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Research Article

Synthesis of some benzothiazole derivatives by using zinc oxide nanoparticles.

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ABSTRACT
Benzothiazole is the heterocyclic compound formed from 2-aminothiophenol substituted aromatic aldehyde by using zinc oxide nanoparticles and because of their diverse biological activity. A new method for synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i) are developed by using simple method and good yields. We have synthesized more than thirty molecules in gram scale. This method is extremely useful for the synthesis of Benzothiazole derivatives in excellent yields.

KEYWORDS
2-aminothiophenol, Benzothiazole, aldehydes, zinc oxide nanoparticles

1. INTRODUCTION

Metal nanoparticles have a high specific surface area and a high fraction of surface atoms. Because of the unique physicochemical characteristics of nanoparticles, including catalytic activity, optical properties, electronic properties, antibacterial properties, and magnetic properties[1-6] they are gaining the interest of scientist for their novel methods of synthesis. Over the past few years, the synthesis of metal nanoparticles is an important topic of research in modern material science. Nano-crystalline silver particles have been found tremendous applications in the fields of high sensitivity biomolecular detection, diagnostics, antimicrobials, therapeutics, catalysis and micro-electronics. However, there is still need for economic commercially viable as well as environmentally clean synthesis route to synthesize the silver nanoparticles. Silver is well known for possessing an inhibitory effect toward many bacterial strains and microorganisms commonly present in medical and industrial processes [7]. In medicines, silver and silver nanoparticles have a ample application including skin ointments and creams containing silver to prevent infection of burns and open wounds [8], medical devices and implants prepared with silver-impregnated polymers [9]. In textile industry, silver-embedded fabrics are now used in sporting equipment [10]. Nanoparticles can be synthesized using various approaches including chemical, physical, and biological. Although chemical method of synthesis requires short period of time for synthesis of large quantity of nanoparticles, this method requires capping agents for size stabilization of the nanoparticles. Chemicals used for nanoparticles synthesis and stabilization are toxic and lead to non-ecofriendly byproducts. The need for environmental non-toxic synthetic protocols for nanoparticles synthesis leads to the developing interest in biological approaches which are free from the use of toxic chemicals as byproducts. Thus, there is an increasing demand for “green nanotechnology” [11]. Many biological approaches for both extracellular and intracellular nanoparticles synthesis have been reported till date using microorganisms including bacteria, fungi and plants [12, 13]. Plants provide a better platform for nanoparticles synthesis as they are free from toxic chemicals as well as provide natural capping agents. Moreover, use of plant extracts also reduces the cost of microorganisms isolation and culture media enhancing the cost competitive feasibility over nanoparticles synthesis by microorganisms sometimes the synthesis of nanoparticles using various plants and their extracts can be advantageous over other biological synthesis processes which involve the very complex procedures of maintaining microbial cultures [14, 15].

This section deals with the synthesis of various Substituted benzimidazole derivatives using nanoparticles capped by plant material .This is Novel approach used for synthesis of benzimidazole derivatives.

2. MATERIALS AND METHODS

Zinc oxide nanoparticles these nanoparticles act as dehydrating agents gives excellent yield of the product.

Scheme 1. Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

2.1.General procedure synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

To a mixture of 2-aminothiophenol substituted aromatic aldehyde 10 mmole and 2-aminobenzenethiol 10 mmole along with crushed with 0.4 gm of zinc oxide grind in pristle and mortar for specified time ,then after TLC complies extract the product using organic solvent ,purify product over column using chloroform and methanol.

Table 1. Physical data of synthesized compounds (3a-i)

Sr. No.	Entry	Aromatic group	Time (min)	Yield (%)	MP (^{0}C)
1.	3a	C_6H_5	30	78	116
2.	3b	2-ClC_6H_4	25	90	75
3.	3c	4- ClC_6H_4	25	90	96
4.	3d	4- $MeOC_6H_4$	40	70	118
5.	3e	2-$NO_2C_6H_4$	30	90	118
6.	3f	3- $NO_2C_6H_4$	25	90	184
7.	3g	4- $NO_2C_6H_4$	25	90	196
8.	3h	4-MeC_6H_4	30	67	85
9.	3i	2-OH-C_6H_5	30	78	132

Silica nanoparticles coated by plant material coated and used in synthesis of benzimidazole using various aromatic aldehydes in microwave and sonication method.

Scheme 2. Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

2.2. General procedure Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

To a mixture of 2-aminobenzenethiol, substituted aromatic aldehyde 10 mmole along with crushed with 0.3 gm of silica nanoparticles subjected to microwave reaction in domestic microwave for specified period. Reaction is monitored by thin layer chromatography, then after

TLC complies extract the product using organic solvent, purify product over column using chloroform and methanol.

