AN EFFICIENT ONE POT SYNTHESIS OF 2-ARYL BENZOXAZOLE BY USING $\text{SiO}_2/\text{MgSnO}_3$ AS A HETEROGENEOUS RECYCLABLE CATALYST

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ABSTRACT

A highly effective, novel and fast processing method are reported the various derivatives of 2-aryl benzoxazole were prepared by using a catalytic amount of Silica containing MgSnO_3 ($\text{SiO}_2/\text{MgSnO}_3$). Low catalytic loading of silica-containing MgSnO_3 is found to be an economical and reusable catalyst for the reaction. The obtained percent yield of the product was satisfactory with a good experimental setup and the final products are pure and facile. The catalyst can be reused several times without loss of product yield and activity.

Keywords: 2-Aryl benzoxazoles, $\text{SiO}_2/\text{MgSnO}_3$, Recyclable Catalyst.

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INTRODUCTION

A Heterocyclic compound like benzoxazole, benzthiazole and benzimidazole derivatives have massive significance in chemical, adhesive, dyestuff, polymers, pharmaceutical and agricultural industries.¹⁻⁵ These derivatives have huge importance due to biological and pharmacological activities such as antihypertensive, antiepileptic, antimicrobial, antiviral and many more activities.⁶⁻¹⁸ Because of such significance, the synthesis of benzoxazole and its derivatives has been an attractive goal for organic as well as medicinal chemists. According to the literature survey, it is found that several synthetic methods are present for benzoxazole.

Many traditional routes are used for the synthesis of various substituted benzoxazole, many other substituents such as $-\text{Cl}$, $-\text{SH}$, and $-\text{SCH}_3$ or OPh with an amine are also synthesized.¹⁹⁻²² Many drawbacks occurred due to these traditional routes such as multiple steps required for preparation, harsh reagent utilization, the formation of undesirable by-products, several intermediate formations etc.

After traditional routes, the second widely followed methods are developed by oxidative cyclization of the phenolic Schiff bases by using wide varieties of oxidizing agents such as $\text{Mn}(\text{OAc})_3$, DDQ, NiO_2 , $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$, PCC-supported silica gel, Dess-Martin reagent, BaMnO_4 and $\text{Pb}(\text{OAc})_4$.²³⁻³⁰ On the other hand, some methods have been reported for the synthesis of benzoxazole using diverse catalysts and other reagents viz, silica-supported NaHSO_4 , CuOTf_3 , InTf_3 , SnCl_2 , $\text{BF}_3 \cdot \text{OEt}_2$, DMBQ, Indion 190 resin, silica-supported sulphuric acid, Co-complex.³¹⁻⁴⁰

Nowadays, the synthesis of 1, 3-benzoxazoles and their derivatives by using silica supported CuO , NPs, ZnO , CeO_2 NPs and various nanoparticles.⁴¹⁻⁴⁸ These methods were found to be an interesting approach due to the presence of large as well as reactive surface areas, good selectivity for reactions with mild conditions, easy separation of the product and catalyst and reusability of the catalyst.

Most of these reported methods suffer from many drawbacks for the synthesis of benzoxazole derivatives, some of the drawbacks such as harmful solvents used in reactions, prolong reaction time with fewer product yields, tedious work-up procedures, use of expensive reagents attracts more attention to overcome.

To overcome these drawbacks, there is a need to develop a new catalyst for the synthesis of benzoxazole derivatives which shows a simple work-up procedure, less toxicity, good recovery and recyclability.

Many organic conversions have been developed by using metal oxides, mixed metal and supported metal for the supported metal mixed oxide composites for the directed synthesis. With the presence of new and innovative strategies, here have been developed new synthesis methods for benzoxazole and its derivatives which are simpler than earlier.

Therefore, in the present study, we report the synthesis of benzoxazoles by using substituted aryl aldehydes and 2-amino phenol in the presence $\text{SiO}_2/\text{MgSnO}_3$ nanocrystalline catalyst (Scheme-1). This method was successfully utilized in several organic conversions to minimize unwanted waste which causes environmental pollution. With high thermal stability, a large surface area, easy recovery of the catalyst and good reaction capacity at normal temperature, such a catalyst and a method received more attention.

EXPERIMENTAL

All chemicals and solvents were purchased from Aldrich Chemical Co. Ltd and used without purification. The given product yield mentions after the purification of the isolated products.

