids, aldehydes, acyl chlorides, substituted nitriles, or esters [LXXXIII]. Many catalysts namely (PmIm)Br [LXXXIV], TMSCl [LXXXV], I_2 [LXXXVI], $ZrOCl_2 \cdot 8H_2O$ [LXXXVII], PCC [LXXXVIII], H_2O_2, CAN [LXXXIX], electro-oxidation [XC], Baker's yeast [XCI], PTSA [XCII], Dowex 50W reusable catalyst in water at 70°C [XCIII], TCCA in 2-MeTHF at ambient temperature [XCIV], PEG,200/400 under microwave heating [XCV], NIBTS at ambient temperature under solvent-free conditions [XCVI], H_2SO_4/SiO_2 as a reusable catalyst at room temperature, H_2O[XCVII], animal bone meal [XCVIII], $Na_2S_2O_5$ in refluxing DMF, $Cu(OAc)_2$/MCM41 supported catalyst under ultrasound irradiation [XCIX], 2,4,6-trichloro-1,3,5-triazine under mild conditions, sulfamic acid [C]. Nevertheless, a number of these methods have one or more drawbacks, including the need for a significant amount of volatile organic solvents, severe reaction conditions, extended reaction times, and low yields with numerous byproduct production. Hence, it is still preferable to build a clean, highly productive, and environmentally friendly strategy. However, there are still some limitations with the existing protocols such as drastic reaction conditions, tedious work-up procedures, poor selectivity, low yields with the formation of many side products and use of large quantities of volatile organic solvents, excess amounts of reagent, and use of toxic solvents. Therefore, we wished to explore the usage of (diacetoxyiodo)benzene (DIB) for the selective synthesis of 2-substituted Benzoxazole/benzimidazole/benzothiazoles which shows short reaction times and enhanced selectivity.

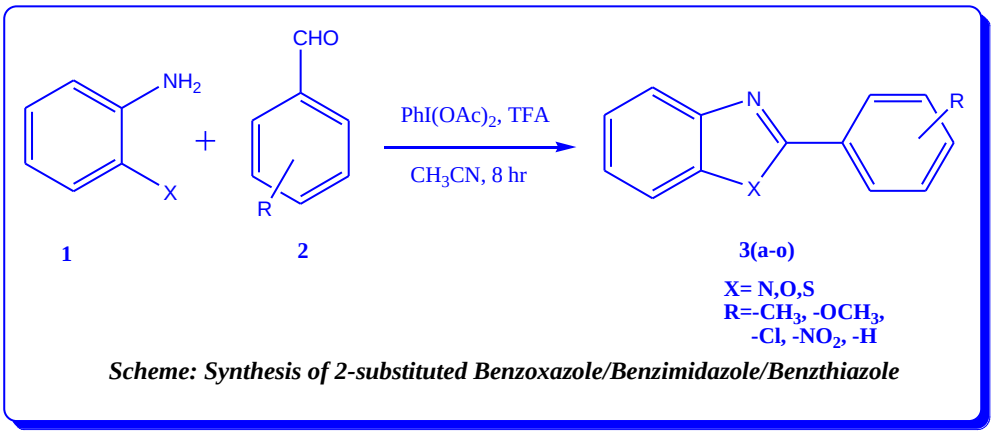
MATERIALS AND METHODS

Uncorrected melting points were found in open-end capillaries. Iodine vapors were used to find spots on silica gel G plates while TLC was used to evaluate the purity of the compounds. The temperature of the water bath was adjusted by adding or removing water while the flask was positioned in the cleaner's maximal energy area. The IR spectra were recorded on the Perkin-Elmer spectrum RX IFT-IR System using KBr pellets. The NMR spectra were measured by using a Bruker Avance spectrometer at 600 MHz, 400.1 MHz, and 100.6 MHz. Chemical shifts are given in ppm (δ) and are referenced to the residual proton resonances of the solvents. Using tetramethylsilane (TMS) in the solvent of $CDCl_3$-*d* or DMSO-d_6 as the internal standard Proton and carbon magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were recorded (^{1}H NMR: TMS at 0.00 ppm, $CDCl_3$ at 7.26 ppm, DMSO at 2.50 ppm; ^{13}C NMR: $CDCl_3$ at 77.16 ppm, DMSO at 40.00 ppm).

