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Sonocatalyzed facile synthesis of 2-aryl benzoxazoles using MnO_2 nanoparticles as oxidant agent under mild conditions

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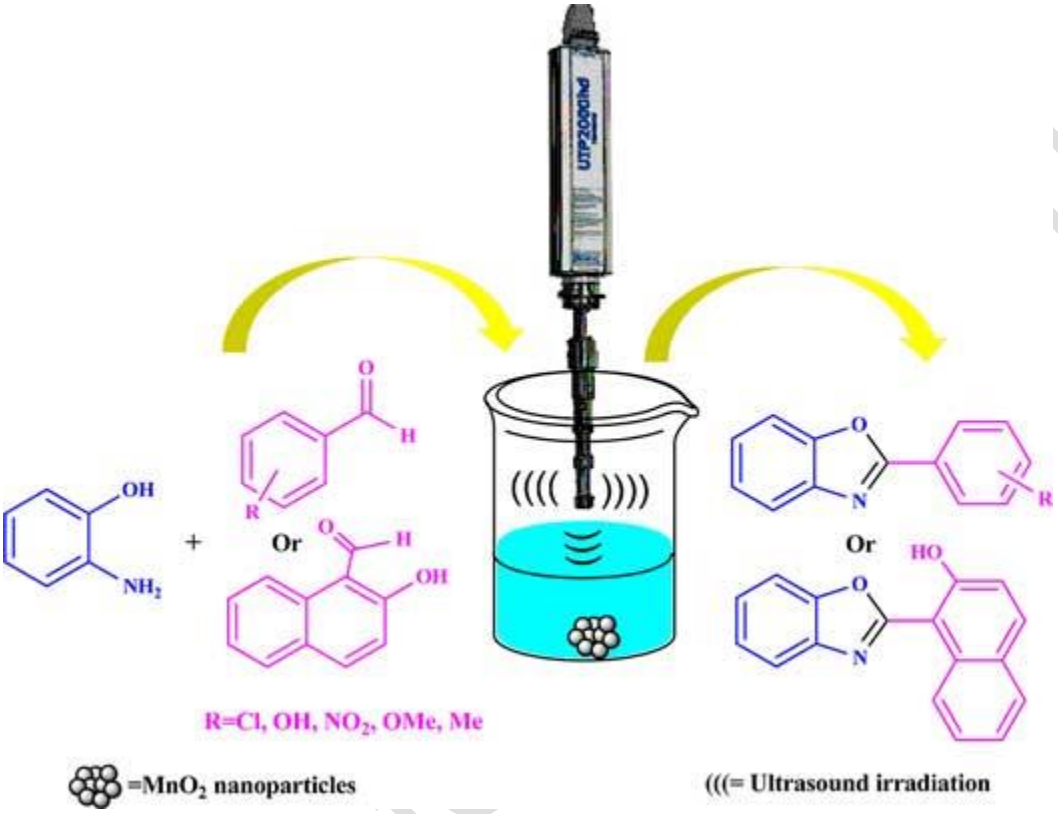
ABSTRACT

Nano MnO_2 was found to be an efficient oxidant agent for the synthesis of 2-substituted benzoxazoles through one-pot reaction of o-aminophenol and different aromatic aldehydes in acetonitrile under ultrasonic irradiation. This method was carried out under mild conditions with high yields, inexpensive and readily available oxidant agent, facile and easy experimental procedure, simple purification of final products and short reaction times. The prepared nano MnO_2 has been characterized by FT-IR, XRD, and SEM techniques. The pure products were identified

and characterized by physical and spectroscopic data such as; melting point, IR, ^{1}H NMR and ^{13}C NMR.

GRAPHICAL ABSTRACT



Ultrasound irradiation was implemented for the convenient synthesis of 2-substitued benzoxazoles through the reaction of 2-aminophenol with aromatic aldehydes in the presence of nano-MnO_2 as catalyst in acetonitrile solvent.

