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ORIGINAL PAPER

$FeCl_3$-promoted synthesis of 1,3,4-thiadiazoles under combined microwave and ultrasound irradiation in water

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Abstract An eco-friendly and efficient synthesis of substituted 1,3,4-thiadiazole derivatives has been developed. This aqueous heterogeneous approach proceeds smoothly and quickly under combined microwave and ultrasound irradiation in the presence of $FeCl_3$.

Keywords Microwave-assisted synthesis · Ultrasound · Heterogeneous · Water · Oxidations

Introduction

The concept of "ideal chemistry" has been widely adopted to meet the fundamental scientific principles of protecting human health and the environment while simultaneously achieving commercial value [1]. In this regard, the replacement of organic solvents is one of the most important goals. Although an ideal and universal green solvent for all problems does not exist, water is a widely explored green solvent [2–4]. Clearly, the development of a sustainable approach for synthesizing biologically active molecules is very important. In this field, combined microwave and ultrasound irradiation has proven to be a powerful tool for both speeding up chemical optimizations and for efficiently preparing new compounds [5–10].

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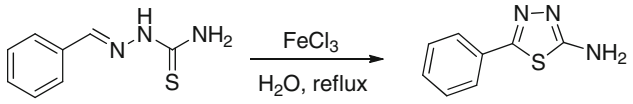
Over the years, heterocycles have become important synthetic intermediates that have found a variety of applications in medicinal, agricultural, and materials chemistry [11–13]. Although many important types of heterocyclic compounds have been synthesized in aqueous media [14–20], the synthesis of new and important types of heterocyclic compounds in water still attracts wide attention. 1,3,4-Thiadiazoles are very important heterocycles with great applicability in medicinal chemistry, agrochemistry, and so on. This core structure can be found in compounds with wide-ranging biological applications [21–27]. The literature reveals that most traditional synthetic methods for the synthesis of 1,3,4-thiadiazole derivatives suffer from one or more drawbacks, such as laborious work-up, strongly acidic conditions, low yields, and the use of organic solvents [28–32]. As part of our continued interest in the application of combined microwave and ultrasound irradiation (CMUI)-assisted heterogeneous reactions [33–35], we here report a mild, efficient, and environmentally benign approach for the synthesis of substituted 1,3,4-thiadiazoles via intramolecular cyclization or the three-component reaction of an aldehyde, a phenylisothiocyanate, and hydrazine hydrate under CMUI in a heterogeneous aqueous medium.

Results and discussion

In our initial study, we employed 1-benzylidenethiosemicarbazide (**1a**) as a model substrate in combination with 3 equiv. of $FeCl_3$ to evaluate the efficiency of microwave and ultrasound irradiation (Table 1, entries 1–3). The results showed that a combination of microwave irradiation at 200 W and ultrasound irradiation at 50 W gave the highest yield after 3 min (Table 1, entry 1). Several control

Table 1 Optimizing the conditions



1a **2a**

Entry	$FeCl_3$/equiv.	Method	Time/min	Yield[a]/%
1	3	MW (200 W) + US (50 W)	3	86
2	3	MW (150 W) + US (50 W)	3	77
3	3	MW (200 W) + US (40 W)	3	63
4	3	US (50 W) + oil bath heating	90	47
5	3	MW (200 W)	20	28
6	3	Conventional reflux	240	45
7	2	MW (200 W) + US (50 W)	3	85
8	1.5	MW (200 W) + US (50 W)	3	69
9	3	MW (200 W) + US (50 W)	4	82
10	3	MW (200 W) + US (50 W)	2	56

Reactions were performed under reflux using 1-benzylidenethiosemicarbazide (1.0 mmol), $FeCl_3$, and 6 cm^3 water