***General procedure for the synthesis of* benzimidazole, benzoxazole & *benzoxazoles* (3a-o):**

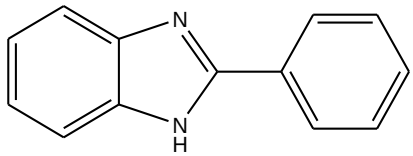
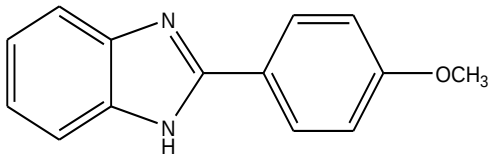
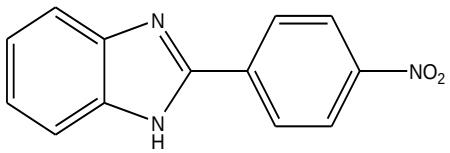
Under purging N_2, solid (diacetoxyiodo)benzene ("DIB", 2.39 g, 7.42 mmol, 0.135 eq) is added, then CH_3CN (15 mL). During the next several steps, the flask is kept in an inert atmosphere after being purged for ten minutes. After carefully adding 0.64 mL, 8.36 mmol, and 0.15 equiv of trifluoroacetic acid at room temperature, the liquid becomes homogenous. The DIB solution was completely mixed with a solution of newly distilled aromatic aldehyde (10 mmol) and *o*-substituted amino aromatics (5 mmol) in a flask at room temperature. The mixture was then stirred for 8 hrs. The reaction progress was monitored by thin layer chromatography (TLC), ethyl acetate: hexane (3:2). Upon completion of the reaction (TLC), the reaction mixture was cooled at rt, ethyl acetate (100 ml) was added, and stirred well followed by filtration through celite under suction. The organic layer was washed with water (2×25 ml) and brine (25 ml). After drying over anhydrous Na_2SO_4, the solvent was evaporated under reduced pressure and the residue upon column chromatography affords the pure product. All the products were identified by spectral (IR, ^{1}H NMR, ^{13}C NMR) and analytical data.

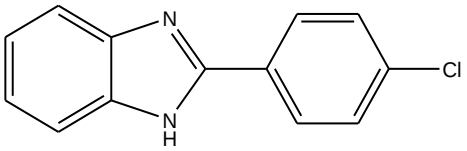
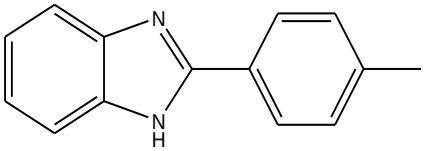
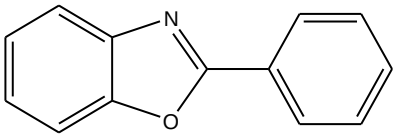
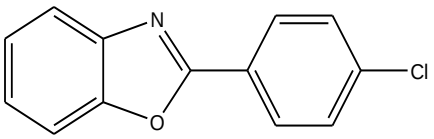
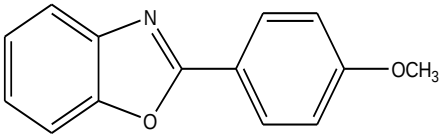
Scheme I: The synthetic route was depicted in Scheme I.

