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$Zn(OAc)_2.2H_2O$: An efficient catalyst for the one-pot synthesis of 2-substituted benzothiazoles

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Abstract: By condensation from aldehyde and 2-aminothiophenol at 80 ^{0}C, environmentally friendly, easily accessible, and inexpensive $Zn(OAc)_2.2H_2O$ (5 mol%) was used to synthesize electronically and structurally divergent benzothiazoles in moderate to good yields (67-96%). All the 2-substituted benzothiazole analogues were thoroughly characterized by IR, NMR and mass spectral analyses.

Keywords: Catalysis; aldehydes; 2-aminothiophenol; benzothiazoles; zinc acetate; solvent-free condition.

1. Introduction

Heterocyclic compounds are tremondously important organic molecules that represent the widest range of chemical compounds with important industrial and clinical applications.[1-3] Highly reactive 2-aminobenzothiazoles are often used as reactants or reaction intermediates to create a range of fused heterocyclic compounds. Numerous anticancer drugs and other pharmaceutical tools contain the benzothiazole nucleus as its primary structural component (Figure 1).[4] The two main protocols for producing benzothiazoles that have been documented are the cyclization of thiobenzanilides and the condensation of 2-aminothiophenol with aldehydes, carboxylic acids, acid chlorides, or esters.[5-9] Other common protocols include the microwave-mediated reaction of 2-aminothiophenol with β-chlorocinnamaldehydes, the Suzuki biaryl coupling of benzothiazoles with aryl bromides and 2-bromobenzothiazole with arylboronic acids, as well as the reaction of thiophenols with aromatic nitriles.[10-25] However, the majority of these approaches have one or more drawbacks, including high reaction temperatures, harsh reaction conditions, multistep processes, prolonged reaction times, the need for an excessive amount of reagents, and the employment of expensive, air-sensitive catalysts, among others.

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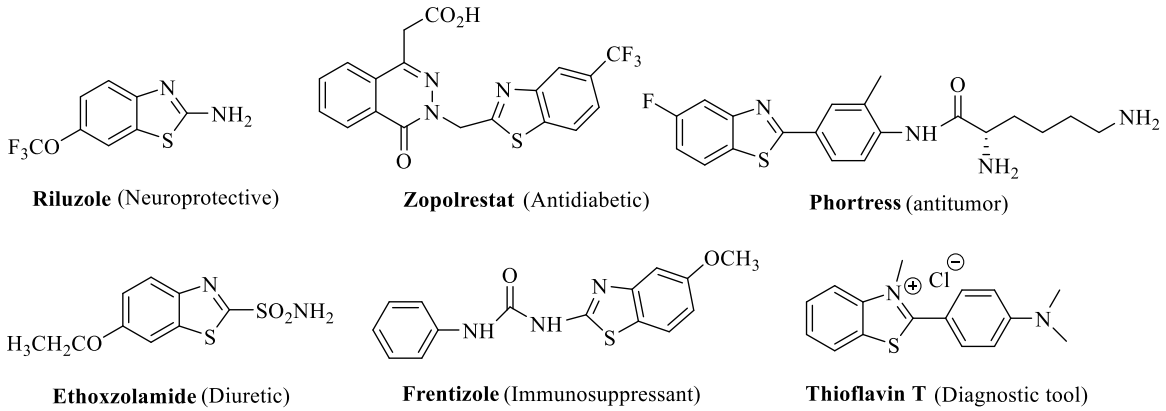


Figure 1. Biologically active 2-substituted benzothiazole derivatives

2. Background

There is still a need to create a straightforward and mild procedure for synthesizing 2-substituted benzothiazoles without the use of hazardous chemicals or reagents. Due to their low production costs and simplicity of usage, applications of solvent-free based synthetic methods to synthesize pharmacologically relevant heterocyclic compounds have grown in favour.[26-27] The Zn-based compounds have been utilized in a variety of different catalytic processes in the past.[28-29] After doing an exhaustive search of the relevant published material, we discovered that $Zn(OAc)_2•2H_2O$ has not been utilized for the reaction of interest. Zinc acetate is inexpensive, easily accessible, and resistant to change in the presence of moisture and air under standard laboratory conditions.[30-44]