Synthesis of the $\text{SiO}_2/\text{MgSnO}_3$ Composite Catalyst

In the present work, we have synthesized $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles by a hydrothermal method. In this method, 1:1 ratio of SiO_2 and MgSnO_3 was taken in Teflon autoclave along with 2N NaOH. The obtained product was kept in the oven for 24 hrs, after 24 hrs reaction mixtures were filtered, washed and dried for 2 hrs. Finally, polycrystalline powder of $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles obtained was used for further characterization and used as a catalyst for degradation of dye industrial wastewater. Here MgSnO_3 was synthesized using the reported method.

General Procedure for the Synthesis of 2-aryl benzoxazole

In a typical run, 2-aminophenol (1.2 mol), and aryl aldehydes (1.1 mol) were allowed to react in presence of methanol along with $\text{SiO}_2/\text{MgSnO}_3$ (0.4 mol) nanoparticles at 80°C for 20-30 min. The obtained mixture was checked by TLC method to get an idea about the product. After the completion of the reaction, the obtained product was directly filtered by a simple filtration method and washed by using methanol. The filtrate was collected and distilled out under reduced pressure to get the pure product. The recovered catalyst was washed with methanol and dried to reuse for further successive reactions. The pure products were confirmed by analytical techniques such as FT-IR, NMR and Mass spectrometry.

Scheme-1: Synthesis of 2-aryl benzoxazole

Spectral Data of Selected Compounds

2-Phenyl-1,3-benzoxazole (1)

FT-IR (KBr): 3449, 1664, 1590, 1121, 1048, 940 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.11 – 7.89 (m, 4H), 7.34-7.87 (m, 5H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 112.72, 121.17, 126.53, 127.06, 129.97, 132.91, 142.02, 152.31; Mass m/z : 196 ($M+1$).

2-(3-nitrophenyl)-1,3-benzoxazole (2)

FT-IR (KBr): 3322, 1650, 1550, 1450, 1350, 1275, 748 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.34-8.20 (m, 4H), 7.14 (dd, $J = 2.1$ & 8.4 Hz, 1H), 7.23 (dd, $J = 2.1$ & 8.4 Hz, 1H), 7.31-7.37 (m, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 111.12, 115.21, 124.23, 125.43, 126.74, 128.32, 131.73, 136.30, 139.21, 142.21, 150.21; Mass m/z : 241.32 ($M+1$).

2-(4-chlorophenyl)-1,3-benzoxazole (3)

FT-IR (KBr): 3330, 1650, 1632, 1486, 1390, 748 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.29-7.47 (m, 4H), 7.53 (d, $J = 8.1$ Hz, 2H), 7.73 (d, $J = 8.1$, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 115.53, 119.06, 121.90, 122.67, 126.74, 127.81, 129.86, 135.30, 139.90, 144.82, 152.73; Mass m/z : 231.21 ($M+2$).

2-(4-bromophenyl)-1,3-benzoxazole (4)

FT-IR (KBr): 3448, 1622, 1610, 1467, 1390, 840 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.21-8.48 (m, 4H), 7.51 (d, $J = 8.4$ Hz, 2H), 7.87 (d, $J = 8.4$, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 112.21,

118.23, 120.76, 124.54, 128.98, 129.81, 132.86, 138.30, 139.90, 142.82, 151.87; Mass m/z : 272.12 ($M+1$).

2-(4-fluorophenyl)-1,3-benzoxazole (5)

FT-IR (KBr): 3330, 1642, 1587, 1490, 13870, 683 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.08-7.34 (m, 4H), 7.48 (d, $J = 8.2$ Hz, 2H), 7.59 (d, $J = 8.2$, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 111.87, 117.76, 122.24, 122.67, 125.39, 128.81, 130.84, 133.94, 136.90, 142.82, 148.54; Mass m/z : 213.45 ($M+1$).

2-(4-methoxyphenyl)-1,3-benzoxazole (6)

FT-IR (KBr): 3229, 1673, 1610, 1532, 1128, 950 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 3.65 (s, 3H, CH_3), 7.16 (d, $J = 8.2$ Hz, 2H), 7.27 (d, $J = 8.4$ Hz, 2H), 7.34-8.03 (m, 4H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 32.81, 112.64, 115.83, 117.57, 120.35, 122.82, 124.11, 128.30, 132.64, 148.84, 156.46, 158.89; Mass m/z : 226.13.