KEYWORDS: 2-aryl benzoxazole, nano mno2, *o*-aminophenol, oxidant, ultrasound

Introduction

Benzoxazole and its derivatives have been receiving much attention in the recent years owing to their pharmaceutical and biological applications.[1] These heterocyclic systems have been

reported to show antifungal,[2] antitumor,[3] antibacterial,[4] anti-inflammatory,[5] anticonvulsant, antitubercular,[6] DNA topoisomerase inhibitor,[7] anti-microbial, anti-HIV-1[8] and antiulcer[9] activities. Furthermore, some of them have been used as dye releaser in instant color chromatography[10] herbicides, fluorescent whitening agents,[8] dye lasers,[11] photoluminescent devices[12] and as ligands in asymmetric synthesis[13] (**Figure 1**).

A number of methods have been reported for the preparation of benzoxazoles including cyclization of 2-halo-*N*-acylanilines,[14] ring closure of *o*-aryl oximes,[15] annulation of 1,2-dihalobenzenes with primary amides,[8] ortho-C-H functionalization of phenols with nitroalkanes,[16] reaction of 2-aminophenol with carbon–carbon triple bonds,[17] 2-aminophenols and *β*-diketones,[18] coupling of 2-aminophenols with different carbonyl derivatives such as aldehydes,[19] carboxylic acids,[20] acyl halides,[21] nitriles,[22] isocyanides,[23] and carbon monoxide.[24]

Ultrasound is an efficient, green, simple, virtually innocuous, inexpensive and valuable tool in synthetic chemistry.[25] The advantages of this powerful technique in driving chemical reactions are (a) low rate of energy consumption, (b) minimization of wastes, (c) utilization of less hazardous reactants and solvents, (d) generation of product with increased selectivity and improved yield, (e) the use of low temperatures and pressures, (f) high reaction rate.[26–28] Hence, ultrasonic irradiation, a means of activation, has been widely used in organic synthesis.[29–31]

In continuation of our previous works on the synthesis of heterocyclic compounds by ultrasonic irradiation [32–34] and considering importance of benzoxazole derivatives, herein, we decided to explore the ability of nano MnO_2 as a mild oxidant for the sonochemical preparation of 2-substituted benzoxazoles. Therefore, nano MnO_2 was used as an oxidant agent for the synthesis

of these compounds by the condensation reaction of *o*-aminophenol with aromatic aldehydes under ultrasound irradiation.

Results and Discussion

Preparation of nano MnO_2 started by grinding reaction between $(NH_4)_2C_2O_4.H_2O$ and $MnSO_4.H_2O$ with a mole ratio of 1:1.2, followed by calcination of manganese oxalate (MnC_2O_4) precursors at 400 ^oC and acid-treatment process using the previous reported method.[35] After preparation of MnO_2 NPs, the oxidant was characterized by FT-IR, SEM and XRD techniques. In the FT-IR spectra of the MnO_2 NPs, the absorption bands in the region of 527 cm^{-1} is attributed to the stretching vibrations of the metal–oxygen bond and confirms the formation of the MnO_2 (Fig. 1S, see Supporting Information (SI)). The position and relative intensities of all peaks confirm well with standard XRD pattern of MnO_2 (JCPDSNo. 14-0644) indicative of the nanocrystalline structure of the prepared MnO_2 (Fig. 2S, see SI). The average crystalline size of MnO_2 particles was 17 nm (determined from the Scherer equation). The scanning electron microscope (SEM) of MnO_2 particles show nanoparticles with dimensions of about 35.5-46.3 nm (Fig. 3S, see SI).

In this research, we have developed a synthetic method in which nano MnO_2 efficiently catalyzes preparing of 2-substituted benzoxazoles through treatment of *o*-aminophenol and aromatic aldehydes under ultrasound conditions (Scheme 1).

In order to optimize the reaction conditions, the reaction of *o*-aminophenol (1.1 mmol, 0.12 g) with 2-chlorobenzaldehyde (1 mmol) was selected as a simple model and the efficiency of oxidant amounts, different solvents and the power of applied ultrasound have been studied.

Initially, the model reaction was carried out without nano MnO_2 but no product was obtained (**Table 1**, entry 1). Hence, nano MnO_2 as a reactive and appropriate oxidant agent was

used in this reaction. In order to optimize the amount of nano MnO_2, the model reaction was carried out in the presence of different quantities of MnO_2 nanoparticles in CH_3CN solution. The results are summarized in **Table 1**. It was shown that 1 mmol of MnO_2 nanoparticles lead to the best results compared to other runs (**Table 1**, entry 4). Therefore, 1 mmol of nano MnO_2 was appropriate and sufficient for this reaction.