Pasha *et al*. used a grindstone approach to synthesize 2-substituted benzoxazoles from 2-aminophenol and substituted aldehydes utilizing a $Zn(OAc)_2•2H_2O$-catalyzed, easy, and clean procedure.[36] Bhanage and colleagues have published a study on the $Zn(OAc)_2•2H_2O$-catalyzed synthesis of benzimidazoles and benzothiazole derivatives utilising *N*-substituted formamides as C1-sources. They used high temperatures (120 ^{0}C) and expensive PMHS for the reaction to progress under neat conditions.[44] These results and our own expertise in zinc catalysis,[32-34] encouraged us to synthesize 2-substituted benzothiazoles by condensation from aldehyde and 2-aminothiophenol using $Zn(OAc)_2•2H_2O$ as catalyst.

3. Experimental

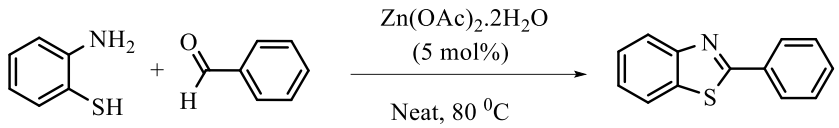
*General procedure for the synthesis of 2-substituted benzothiazoles (**3a-p**)*

Aldehyde (**2**, 1 mmol), 2-aminothiophenol (**1**, 1 mmol), and $Zn(OAc)_2•2H_2O$ (5 mol%) were vigorously heated at 80 °C in an open environment until the entire mixture solidified. The reaction's progress was observed by TLC for the duration listed in Table 3 throughout this period. Following completion, the solid was washed with water, and the product was either purified using column chromatography using a short silica-gel column or crystallized from ethanol. Each product (**3**) was adequately characterized with the help of FT-IR, NMR, and mass spectrum analysis, and the results were compared with those found in the relevant literature (Supporting Information available).[10-25]

*Compound **3d***: Yellow solid; mp 151-152 ºC; IR (KBr): ν 3658, 2921, 1318, 1115, 839 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ = 8.09 (s, 1H), 8.06 (d, *J* = 8.59 Hz, 1H), 8.03 (dd, *J* = 7.41, 2.12 Hz, 2H), 7.92 (d, *J* = 8.59 Hz, 1H), 7.48-7.51 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ= 168.63, 152.90, 136.64, 133.13, 131.35, 129.89, 129.13, 127.61, 124.29, 124.18, 118.80

4. Present Study

Initially, the coupling reaction was screened for 2-aminothiphenol (**1a**, 1 mmol) and benzaldehyde(**2a**, 1 mmol) as substrates for the model reaction and toluene as solvent in the presence of $Zn(OAc)_2•2H_2O$ (10 mol%) at ambient temperature (Scheme 1).



1a **2a** **3a**

Scheme 1. $Zn(OAc)_2•2H_2O$-catalyzed synthesis of 2-substituted benzothiazoles

After stirring the reaction mixture for a period of twenty-four hours, thin-layer chromatography (TLC) and further purification revealed that there was a small quantity of corresponding benzothiazole production (**3a**). When determining whether or not a chemical reaction could be successful, one important consideration was the temperature at which the reaction took place. Above 80 °C, there was no notable change in the yield of the product, which reached its maximum of 72% at that temperature. The reaction was carried out at a temperature of 80 °C in different solvents, including acetonitrile, DMF, ethyl acetate, chloroform and solvent-free conditions. $Zn(OAc)_2•2H_2O$ (ZA) was utilized as the catalyst during this process (Table 1). In contrast to the reaction that was carried out in the chosen solvents, we found that conditions without any solvents produced the intended product with an excellent yield in 30 min (entries 1-3, Table 1). In addition, it was found that a catalyst concentration of 5 mol% was sufficient to maintain the reaction and give the maximum isolated yield (94% yield, entry 2 in Table 1).

