Iranian Journal of Catalysis 9(2), 2019, 173-179

IRANIAN JOURNAL OF CATALYSIS

Acacia concinna pod catalyzed synthesis of 2-arylbenzothia/(oxa)zole derivatives

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Received 15 June 2018; received in revised form 22 March 2019; accepted 1 April 2019

ABSTRACT

The expeditious synthesis of 2-aryl-benzothia/(oxa)zoles was carried out by the condensation of 2-aminothiophenol/ 2-aminophenol and diverse aryl aldehydes in presence of *Acacia concinna* as a biocatalyst under microwave irradiation. The catalytic process is associated with excellent yields, greener reaction conditions and the environmentally friendly microwave technique which are the striking features of the present protocol.

Keywords*: Acacia concinna, 2-Aminophenol, 2-Aminothiophenol, 2-Arylbenzothia/(oxa)zoles, Microwave irradiation.*

1. Introduction

Heterocyclic compounds are the important key structural features of a large number of biologically active natural products, pharmaceutical compounds and agrochemicals. Benzoxazoles and their derivatives are potential heterocyclic compounds which have some applications in medicinal chemistry, particularly in drug discovery and are found in many biologically active natural products and pharmaceutical targets [1].

Also, benzothiazole derivatives have significant biological and pharmaceutical activities, such as antitumor [2], antimicrobial [3], antiglutamate/ antiparkinson [4], broad spectrum Ca^{2+} channel antagonist [5], anti-inflammatory [6], anti-stress ulcer [7], antibiotics [8], and antiviral activities [9].

The 2-substituted benzoxazoles were also synthesized by various catalysts such as NaCN [10], SBA-Pr-SO_3H [11], $PhB(OH)_2$/KCN [12], Fe_3O_4@SiO_2@Am-PPC-SO_3H][HSO_4] [13] and CdO nanoparticles [14]. Under microwave irradiation, the synthesis of benzoxazole using some catalysts like copper-catalyzed domino annulation [15], solid supported reagents like polymer-bound *p*-toluene sulfonic acid [16], H_3PO_4 [17] and I_2 [18] had been reported.

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2-Substituted benzothiazole is synthesized using several catalysts including $Pd(PPh_3)_4/K_2CO_3$ [19], DMSO/120°C [20], ionic liquid 1-phenyl-3-methyl imidazolium bromide [21], $Al(HSO_4)_3$ [22], Nano-CrY zeolite [23], Ag@TiO_2 [24], H_2SO_4-SiO_2 [25] and $AlCl_3.6H_2O$ [26]. On the other hand, benzothiazoles were synthesized under microwave irradiation using some catalysts such as *p*-TsOH [27], SiO_2 [28], acetic acid [29] and anhydrous $CuSO_4/ZrOCl_2.8H_2O$ [30].

Green chemistry follows a set of principles that minimizes or eliminates the use or generation of hazardous substances in the design, manufacture and applications of chemical products [31,32]. Bio-catalyzed reaction is the oldest chemical transformations known to humans with great advantages of green chemistry. The present protocol describes the use of *Acacia concinna* for synthesis of benzothia/(oxa)zole with 2-aminothiophenol/ 2-aminophenol and various aldehydes. It is found that they have surfactant properties similar to dodecylbenzene sulfonates [33]. The surfactant contains saponin of acacic acid having trihydroxy monocarboxylic acid as a functional group (Fig. 1). The aqueous extract of these pods displayed acidic pH (2.1) which is due to existence of a carboxylic group in the monoterpenoid portion. Despite impressive catalytic potential of *Acacia concinna*, its utility in synthetic chemistry was not fully exploited [34].

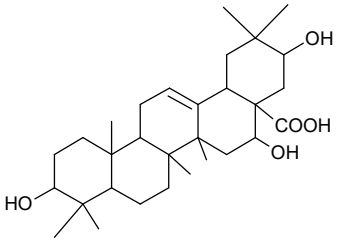


Fig. 1. Structure of Acaciac acid in the saponin of pods of *Acacia concinna*.

This spurred us to investigate the compatibility of *Acacia concinna* as a catalyst in the synthesis of biologically active compound like benzothia/(oxa)zole (Scheme 1).

2. Experimental

All the chemicals were purchased from Sigma Aldrich, Spectrochem and Thomas Baker and were used without further purification. All reactions were carried out in dried glassware. IR spectra were recorded by an Agilent Technologies Cary 630 FTIR spectrometer. Bruker AC NMR spectrometer (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) was used to record NMR spectra using $CDCl_3$ as a solvent and chemical shifts are expressed in parts per million (ppm) value with TMS as the internal reference and coupling constants are expressed in hertz (Hz). The microwave oven Cata R system was utilized for this work (700 W, 120°C). The reaction was performed in glass tube (10 mL). Melting points were determined by melting point apparatus with an open capillary and were uncorrected. The mass spectra were recorded on a Shimadzu QP2010 GCMS.