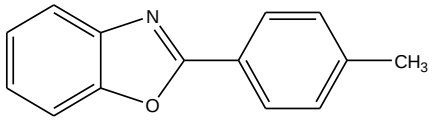
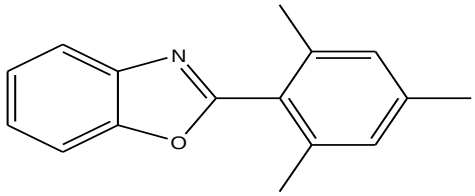
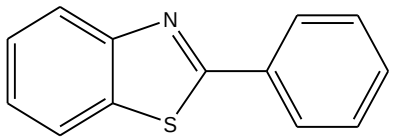
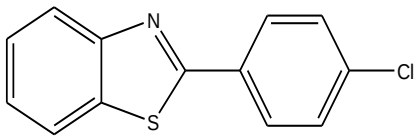
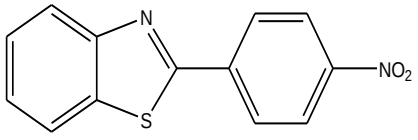


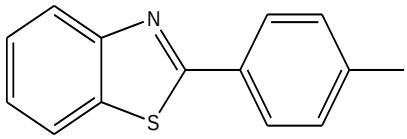
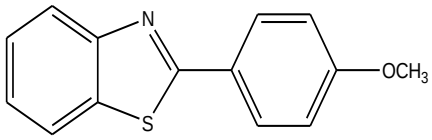
Scheme: Synthesis of 2-substituted Benzoxazole/Benzimidazole/Benzthiazole

Product characterization data (IR, 'H NMR, ^{13}C NMR, HRMS):

Compound	Spectral data	Yield (%)
2-Phenyl-1*H*-benzimidazole(3a): 	Pale yellow crystals IR (KBr): 1627 (C=N), 3435 (NH) cm^{1}. 'H NMR (600 MHz, DMSO-d$_6$): δ 12.96 (s, 1H), 8.21-8.21 (t, *J*= 9.0 Hz, 2H), 7.63-7.49 (m, 5H), 7.23-7.20 (m, 2H). ^{13}C NMR (150 MHz, DMSO- d$_6$): δ 151.70, 130.64, 130.31, 129.42, 126.91, 122.58. HRMS (ESI) Calc, for $C_{13}H_{11}N_2$ $[M+H]^+$: 195.0917, found: 195.0916.	89%
2-(4-Methoxyphenyl)-1*H*-benzimidazole(3b): 	White crystals IR (KBr): 1242 (C-O), 1612 (C=N), 2964 (CH_3), 3439 (NH) cm^{1}. 'H NMR (600 MHz, DMSO- d$_6$): δ 12.75 (s, 1H), 8.13 (d, *J*= 3.0 Hz, 2H), 7.55 (s, 2H), 7.17 (dd, $J_{1=}5.1$ Hz, J_2 = 3.0 Hz, 2H), 7.12 (d, *J* = 2.4 Hz, 2H), 3.84 (s, 3H). ^{13}C NMR (150 MHz, DMSO- d$_6$): δ 161.06, 151.81, 128.47, 123.18, 122.25, 114.83, 55.79. HRMS (ESI) Calc, for $C_{14}H_{13}N_2O$ $[M+H]^+$: 225.1022, found: 225.1021	93%
2-(4-Nitrophenyl)- *1H* -benzimidazole(3c): 	Yellow crystals IR (KBr): 1338, 1515 (NO_2), 1607 (C=N), 3436 (NH) cm^{1}. 'H NMR (600 MHz, DMSO- d6): δ 13.31 (s, 1H), 8.44-8.45 (m, 4H), 7.73-7.66 (m, 2H), 7.27 (s, 2H). ^{13}C NMR (150 MHz, DMSO- d$_6$): δ 149.45, 148.26, 136.51, 127.85, 124.76. HRMS (ESI) Calc, for C13H10N3O2 $[M+H]^+$: 240.0768, found: 240.0768.	88%
2-(4-Chlorophenyl)-*1H*-benzimidazole(3d):	White crystals, IR (KBr): 1623 (C=N), 3442 (NH) cm^{1}. 'H NMR (600 MHz, DMSO- d$_6$): δ 13.00 (s, 1H), 8.20 (dd, *Ji* = 6.6 Hz, J_2 = 1.8 Hz, 2H), 7.65-7.63 (m, 4H), 7.23 (d, *J* = 3.0 Hz, 2H). ^{13}C NMR (150 MHz, DMSO- d$_6$): δ	93%