2-(4-hydroxyphenyl)-1,3-benzoxazole (7)

FT-IR (KBr): 3390, 1687, 1634, 1450, 1392, 1122 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.17-7.64 (m, 4H), 7.34 (d, $J = 8.1$ Hz, 2H), 7.59 (d, $J = 8.1$, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 111.89, 116.48, 120.65, 123.85, 126.43, 128.72, 129.91, 133.30, 137.90, 142.91, 154.10; Mass m/z : 212.32.

2-(4-N,Ndimethyl-phenyl)-1,3-benzoxazole (8)

FT-IR (KBr): 3489, 1673, 1642, 1550, 1223, 1182 cm^{-1} ; ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ 7.13-7.43 (m, 4H), 7.64 (d, $J = 8.2$ Hz, 2H), 7.66 (d, $J = 8.2$, 2H); ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$): δ 116.53, 120.06, 122.98, 124.67, 126.81, 128.62, 131.65, 136.87, 138.94, 146.61, 154.74; Mass m/z : 239.12.

RESULTS AND DISCUSSION

Figure-1 shows the XRD pattern of $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles. The crystal structure of MgSnO_3 nanoparticles is cubic and all peaks well match with JCPDS data card no-28-625. The presence of strong and sharp diffraction peaks with 2θ angles are found at 25.34, 28.31, 34.87, 36.12, 38.32, 42.93, 52.49, 62.32, 74.71 and 75.29 corresponds to 110, 100, 222, 200, 111, 200, 111, 220, 110 and 311 crystal planes shows the presence of cubic Sn-O. The diffraction peaks of SiO_2 can be observed in the entire sample well match with JCPDS data which confirmed the formation of $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles.

Fig.-1: XRD Pattern of $\text{SiO}_2/\text{MgSnO}_3$ Composite Nanoparticles

Figure-2 shows the SEM analysis of $\text{SiO}_2/\text{MgSnO}_3$ nanoparticles. The SEM analysis shows the formation of spherical self-aligned, uniform and crystalline nanoparticles. The SEM analysis also indicates that after the addition of SiO_2 in MgSnO_3 , the surface morphology may affect however there is no large effect occurred on particle size. The aggregate sizes of the nanoparticles were relatively uniform in the range of 20-100 nm.

Fig.-2: SEM Analysis of $\text{SiO}_2/\text{MgSnO}_3$ Composite Nanoparticles

In Fig.-3, the TEM image along with SAED pattern was recorded for the sample $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles. TEM shows the presence of elliptical nanoparticles; however some cubic-shaped crystallites are also present. The dark spot in the TEM micrograph can be alluded to $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles as SAED pattern associated with such spots which are associated with tetragonal $\text{SiO}_2/\text{MgSnO}_3$ geometry and are agree with XRD data.

Fig.-3: TEM Analysis of $\text{SiO}_2/\text{MgSnO}_3$ Composite Nanoparticles

Figure-4 shows the N_2 adsorption-desorption isotherm along with BJH pore size distribution of synthesized $\text{SiO}_2/\text{MgSnO}_3$ composite nanoparticles. This isotherm shows that the sample has typical IV N_2 adsorption-desorption isotherms with H_1 hysteresis which indicates the presence of cylindrical mesopores in the sample. The BJH pore size distribution reveals that the sample has a narrow pore diameter. Based on the isotherms, the surface area (S_{BET}), pore volume (V_{p}) and pore diameter (d_{p}) were found $31.25 \text{ cm}^2/\text{gm}$, 0.247 cc/g and $45.12 \text{ \AA}$ respectively.

To determine the most promising reaction conditions for the synthesis of 2-aryl benzoxazole and its derivatives by evaluating the catalytic efficiency of $\text{SiO}_2/\text{MgSnO}_3$ catalyst; initially, a model study was carried out by using p-methoxy benzaldehyde as a model substrate for the synthesis of 2-aryl benzoxazole (Scheme-1).

To evaluate and enhance the effectiveness of $\text{SiO}_2/\text{MgSnO}_3$ with a different catalyst, we tried MgO , SnO_2 , MgSnO_3 and $\text{SiO}_2/\text{MgSnO}_3$ for the cyclization reaction of 2-aminophenol and 2-methoxy benzaldehyde. MgO and SnO_2 both gave poor yield as compared to $\text{SiO}_2/\text{MgSnO}_3$, whereas MgSnO_3 gave a good yield but required more time as compared to $\text{SiO}_2/\text{MgSnO}_3$ (Table-1).