2.1. General procedure for the preparation of catalyst

In 250 mL beaker, 10 g of *Acacia concinna* fruit powder was added into 100 mL water and boiled for 15 min. The resulting solution was then filtered off, and the aqueous extract was used as a catalyst (10 %, w/v) for the preparation of benzothia/(oxa)zole derivatives.

2.2. General procedure for synthesis of 2-substituted benzothia/(oxa)zole derivatives

2.2.1. Conventional Method

In a 25 mL round bottom flask, a mixture of 2-aminothiophenol/2-aminophenol (1 mmol), aromatic aldehyde (1 mmol) and a catalytic amount of *Acacia concinna* 5 mL (10 %, w/v) were stirred at room temperature. After the complete conversion of the substrates as monitored by TLC (*n*-hexane: ethyl acetate, 1:4), water (5 mL) was added and stirred continuously until solid was obtained in the round bottom flask. The resulting solid was filtered, washed with water, and purified by recrystallization from EtOH. The structures of the prepared products were characterized by the spectral analysis.

2.2.2. Microwave Irradiation

In a 25 mL round bottom flask, aromatic aldehyde (1 mmol) and 2-aminothiophenol/2-aminophenol (1 mmol) were thoroughly mixed, then a catalytic amount of *Acacia concinna* 5 mL (10 % w/v) was added and the reaction mixture was kept in the microwave oven at optimized power level 40% with intermittent cooling after each 10 sec of irradiation. Afterwards, the procedure is similar to conventional method.

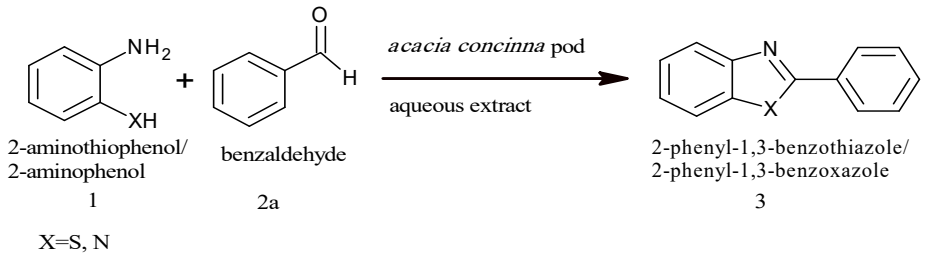
Spectral data of new compound

*2- (4- hydroxy- 3- ethoxyphenyl)- 1,3- Benzoxazole (**Table 3, entry 31**):*

m.p.= 150-152 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ= 7.15-7.21 (m, 5H, Benzoxazole-H4, H5, H6, H7, Ph-H5), 6.58-6.71 (m, 2H, Ph-H2, H6), 5.38 (s, 1H, OH), 4.05 (s, 2H, O-CH_2), 1.19 (s, 3H, CH_3) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ= 165.4 (Benzoxazole-C2), 152.8 (Benzoxazole-C3a), 149.2 (Ph-C3), 144.5 (Ph-C4), 139.9 (Benzoxazole-C7a), 126.7(Ph-C1), 124.6 (Benzoxazole-C5), 123.2 (Benzoxazole-C6), 122.5 (Benzoxazole-C4), 119.9 (Benzoxazole-C7), 115.8 (Ph-C5), 110.2 (Ph-C2), 109.9 (Ph-C6), 63.7 (O-CH_2), 15.5 (CH_3) ppm. IR: $\bar{\nu}$= 3126, 2925, 2087, 1632, 1517, 148, 1173, 931, 741, 696, 655 cm^{-1}. MS (EI): m/z= 256.27 $[M+H]^+$.