In continuation of this research, to investigate the effect of various powers of ultrasonic irradiation the model reaction was performed under silent condition and different powers of ultrasound irradiation (**Table 2**). The results confirmed positive effect of sonication on our reaction system. It was decreased the time of reaction and enhanced the yield of the product (**Table 2**, entry 3), whereas, the reactions without ultrasonic irradiation was carried out in low yields and long reaction times (**Table 3**, entry 1 and 2). The reaction in the presence of nano MnO_2 and ultrasonic irradiation with power of 80 W afforded the best yield of product, with 86**%** isolated yield after 15 min **(Table 2**, entry 5).

As it is described in "hot spot" theory, collapse of cavitating bubbles leads to production of intensive local temperatures and pressures. This process provides the kinetic energy that drives the chemical reaction. High temperature, pressure and reactant contact surface area related to cavitation phenomenon and formation of hot spots are driving force for the fast and high yield formation of product under ultrasound irradiation. High temperature and pressure, formation of free radicals, high reactant contact surface area, rapid mixing and renewing of the liquid at the solid-liquid interface are responsible for increasing the yields of products and enhancing the rate of sonocatalyzed chemical reactions.[27], [28]

Solvents have important effect on the organic reactions. Hence, the model reaction, with 1 mmol of nano MnO_2, was investigated in different solvent systems under reflux and sonic

conditions. The results are summarized in **Table 3**. It can be seen from the **Table 3** that the best solvent for this reaction is acetonitrile. Furthermore, ultrasound mediated reaction in acetonitrile solution is faster and more efficient than the reaction performed under reflux condition.

Using the optimized conditions, we extended the study with *o*-aminophenol and several aldehydes for the synthesis of 2-substituted benzoxazoles in the presence of nano MnO_2 NPs. The pertinent results are presented in **Table 4**. The cyclocondensation-oxidation reaction between *o*-aminophenol and aromatic aldehydes proceeded well and afforded the desired products. The yields of the products vary in the range of 67–90% yields. The reaction of *o*-aminophenol with propanal as an aliphatic aldehyde was any product yield that can be due to less stability of 2-alkyl benzoxazole than the 2-aryl benzoxazole (**Table 4**, entry 14). This method offered various advantages including good yields, mild reaction conditions, high purity, high reaction rates and easy work up procedures. The structures of products were completely characterized by spectroscopic data such as; IR, ^{1}H NMR, ^{13}C NMR and melting point and they were consistent with those of authentic samples.[36–43]

The suggested mechanism for synthesis of 2-substituted benzoxazoles using aromatic aldehydes and *o*-aminophenol by nano MnO_2 under ultrasonic condition is presented in Scheme 2. The reaction proceeds via a cyclocondensation-oxidation reaction. First, nucleophilic attack of the nitrogen of *o*-aminophenol on electrophilic carbon atom of aldehyde and elimination of H_2O afford species 1. Then, nucleophilic attack of ortho-hydroxyl group on the carbon atom of imine 1 gives benzoxazoline 2. Radical oxidation of benzoxazoline by nano MnO_2 and elimination of $Mn(OH)_2$ afford the corresponding product 3. Surely, all steps are promoted by sonication. The physical effects of ultrasound (mixing of the reagents, dispersion of solids and emulsification of liquids) in

combination with the chemical effects (cavitation) can increase the reactivity of chemical species and accelerate the rate of reaction.[27]

In conclusions, in this study, nano MnO_2 were prepared and were investigated in the synthesis of 2-substitued benzoxazoles via reaction of a variety of aryl aldehydes with *o*-aminophenol in acetonitrile solution under ultrasound irradiation at room temperature. The reaction was efficiently preceded under ambient conditions, giving the corresponding products in good to high isolated yields. This method has several advantages including available and inexpensive oxidant, short reaction time, and simple workup procedure. The products were confirmed by spectroscopic and physical data such as; IR, ^{1}H NMR, ^{13}C NMR and the melting point.