Table 1. Effect of solvent and catalyst loading

Entry	Solvent	Yield (%)[a]
1	solvent-free[b]	90
2	solvent-free[c]	94
3	solvent-free[d]	78
4	toluene	72
5	$CHCl_3$	42
6	DMF	37
7	CH_3CN	18
8	EtOAc	7

[a]Isoalted yields. [b]ZA, 10 mol%); [c]ZA, 5 mol% and [d]ZA, 2.5 mol%

We compared the catalytic effectiveness of different readily available zinc salts for the control reaction in order to evaluate performance of $Zn(OAc)_2•2H_2O$ in the test reaction. The findings were shown in Table 2. It was noted that only minimal conversion of the corresponding benzothiazole was accomplished when all other zinc salts were utilized as catalysts. When different zinc salts were evaluated, $Zn(OAc)_2•2H_2O$'s catalytic effectiveness outperformed them in terms of isolated yield and reaction time (entry 6, Table 2).

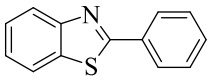
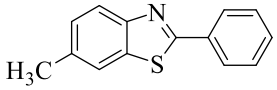
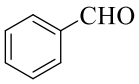
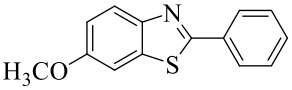
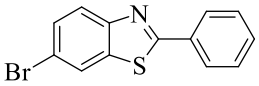
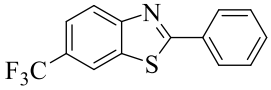
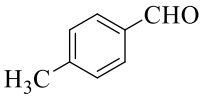
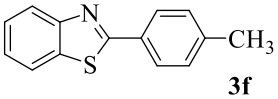
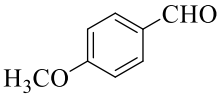
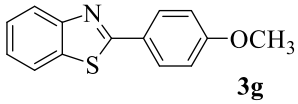
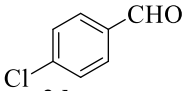
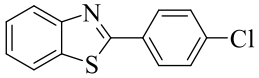
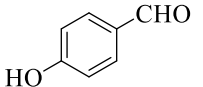
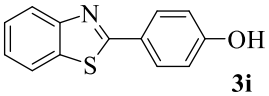
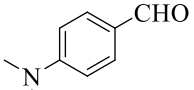
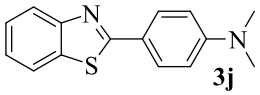
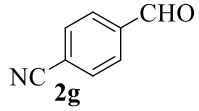
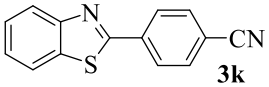
In order to evaluate the performance of this condensation method, a number of 2-substituted benzothiazoles (**3a-p**) were prepared by reacting substituted 2-aminothiophenols (**1a-e**) with aromatic, aliphatic, and heterocyclic aldehydes **(2a-l)** under the optimized reaction conditions (Table 3).

Table 2. Effect of zinc salts on control reaction

Entry	Zinc salt	Reaction Time (h/min)	Yield[a] (%)
1	Zinc granules (crystalline)	20/	-
2	$Zn(NO_3)_2$	12/	17
3	$ZnCl_2$	/60	55
4	$ZnSO_4$	12/	12
5	$Zn_3(PO_4)_2$	12/	28
6	$Zn(OAc)_2•2H_2O$	/30	94

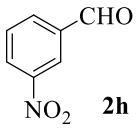
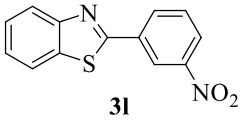
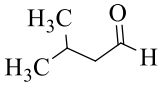
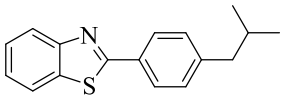
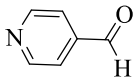
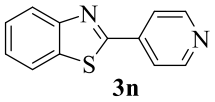
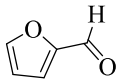
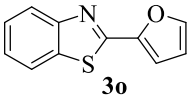
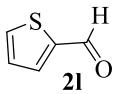
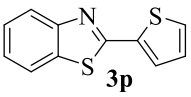
[a]Isoalted yields.