3. Results and Discussion

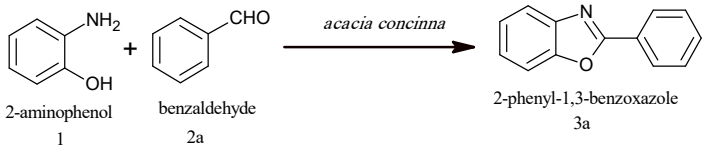
We have selected *Acacia concinna* pod as an acid catalyst for the synthesis of benzothia/(oxa)zoles. The reaction of 2-aminophenol and benzaldehyde was investigated as the model reaction under both conventional and microwave irradiations at different powers of microwave oven with or without the catalytic amount of *Acacia concinna*. Also, the reaction was carried out with or without catalyst by conventional thermal heating at various temperatures in different solvents (Table 1). It was observed that, by increasing in concentration of catalyst, reaction rate and the product yield is increased. Also, the microwave irradiation method provides excellent yield for 10 % (W/V) of catalyst amount (Table 2). The optimization clearly suggested that the power of microwave oven was 40 % (Table 2, entry 4) and conventional heating at room temperature in water solvent (Table 1, entry 9) and also the catalytic amount of 10 % (W/V) *Acacia concinna*. After optimizing the reaction condition, we generalized the scope of reaction for the diverse aldehydes.



Scheme 1. *Acacia concinna* catalyzed synthesis of 2-phenyl benzothia/(oxa)zole.

The reaction was applied to various aromatic aldehydes with 2-aminophenol/2-aminothiophenol in both conventional method and microwave irradiation (**Table 3**). This procedure generated a collection of functionalized benzothia/(oxa)zoles with a high level of functional group tolerance for both electron-withdrawing and electron-donating groups. It was observed that, electron donating substituent on the aromatic ring of aldehyde increases the reaction time and decreases the reaction rate (**Table 3, entries 3-14**), while electron withdrawing substituent decreases the reaction time and increases the reaction rate (**Table 3, entries 15-24**). Interestingly, hetero-aromatic aldehydes such as thiophene-2-carboxaldehyde and furfural aldehyde also undergo this reaction giving anticipated product in satisfactory yields (**Table 3, entries 25-28**). The reaction course is quite eco-friendly since no solvent is used in this procedure. It is observed that the use of the microwave irradiation technique has an intense effect on the acceleration of these reaction rates, providing significant yields of the products in shorter reaction period. In comparison with the microwave irradiation technique, the conventional method shows the prolonged reaction time with lower yields of the anticipated products.

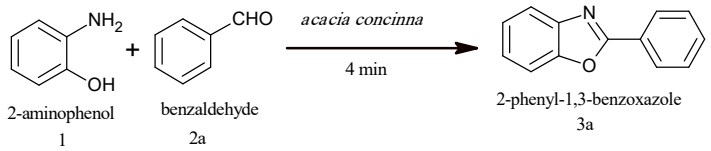
Table 1. Optimized conditions for synthesis of 2-phenyl benzoxazole by the conventional method.[a]



Entries	*Acacia concinna* pod % (W/V)	Solvent	Temp. (°C)	Time (min)	Yield (%)[b]
1	2.5	Water	r.t.	60	20
2	5	Water	r.t.	60	36
3	10	Acetone	r.t.	60	55
4	10	Methanol	r.t.	60	48
5	10	Acetonitrile	r.t.	60	38
6	10	Ethanol	r.t.	60	58
7	10	Ethyl acetate	r.t.	60	62
8	10	Chloroform	r.t.	60	46
9	**10**	**Water**	**r.t.**	**30**	**78**
10	10	Water	40	60	78
11	10	Water	50	60	78
12	20	Water	80	60	80

[a]Reaction condition: 2-aminophenol (1 mmol), benzaldehyde (1 mmol) and catalyst under the convention method.
[b]Isolated yields.

Table 2. Optimized conditions for synthesis of 2-phenyl benzoxazole by microwave irradiation.[a]



2-aminophenol (1) benzaldehyde (2a) 2-phenyl-1,3-benzoxazole (3a)

Entries	Power of MW oven (%)	*Acacia concinna* pod % (W/V)	Yield (%)[b]
1	40	--	25
2	40	5	70
3	20	10	55
4	**40**	**10**	**92**
5	40	15	92
6	40	20	92
7	60	10	92
8	80	10	92
9	100	10	92

[a]Reaction condition: 2-aminophenol (1 mmol), benzaldehyde (1 mmol) and catalyst under microwave irradiation, at 50°C for 4 minutes.
[b]Isolated yields.

The probable sequence of events of *Acacia concinna* catalyzed synthesis of 2-Phenyl benzoxazole from 2-aminophenol and benzaldehyde are shown in the Scheme 2. Initially *Acacia concinna* as an acid catalyst is protonated the carbonyl oxygen of aromatic aldehyde and form the intermediate **1**. Then, the amino alcohol **2** is produced by nucleophilic attack of the amino group of 2-aminophenol. After loss of water, Schiff's base is formed and then converted to the target molecule by intramolecular cyclization and subsequent air oxidation.