Experimental

All commercially available reagents were used without further purification and purchased from Merck and Aldrich Company in high purity. The used solvents were purified by standard procedure. FT-IR spectra were obtained with KBr pellets in the range 400–4000 cm−1 with a Perkin Elmer 550 spectrometer. 1H NMR and 13C NMR were recorded in $CDCl_3$ solvent on a Bruker DRX-400 spectrometer with tetramethylsilane as the internal reference. A BANDELIN ultrasonic HD 3200 with probe model KE 76, with the diameter of 6 mm, was used to produce ultrasonic irradiation and homogenize the reaction mixture. Piezoelectric crystal of this kind of probe normally worked in the range of 700 kHz, but by use of some proper clamps, the output frequency of piezoelectric crystal was controlled and reduced to 20 kHz in the reaction mixture. X-ray diffraction patterns of samples were taken by using CuKα radiation (λ =1.5406A) on a Rigaku D−Max CIII diffractometer. Scanning electron microscope (SEM) of nano MnO_2 was

performed on a XL30 Philips SEM instrument. Melting points obtained with a Yanagimoto micro melting point apparatus and are uncorrected. The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

Shortly, $MnSO_4.H_2O$ (1 mmol, 0.169 g) and $(NH_4)_2C_2O_4.H_2O$ (1.2 mmol, 0.17 g) were taken in a mortar. After grinding at room temperature for 40 min, the mixture was placed in a water bath of 80 ^oC. A dry mixture was obtained after several hours. The mixture was washed with distilled water, dried at 110 ^oC for 10 h and calcined at 400 ^oC for 10 h. Then, the calcined product in 2 M H_2SO_4 solution was stirred magnetically at 80 ^oC for 2 h. The final product was washed with distilled water, filtered, and dried at 105 ^oC.

A mixture of 2-aminophenol (1.1 mmol, 0.12 g), aromatic aldehyde (1 mmol), and MnO_2 nanoparticles (1 mmol, 0.086 g) in acetonitrile (4 mL) was sonicated in ultrasonic apparatus with the power 80 Watt for the time indicated in **Table 4**. Progress of the reaction was monitored by thin-layer chromatography (petroleum ether/ethyl acetate, 6/2). After completion, the reaction mixture was cooled to room temperature. The minerals were separated by simple filtration. Then filtrate solution was poured onto ice. The solid crude product was filtered, washed with ice-water and recrystallized from ethanol/H_2O (1:1) to give the pure product. All of the 2-substituted benzoxazole products were identified by physical and spectroscopic data.[36–43]

Acknowledgments

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Supporting Information

Supporting Information: Full experimental detail, the IR, ^{1}H and ^{13}C NMR spectra, XRD, FT-IR and SEM. This material can be found via the "Supplementary Content" section of this article's webpage.

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Table 1. Optimization of nano MnO_2 amounts for synthesis of 2-(2-chlorophenyl)-1,3-benzoxazole[a].

Entry	Oxidant amount (mmol)	Time (min)	Yield (%)[b]
1	–	30	–
2	0.5	15	30
3	0.8	15	65
4	1	15	86
5	1	20	86
6	1.2	15	86

[a]Reaction conditions: *o*-aminophenol (1.1 mmol), 2-chloro benzaldehyde (1 mmol), and MnO_2 nanoparticles in acetonitrile solution (4 mL) under ultrasonic irradiation (80 W) at room temperature.

[b]Isolated yields.

Table 2. Survey the effect of ultrasonic irradiation on the formation of 2-(2-chlorophenyl)-1,3-benzoxazole[a]

Entry	Power (W)	Time (min)	Yield (%)[b]
1	Silent (r.t.)	90	trace
2	Silent (80°C)	80	10
3	70	15	49
4	75	15	68
5	80	15	86
6	85	15	86
7	80	20	86

[a]Reaction conditions: *o*-aminophenol (1.1 mmol), 2-chlorobenzaldehyde (1 mmol), and MnO_2 nanoparticles (1 mmol) in acetonitrile as solvent (4 mL) at room temperature.

[b]Isolated yields.