Table 3. $Zn(OAc)_2•2H_2O$-catalyzed synthesis of 2-substituted benzothiazoles

Entry	Aldehyde	Product	Time (min)	Yield (%)[a]	Melting point (^oC)
1		 **3a**	30	94	112-113
2		 **3b**	60	92	125-126
3	 **2a**	 **3c**	60	94	116-117
4		 **3d**	75	89	151-152
5		 **3e**	60	91	156-157
6	 **2b**	 **3f**	60	89	82-83
7	 **2c**	 **3g**	45	96	120-121
8	 **2d**	 **3h**	45	90	118-119
9	 **2e**	 **3i**	60	85	228-230
10	 **2f**	 **3j**	60	85	171-172
11	 **2g**	 **3k**	60	90	172-173

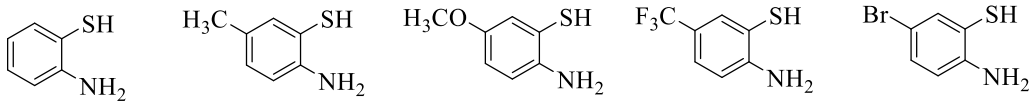
[a]Isolated yields after column chromatography

(Continued..)

Entry	Aldehyde	Product	Time (min)	Yield (%)[a]	Melting point (^oC)
12	 **2h**	 **3l**	30	94	112-113
13	 **2i**	 **3m**	120	67	colourless oil
14	 **2j**	 **3n**	90	79	136-137
15	 **2k**	 **3o**	75	81	101-102
16	 **2l**	 **3p**	60	84	99-100