	150.63, 134.96, 129.53, 128.61, 123.20, 122.29, 119.44, 111.88. HRMS (ESI) Calc, for C13H10CIN2 $[M+H]^+$: 229.0527, found: 229.0523.	
2-(4-Methylphenyl)- *1H*-benzimidazole (3e): 	Yellow crystals IR (KBr): 1623 (C=N), 2965 (CH_3), 3449 (NH) cm^1. 'H NMR (600 MHz, DMSO- d_6): δ 12.82 (s, 1H), 8.08 (d, *J*= 8.4 Hz, 2H), 7.57 (s, 2H), 7.37 (d, *J*= 7.8 Hz, 2H), 7.20 (dd, *Ji*=6.0 Hz, J_2 = 3.0 Hz, 2H), 2.38 (s, 3H). ^{13}C NMR (150 MHz, DMSO- d_6): δ 151.83, 140.04, 129.98, 127.90, 126.86, 122.43, 21.43. HRMS (ESI) Calc, for C14H13N2 $[M+H]^+$: 209.1073, found: 209.1072.	91%
2-phenyl benzoxazole (3f): 	white solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.29-8.25 (m, 2H), 7.82-7.78 (m, 1H), 7.62-7.58 (m, 1H), 7.56-7.53 (m, 3H), 7.39-7.35 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.0, 150.7, 142.0, 131.5, 128.9, 127.6, 127.1, 125.1, 124.6, 120.0, 110.6 ppm.	90%
2-(4-chlorophenyl)benzoxazole (3g): 	white solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.20 (d, *J* = 8.4 Hz, 2H), 7.79-7.76 (m, 1H), 7.60-7.58 (m, 1H), 7.51 (d, *J* = 8.4 Hz, 2H), 7.40-7.36 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0, 150.8, 142.0, 137.3, 129.3, 128.9, 125.7, 125.5, 124.8, 120.2, 110 ppm.	94%
2-(4-methoxyphenyl)benzoxazole (3h): 	white solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 8.8 Hz, 2H), 7.76-7.73 (m, 1H), 7.57-7.55 (m, 1H), 7.36-7.30 (m, 2H), 7.03 (d, *J* = 8.4 Hz, 2H), 3.90 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.1, 162.3, 150.6, 142.2, 129.3, 124.6, 124.4, 119.7, 119.6, 114.3, 110.3, 55.4 ppm.	96%
2-(p-tolyl)benzoxazole (3i):	white solid. ^1H NMR (400 MHz, $CDCl_3$): δ 8.17	87%

	(d, *J* = 8.4 Hz, 2H), 7.78-7.75 (m, 1H), 7.59-7.57 (m, 1H), 7.36-7.33 (m, 4H), 2.44 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.3, 150.6, 142.1, 129.6, 127.6, 124.9, 124.5, 124.2, 119.8, 110.5, 21.7 ppm.	
2-(2,4,6-trimethylphenyl)benzoxazole (3j): 	white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84-7.83 (m, 1H), 7.61-7.58 (m, 1H), 7.42-7.39 (m, 2H), 6.98 (m, 2H), 2.37 (m, 3H), 2.31 (s, 6H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2, 150.6, 141.5, 140.3, 138.2, 128.6, 124.9, 124.1, 120.1, 110.6, 21.3, 20.3 ppm.	90%
2-Phenyl-1,3-benzothiazole (3k): 	IR (KBr): 3060, 3021, 1630, 1592, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 – 8.06 (m, 3H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.51 – 7.47 (m, 4H), 7.38 (t, *J* = 7.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.1, 154.2, 135.1, 133.6, 130.9, 129.2, 127.6, 126.3, 125.1, 123.2, 121.6.	93%
2-(4-Chlorophenyl)benzothiazole (3l): 	IR (KBr): 3080, 3032, 1628, 1592, 695 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.2 Hz, 1H), 8.01 (d, *J* = 8.5 Hz, 2H), 7.88 (d, *J* = 8.0 Hz, 1H), 7.52–7.36 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 154.0, 137.1, 135.0, 132.1, 129.2, 128.7, 126.5, 125.4, 123.3, 121.6.	89%
2-(4-Nitrophenyl)benzothiazole (3m): 	IR (KBr): 1605, 1517, 1341, 1311, 1250, 1107, 968, 851, 765, 750, 729, 685 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.45 (brs, 4H), 8.19 (d, *J* = 7.2 Hz, 1H), 8.17 (d, *J* = 7.2 Hz, 1H), 7.64 (t, *J* = 7.1 Hz, 1H), 7.57 (t, *J* = 7.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.8, 154.5, 146.8, 139.2, 134.9, 128.2, 127.0, 126.3, 124.1, 123.8, 121.6.	87%
2-(4-Methylphenyl)benzothiazole (3n):	IR (KBr): 3026, 2811, 2343, 1606, 1581, 1520,760, 685 cm^{-1}.	91%