Table 3. Effect of solvent on the rate of sonocatalyzed reaction using nano MnO_2[a]

Entry	Solvent	Yield (%)[b]
		US[c] (Silent[d])
1	H_2O	0 (0)
2	H_2O/CH_3CN (1:1)	0 (0)
3	CH_3CN	86 (17)
4	EtOH	67 (10)
5	MeOH	75 (15)

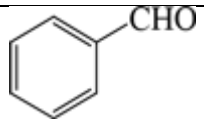
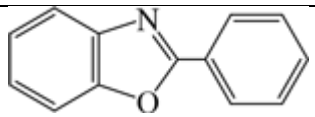
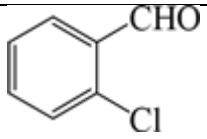
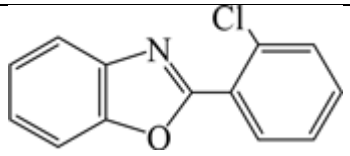
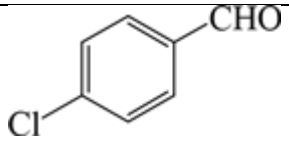
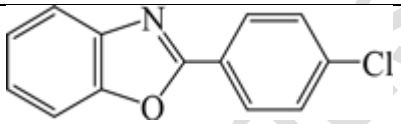
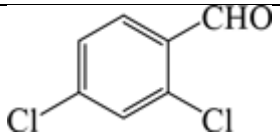
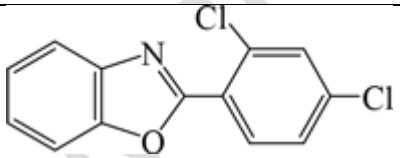
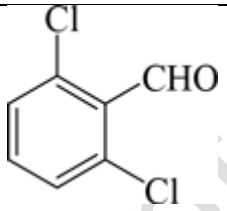
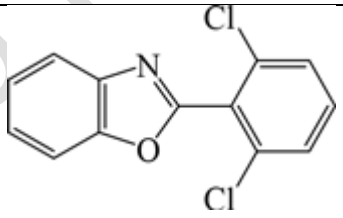
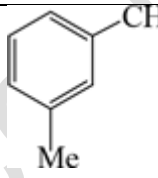
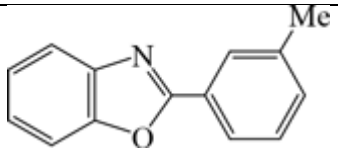
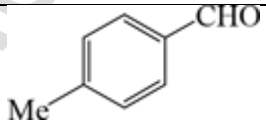
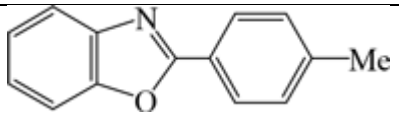
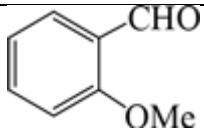
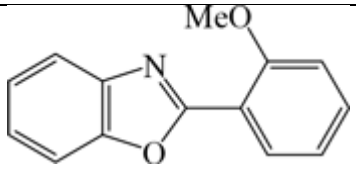
[a]Reaction conditions: *o*-aminophenol (1.1 mmol), 2-chlorobenzaldehyde (1 mmol), and MnO_2 nanoparticles (1 mm**ol) in selected solvent (4 ml).**

[b]Isolated yields of products.

[c]Room temperature, 15 min.

[d]Reflux, 2 h.

Table 4. Preparation of 2-substituted benzoxazoles under optimized conditions[a]

Entry	Substrate	Product	Time (min)	Yield (%)[b]
1			16	87
2			15	86
3			14	88
4			13	89
5			14	88
6			16	87
7			17	87
8			17	85