The advantages of Acacia concinna were compared with those of other catalysts. As it was shown in table 4, *Acacia concinna* is a highly effective catalyst for synthesis of 2-arylsubstituted benzoxazole derivatives by microwave irradiated through the condensation of aromatic aldehydes with 2-aminophenol. The high yield of products in a shorter reaction time, eco-friendly catalyst, mild reaction condition and easy workup method make this method more advantageous than existing methodology.

4. Conclusions

In summary, we have developed a facile and efficient protocol for the synthesis of 2-substituted benzothia/(oxa)zoles from the condensation reaction of aromatic aldehyde with 2-aminophenol/2-aminothiophenol using an inexpensive and non-toxic *Acacia concinna* as a catalyst in both conventional and microwave irradiation conditions. It was found that the microwave method is superior to the conventional method. The noteworthy aspects of the present protocol are the fast reaction rate, high yield, mild reaction conditions, cost-effective and environmentally friendly.

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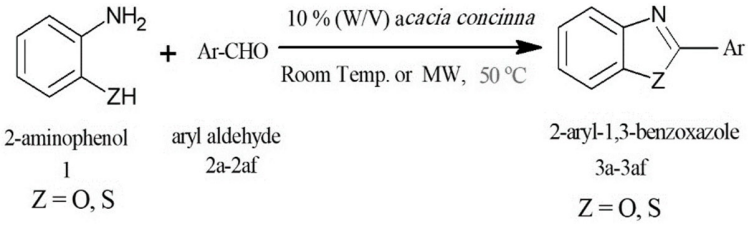
Table 3. Synthesis of 2-arylbenzoxazole by *Acacia concinna* catalyzed reaction of 2-aminophenol with aryl aldehydes under microwave irradiation and conventional heating conditions.[a]