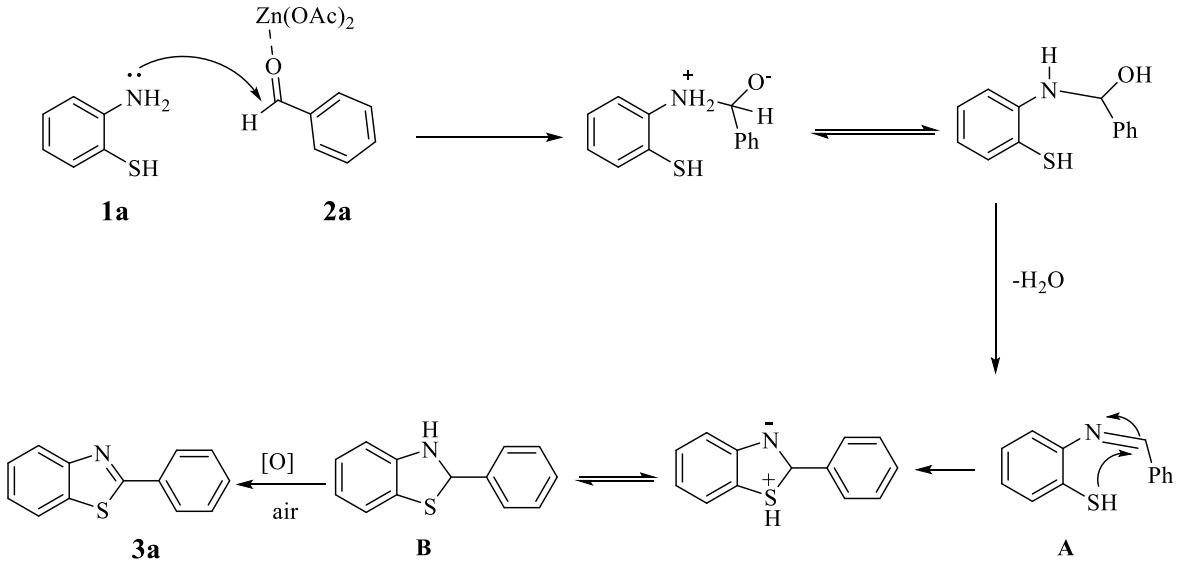
[a]Isolated yields after purification

[b]Substituted 2-thiophenols



(1a) **(1b)** **(1c)** **(1d)** **(1e)**

Both the electron-donating and electron-drawing groups that can be found in aromatic aldehydes were able to interact without any problems (**3f-l**).



Scheme 2. Plausible mechanism for the formation of benzothiazoles

The reaction also included alkyl aldehydes, such as isobutyraldehyde, which resulted in a moderate amount of the corresponding compound being produced (**3m**). It was interesting to note that the reaction involving heterocyclic aldehydes, resulted in the generation of corresponding benzothiazoles in good yields (**3n-p**). We were pleased with the results, so applied the process to the synthesis of divergent 2-aminothiophenols, and obtained the corresponding products in yields that ranged from moderate to good (**3b-e**, Table 3).

The most plausible mechanism of the reaction is the creation of an intermediate imine (**A**), which may undergo cyclization with the 2-aminothiophenol's *o*-thiol group to yield 2-substituted-2,3-dihydro-benzothiazole (**B**), which was then aerially oxidized to generate benzothiazole (**3a**), as shown in Scheme 2.

In Table 4, we compared our findings to prior reports,[19-25] and the results showed that $Zn(OAc)_2•2H_2O$ had good catalytic performance for the synthesis of benzothiazole. Because of the mild working conditions, shorter reaction times, and solvent-free environment, the approach was more appealing from the standpoint of green chemistry.

Table 4. Comparison of the present work with the previous literature for the synthesis of 2-phenylbenzothiazole

Entry	Catalyst	Quantity	Condition	Time h/min	Solvent	Yield[ref]
1	Imidazolium chlorozincate (II) ionic liquid supported into Fe_3O_4 nanoparticles	4 mg	Sonication@70^{0}C	/30	Solvent-free	90[18]
2	Cetyltrimethyl ammonium bromide (CTAB)	5 mol%	100 ^{o}C	1.5/	H_2O	98[14]
3	I_2	50 mol%	100 ^{o}C	/25	DMF	88[11]
4	4-Methoxy TEMPO	5 mol%	100/120 ^{o}C	9/	Xylene	80[19]
5	**$Zn(OAc)_2•2H_2O$**	**5 mol%**	**80 ^{o}C**	**/30**	**Solvent-free**	**94**
6	Bi_2O_3 nanoparticles	10 mol%	60 ^{o}C	1/	Ethanol	95[21]
7	H_2O_2/HCl	30% and 37% respectively	RT	/45	-	90[22]
8	Poly[4-diacetoxyiodo] styrene (PDAIS)	1.5 equiv.	RT	/10	CH_2Cl_2	92[23]
9	NH_4Cl	70 mol%	RT	/40	MeOH:H_2O	80[24]
10	Laccase (Suberase)	-	RT	24/	CH_3CN and acetate buffer	85[25]

In conclusion**,** The one-pot condensation of substituted 2-aminothiophenols and aldehydes at 80 ^{0}C under neat conditions has been successfully achieved for the first time using $Zn(OAc)_2•2H_2O$ as the only catalyst. The developed method has several advantages such as use of environmentally benign, inexpensive and readily available catalyst. Furthermore, the reaction did not produce any undesirable byproducts.

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Supporting Information

Supporting information accompanies this paper on http://www.acgpubs.org/journal/organic-communications

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