Table 2. Physical data of synthesized compounds (3a-i)

Sr. No.	Entry	Aromatic group	Time (min)	Yield (%)	MP (^{0}C)
1.	3a	C_6H_5	25	75	117
2.	3b	2-ClC_6H_4	20	78	78
3.	3c	4- ClC_6H_4	20	84	95
4.	3d	4- $MeOC_6H_4$	21	80	118
5.	3e	2-$NO_2C_6H_4$	24	90	156
6.	3f	3- $NO_2C_6H_4$	24	92	189
7.	3g	4- $NO_2C_6H_4$	24	90	194
8.	3h	4-MeC_6H_4	25	78	87
9.	3i	2-OH-C_6H_5	23	80	136

Aluminum oxide nanoparticles are prepared by using plant extract and used in synthesis.

Scheme 3. Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

2.3. General procedure Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

To a mixture of 2-aminothiophenol substituted aromatic aldehyde 10mmole and orthophenyldiamine 10mmole along with crushed with 0.3 gm of aluminum oxide nanoparticles add 5-6 ml of water subjected to sonication reaction for specified period .Reaction is monitored by thin layer chromatography. Then after TLC complies extract the product using organic solvent, purify product over column using chloroform and methanol.

Table 3. Physical data of synthesized compounds (3a-i)

Sr. No.	Entry	Aromatic group	Time (min)	Yield (%)	MP (^{0}C)
1	3a	C_6H_5	40	67	115
2	3b	2-ClC_6H_4	50	80	76
3	3c	4- ClC_6H_4	56	80	94
4	3d	4- $MeOC_6H_4$	60	58	116
5	3e	2-$NO_2C_6H_4$	56	86	154
6	3f	3- $NO_2C_6H_4$	60	85	184
7	3g	4- $NO_2C_6H_4$	60	85	193
8	3h	4-MeC_6H_4	70	60	85
9	3i	2-OH-C_6H_5	70	70	134

Scheme 4. Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

2.4. General procedure Synthesis of 2-(substituted phenyl)benzo[d]thiazole (3a-i)

To a mixture of 2-aminothiophenol substituted aromatic aldehyde 10mmole and orthophenyldiamine 10mmole along with crushed with 0.2 gm of plant material silver coated nanoparticles add 5-6 ml of water subjected to sonication reaction for specified period .Reaction is monitored by thin layer chromatography. Then after TLC complies extract the product using organic solvent, purify product over column using chloroform and methanol

Table 4. Physical data of synthesized compounds (3a-i)

Sr. No.	Entry	Aromatic group	Time (min)	Yield (%)	MP (^{0}C)
1	3a	C_6H_5	60	78	113
2	3b	2-ClC_6H_4	55	67	74
3	3c	4- ClC_6H_4	55	80	96
4	3d	4- MeOC_6H_4	50	84	116
5	3e	2-$NO_2C_6H_4$	50	87	152
6	3f	3- $NO_2C_6H_4$	54	88	184
7	3g	4- $NO_2C_6H_4$	54	88	193
8	3h	4-MeC_6H_4	60	70	84
9	3i	2-OH-C_6H_5	55	74	132

3. RESULTS AND DISCUSSION

In this method (Scheme 1) we have synthesized zinc oxide nanoparticles which are characterized by SEM and TEM. During synthesis of these nanoparticles we are using plant extract. Plant material contains various biological polymers which act as capping agent. These nanoparticles act as dehydrating agent and gives good yield of product, this reaction gives better yield and catalyst require in very small amount and recyclable.

In this method (scheme 2) we have synthesize zinc oxide nanoparticles which are characterized by SEM and TEM. During synthesis of these nanoparticles we are using plant extract .Plant material contains various biological polymers which act as capping agent. Method of synthesis of nanoparticle is green and synthesis of Benzothiazole derivatives using microwave method. This reaction gives 80-90 % yield and good purity.

In this method we can synthesize zinc oxide nanoparticles which are characterized by SEM and TEM. During synthesis of these nanoparticles we are using plant extract .Plant material contains various biological polymers which act as capping agent. Aluminum oxide nanoparticles under sonication which gives good yield, in small time with high purity.

Silver nanoparticles are prepared by using lemon grass and used for preparation of benzimidazole using sonication method.

Nanoparticles are prepared by green method using plant extract and silver nitrate. These nanoparticles are used for synthesis of Benzothiazole and this reaction gives good yield and purity at room temperature.

4. CONCLUSION

In summary, an efficient and convenient synthesis and this is better approach for the synthesis of benzothiazole derivatives in good to excellent yields. The importance of the substituted benzothiazole derivatives would render this protocol attractive for both synthetic and medicinal chemistry.