Fig.-4: BET Surface Area of SiO₂/MgSnO₃ Composite Nanoparticles

Table-1: Effect of Different Catalyst on Reaction Time and Yield

Entry	Catalyst	Time (Min)	Yield
1	MgO	90	57
2	SnO ₂	80	64
3	MgSnO ₃	40	90
4	SiO ₂ /MgSnO ₃	20	94

Thus, it is found that SiO₂/MgSnO₃ was superior for the cyclization reaction of 2-aryl benzoxazole with a good yield in a short time. To optimize the amount of catalyst required for the cyclization, we tried various mol equivalents of the catalyst compared with the amount of 2-amino phenol (Table 2). The result was found that when the reaction was carried out by 0.4 mol equivalent of SiO₂/MgSnO₃ catalyst, the cyclization gave 94% yield in 20 minutes.

Table-2: Effect of Mole Equivalent of SiO₂/MgSnO₃

Entry	Mole equivalent of SiO ₂ /MgSnO ₃	Time (Min)	Yield
1.	0.2 mol	80	68
2.	0.4 mol	20	94
3.	0.6 mol	30	87
4.	0.8 mol	30	86

When cyclization reaction was carried out in presence of different solvents such as DMF, methanol, ethanol, acetonitrile, dichloromethane, the result found that methanol gave the better result as compared to another solvent (Table-3). Also, the reaction temperature was varied and found that at 80⁰ C the reaction was completed in a short time with high yield.

Table-3: Effect of Solvent for 2-aryl benzoxazoles using SiO₂/MgSnO₃

Entry	Solvent	Temp. ⁰ C	Time	Yield
1.	Methanol	R.T.	40	30
2.	Methanol	60	40	74
3.	Methanol	80	20	95
4.	Methanol	100	20	84
5.	Ethanol	80	20	87
6.	DMF	80	40	82
7.	CH ₃ CN	80	40	78
8.	CH ₂ Cl ₂	80	60	58

To estimate the generalization of the procedure, several diversified examples illustrating the present method for the synthesis of 2-aryl benzoxazole was studied (Table 4). The cyclization reaction 2-amino phenol was checked by treating with a wide range of electron-donating (such as -OH, -MeO, -NH₂, -CH₃) or electron-withdrawing (-NO₂, -X) substituted aldehydes in presence of SiO₂/MgSnO₃ catalyst. When the reaction was carried out, it was found that, the aromatic aldehydes with electron-donating groups react faster as compared to electron-withdrawing groups. Also Found that, the reaction of substituted aldehydes with 2-amino phenol in presence of methanol and SiO₂/MgSnO₃ (0.4 mol) at 80⁰C temperature gave an excellent yield of 2-amino benzoxazole. The results obtained are given in table 4. All the products obtained were characterized by IR, ¹H NMR, ¹³C NMR and Mass spectroscopy.

Table-4: Synthesis of 2-aryl benzoxazoles in presence of SiO₂/MgSnO₃

Entry	Substituted Aldehydes	Time (Min)	Isolated Yield (%)
1.		40	87
2.		35	89
3.		40	83
4.		45	81
5.		35	87
6.		20	95
7.		30	91
8.		20	90

The reusability of catalyst was checked for the synthesis of 2-aryl benzoxazole as shown in table 5. After the successive run, from the reaction mixture, the catalyst was recovered, washed with acetone several times and dried in an oven at 120⁰C for 3 hrs. Before use and tested for its activity in the subsequent run. The catalyst was tested for 5 runs and found that the catalyst gave very good reusability property.

Table-5: Reaction Run in the Presence of a Recycled Catalyst

Entry	Catalyst Run	Time (Min)	Yield*
1.	1	20	93
2.	2	20	93
3.	3	20	92
4.	4	20	89
5.	5	20	90

*All reactions (Table.1-4) are carried at 80⁰C using SiO₂/MgSnO₃.

CONCLUSION

In Conclusion, we have developed an efficient and simple alternative for the synthesis of 2-aryl benzoxazole using SiO₂/MgSnO₃ catalyst at 80°C temperature. Noticeable advantages of this new method are simple procedure, easy workup procedure, short reaction time with good yield, use of cheap, non-toxic, environmentally benign nature make it an attractive process and the employment of a cost-effective catalyst.

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