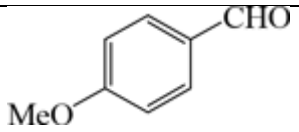
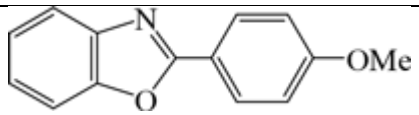
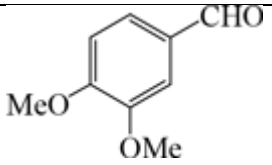
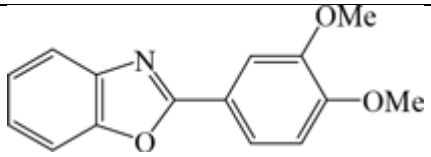
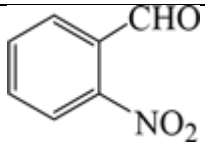
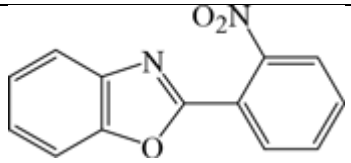
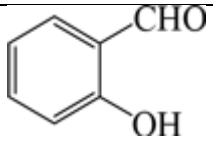
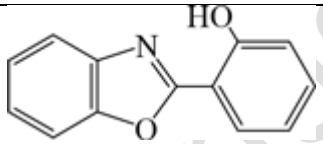
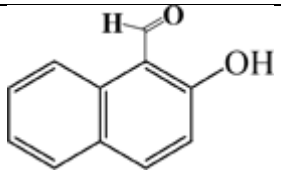
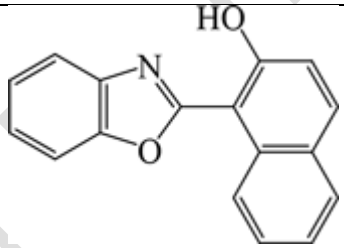
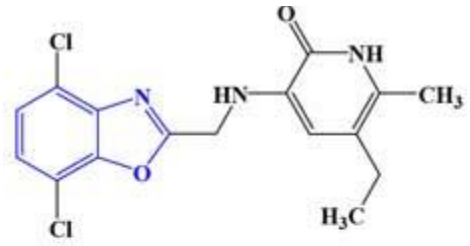
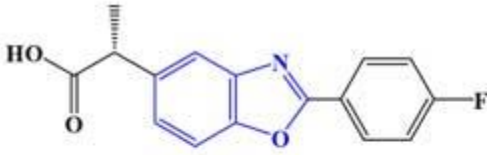
9			16	85
10			44	86
11			13	67
12			12	89
13			10	90
14	CH_3-CH_2-CHO	-	80	-

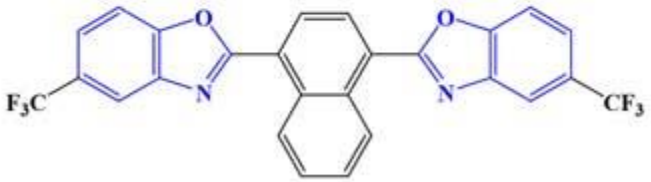
Figure 1. Representative compounds containing benzoxazole.



HIV reverse transcriptase inhibitor

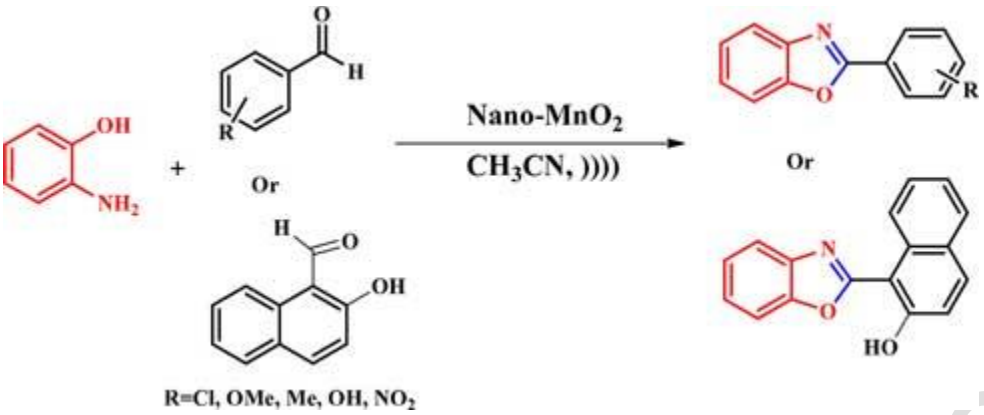


Anti-inflammatory



Fluorescent whitening agent dye

Scheme 1. Synthesis of 2-substituted benzoxazoles.



Scheme 2. Proposed reaction mechanism for the sonocatalyzed formation of 2-substitued benzoxazoles by nano MnO_2 as an oxidant agent.