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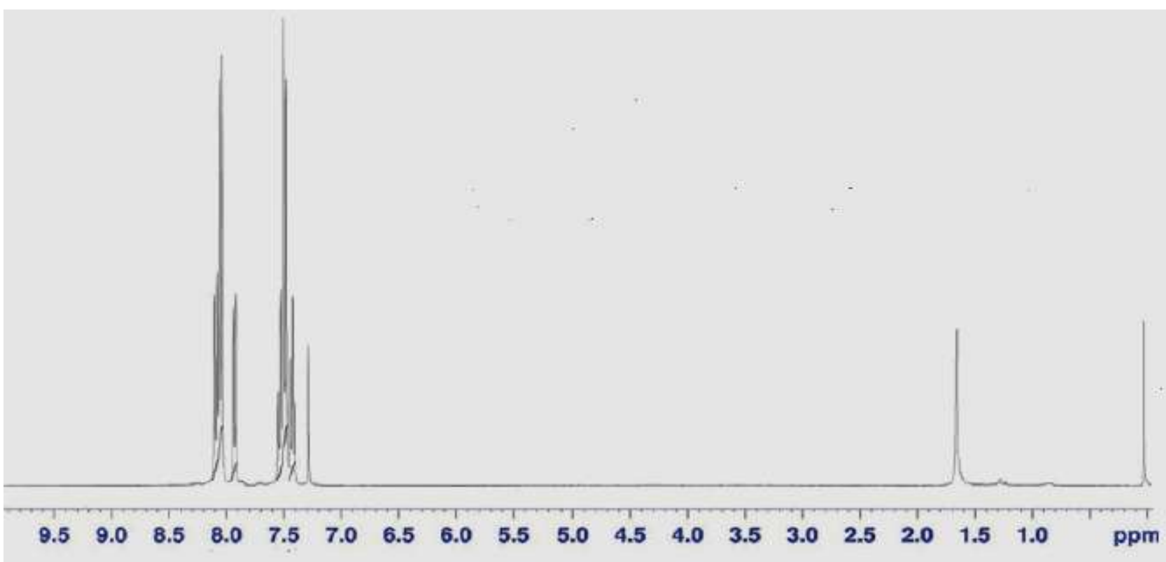

Figure 1. ^{1}H-NMR spectra of 2-(4-chlorophenyl) benzothiazole

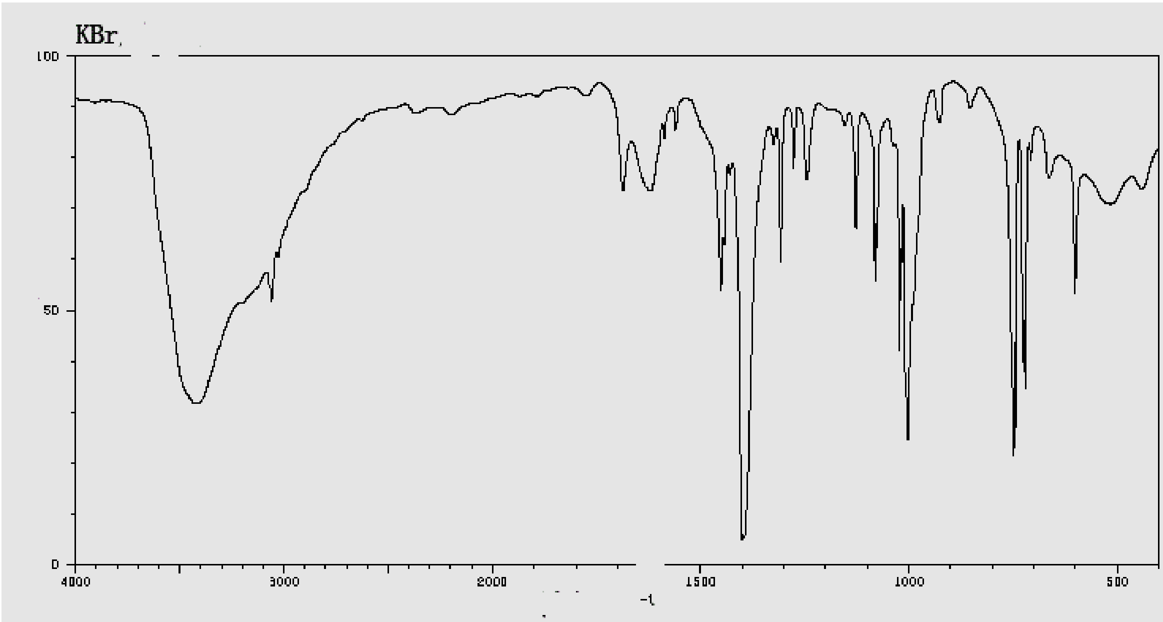

Figure 2. IR spectra of 2-(4-chlorophenyl) benzothiazole.