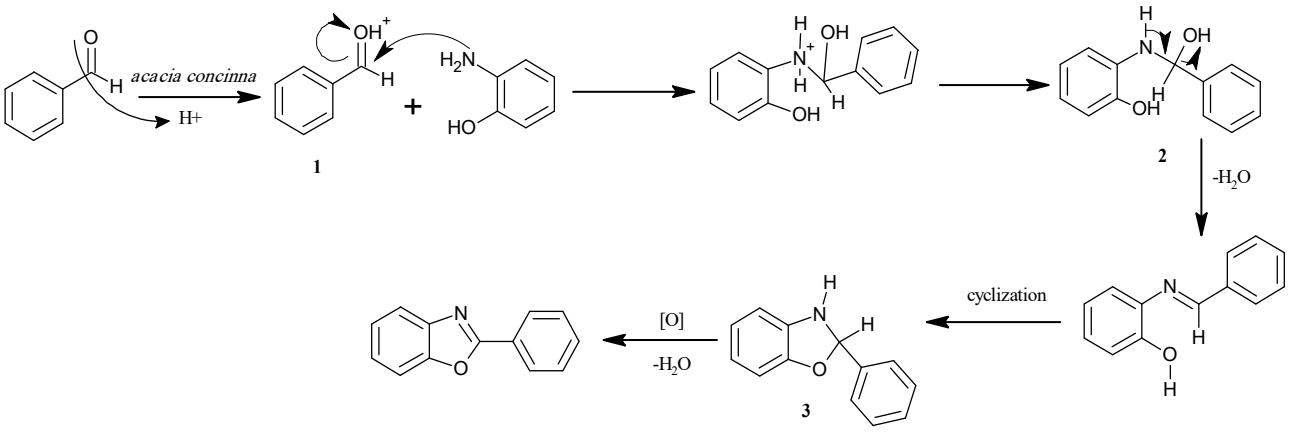


Entry	Aryl aldehyde (2)	Z	Time (min)	Product[b]	Yield[c] (%)	m.p.(°C)		Ref.
						Found	Reported	
1	Benzaldehyde	O	30 (4)	3a	78 (92)	98-100	100-102	[35]
2	Benzaldehyde	S	30 (4)	3b	72 (93)	108-110	110-112	[36]
3	4-Methybenzaldehyde	O	50 (5)	3c	70 (86)	114-116	116-118	[37]
4	4-Methybenzaldehyde	S	45 (4)	3d	75 (88)	80-82	82-84	[37]
5	3-MethoxyBenzaldehyde	O	55 (5)	3e	67 (85)	70-71	71.3-73.8	[38]
6	3-MethoxyBenzaldehyde	S	40 (5)	3f	70 (82)	98-100	100-102	[38]
7	4-MethoxyBenzaldehyde	O	55 (5)	3g	65 (86)	100-102	103-105	[39]
8	4-MethoxyBenzaldehyde	S	40 (5)	3h	70 (89)	118-120	118-120	[36]
9	2,5-Dimethoxy-benzaldehyde	O	40 (5)	3i	70 (88)	70-72	70-72	[40]
10	2,5-Dimethoxy-benzaldehyde	S	45 (4)	3j	65 (85)	104-106	104-106	[41]
11	4-Hydroxybenzaldehyde	O	40 (4)	3k	70 (91)	280-282	282-284	[42]
12	4-Hydroxybenzaldehyde	S	40 (5)	3l	75 (95)	226-228	228-230	[40]
13	2-Hydroxybenzaldehyde	O	50 (5)	3m	72 (93)	114-116	120-122	[43]
14	2-Hydroxybenzaldehyde	S	45 (4)	3n	75 (95)	130-132	130-132	[36]
15	4-Nitrobenzaldehyde	O	40 (4)	3o	75 (93)	263-265	268-270	[38]
16	4-Nitrobenzaldehyde	S	35 (3)	3p	80 (95)	224-226	222-224	[36]
17	4-Cynobenzaldehyde	O	45 (4)	3q	73 (92)	96-98	101-103	[37]
18	4-Cynobenzaldehyde	S	45 (3)	3r	80 (90)	164-166	164-166	[37]
19	4-Chlorobenzaldehyde	O	40 (4)	3s	68 (91)	150-152	153-154	[37]
20	4-Chlorobenzaldehyde	S	35 (4)	3t	72 (95)	110-112	112-114	[44]
21	4-Bromobenzaldehyde	O	40 (4)	3u	72 (92)	160-162	162-165	[45]
22	4-Bromobenzaldehyde	S	30 (3)	3v	75 (90)	128-130	128-130	[44]
23	2-Nitrobenzaldehyde	O	35 (4)	3w	76 (92)	268-270	270-272	[46]
24	2-Nitrobenzaldehyde	S	30 (3)	3x	82 (93)	134-136	136-138	[47]
25	2-Furfural	O	45 (5)	3y	69 (88)	86-88	89-90	[46]
26	2-Furfural	S	50 (6)	3z	75 (90)	98-100	100-102	[48]
27	Thiophene-2-carboxaldehyde	O	50 (5)	3aa	68 (86)	104-106	108	[46]
28	Thiophene-2-carboxaldehyde	S	55 (6)	3ab	75 (90)	96-98	96-98	[48]

Table 3. (*Continued*).

29	4-Hydroxy-3,5-dimethoxybenzaldehyde	O	50 (6)	3ac	72 (85)	210-212	203-204	[53]
30	4-Hydroxy-3,5-dimethoxybenzaldehyde	S	45 (5)	3ad	75 (92)	178-180	153.5-156.1	[54]
31	3-Ethoxy-4-hydroxybenzaldehyde	O	35 (4)	3ae	65 (82)	150-152	Novel	-
32	3-Ethoxy-4-hydroxybenzaldehyde	S	40 (4)	3af	75 (88)	126-128	125.6-126.5	[54]

[a]Reaction condition: 1 (1 mmol), 2a-2af (1 mmol) and catalyst 10 % (w/v) under microwave irradiation and conventional condition. The reaction time and yields under microwave irradiation condition are shown in the parenthesis.
[b]Products were characterized by their physical properties, their comparison with authentic samples, and their spectral (^{1}H NMR, ^{13}C NMR. IR, MS) analysis.
[c]Isolated yields.



Scheme 2: Plausible mechanistic pathway for the synthesis of 3a.

Table 4. Comparison of *Acacia concinna* with variety of catalysts for the synthesis of 3a.

No.	Catalyst used	Amount of catalyst	Temp (°C)	Time (min)	Yield (%)	Ref.
1	***Acacia concinna***	**10 % (W/V)**	**50 (MW)**	**4**	**92**	**This Work**
2	*p*-TSA	20 % mol	150	10	82	[49]
3	Iodine 190	15 % mol	140	15	87	[50]
4	Merrifield resin	25 % mol	150	60	80	[51]
5	PS-PPh_3 resin	20% mol	150	15	94	[52]
6	NaCN	100 % mol	r.t.	60	92	[10]
7	SBA-Pr-SO_3H	2.0 g	70	480	91	[11]
8	$PhB(OH)_2$/KCN	10 % mol	r.t.	240	48	[12]
9	*tert*-butyl hypochloride	10 % mol	70	60	85	[13]
10	H_3PO_3	15 % mol	110	10	90	[14]

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