	^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00-8.06 (m, 3H), 7.96 (d, *J* = 8.0 Hz, 1H), 7.51 (t, *J* = 8.4 Hz, 1H), 7.41 (t, *J* = 8.4 Hz, 1H), 7.36 (d, *J* = 8.1 Hz, 2H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.0, 154.2, 141.5, 135.0, 131.1, 129.7, 127.3, 126.3, 125.0, 122.9, 121.6, 21.2.	
2-(4-Methoxyphenyl)benzothiazole (3o): 	IR (KBr): 3545, 1755, 1631, 1450, 1100, 1201, 950, 650 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 1H), 7.67-7.52 (m, 2H), 7.49-7.30 (m, 3H), 7.18-7.00 (m, 1H), 3.88 (3H, s). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.8, 158.4, 153.1, 135.1, 133.7, 129.9, 126.5, 125.8, 123.5, 121.3, 120.8, 117.9, 113.2, 55.2.	94%

RESULT AND DISCUSSIONS

Our initial study of the model reaction between *o*-substituted amino aromatics and benzaldehyde revealed that, in the presence of (diacetoxyiodo)benzene (DIB), the reaction could be completed under very basic reaction conditions, yielding the desired benzimidazole, benzoxazole, and benzothiazole derivatives in good yield (**Scheme 1**). It was discovered that (diacetoxyiodo)benzene (DIB) at room temperature provided the best reaction conditions for the reaction (**Table 1**). Diacetoxyiodobenzene (DIB) has demonstrated efficacy as a catalyst in the synthesis of multiple targeted derivatives such as benzoxazole, benzimidazole, and benzothiazole.

Table 1: Optimization of reaction conditions

Entry	Catalyst	Solvent	Temp(^{0}C)	Yield (%)
1	DIB	Water	rt	43
2	DIB	THF	50	38
3	DIB	Toluene	120	52
4	DIB	Ethanol	65	61
5	DIB	Methanol	65	62
6	DIB	DMSO	130	69
7	DIB	DMF	110	59
8	DIB	DCM	rt	76
9	DIB	Acetone	rt	54
10	DIB	CH_3CN	rt	83

Different substituted aldehydes were used to explore the reaction's potential, and the results show that the substituent's location on the aromatic ring of the aldehydes had no bearing on yield.

CONCLUSION

Using (diacetoxyiodo)benzene (DIB) as an effective eco-friendly and efficient catalyst, we have shown how to synthesize 2-substituted desirable benzimidazole, benzoxazole, and benzothiazole derivatives straightforwardly. This method's benefits include short response times, high yields, a clean process, an easy-to-follow technique, quick and simple work-up, and environmentally friendly settings. The current approach is extremely straightforward and offers improved selectivity, distinct benefits, and a different path to the targeted products. Finally, we developed that synthesis from *o*-substituted amino aromatics and aldehydes in the presence of (diacetoxyiodo)benzene (DIB) in CH_3CN in good yields at room temperature, resulting in the required 2-substituted benzimidazole, benzoxazole, and benzothiazole derivatives.

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