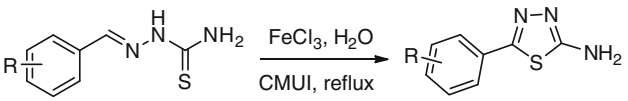
[a] Isolated yield

experiments were carried out in order to demonstrate the superiority of CMUI over other methods (Table 1, entries 1, 4–6). The results clearly show that CMUI achieved the best results in terms of both reaction time and yield. This drastic acceleration effect may be attributed to a combination of enforced heat transfer due to microwave irradiation and intensive mass transfer at phase interfaces caused by sonication. Regarding the concentrations of $FeCl_3$, we found that lower yields were obtained when using less than 3 equiv. of $FeCl_3$ (Table 1, entries 7, 8). As a compromise between eco-efficiency and yield, 2 equiv. of $FeCl_3$ were adopted for further elaboration. The desired 5-phenyl-1,3,4-thiadiazol-2-amine (**2a**) was obtained in 82 % yield after an increased irradiation time of 4 min (Table 1, entry 9), while reducing the irradiation time to 2 min resulted in a decreased yield of 56 % (Table 1, entry 10).

Having identified the optimal conditions (Table 1, entry 7), we next investigated the scope and limitation of the process with variously substituted 1-benzylidenethiosemicarbazides **1** (Table 2). Both electron-withdrawing (Table 2, entries 2–5) and electron-donating (Table 2, entries 6–8) groups were well tolerated, although a slightly lower yield was obtained with a *para*-methyl substituent (Table 2, entry 6).

Considering that Schiff bases with heterocycles express biological activities [36, 37], we next evaluated the scope of the CMUI technology for the synthesis of 5-aryl-*N*-arylidene-1,3,4-thiadiazol-2-amines **4** employing a variety of 5-substituted 1,3,4-thiadiazol-2-amines **2** in combination with 4-hydroxybenzaldehyde (**3**, Table 3). The result showed that the 1,3,4-thiadiazol-2-amines **2** with electron-donating groups

Table 2 Scope and limitations of the protocol



1 **2**

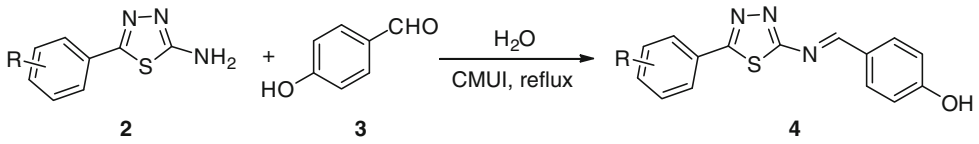
Entry	R	Product **2**	Reported yield/%	Yield[a]/%
1	H	**2a**	65 [26]	85
2	4-F	**2b**	80 [21]	90
3	3-NO_2	**2c**	58 [23]	89
4	4-Cl	**2d**	70 [26]	85
5	2,4-di-Cl	**2e**	84 [22]	84
6	4-CH_3	**2f**	62 [26]	73
7	4-OCH_3	**2g**	60 [26]	88
8	3,4-OCH_2O–	**2h**	Not mentioned [30]	86

Reactions were performed under reflux using **1** (1.0 mmol), $FeCl_3$ (2.0 mmol), and 6 cm^3 water for 3 min under CMUI (microwave: 200 W; ultrasound: 50 W)

[a] Isolated yields

reacted rapidly and gave a high yield of **4** (Table 3, entries 6–8), while those with electron-withdrawing groups required longer reaction times and gave lower yields (Table 3, entries 2–5). Interestingly, when the 1,3,4-thiadiazol-2-amines **2c** and **2e** were used, increased yields of the desired compounds **4c** and **4e** were obtained when 2 mol % of concentrated hydrochloric acid was added as a catalyst.

Table 3 Synthesis of 5-aryl-*N*-arylidene-1,3,4-thiadiazol-2-amines



2 **3** **4**

Entry	R	Product **4**	Time/min	Reported yield/%	Yield[a]/%
1	H	**4a**	3	70 [28]	89
2	4-F	**4b**	8	–	87
3	3-NO_2	**4c**	10	Not mentioned [33]	32 (88[b])
4	4-Cl	**4d**	10	–	77
5	2,4-di-Cl	**4e**	10	–	24 (77[c])
6	4-CH_3	**4f**	30	–	98
7	4-OCH_3	**4g**	3	65 [31]	90
8	3,4-OCH_2O–	**4h**	3	–	92

A mixture of **2** (1.0 mmol), **3** (1.0 mmol), and 6 cm^3 water was brought to reflux upon CMUI (microwave: 200 W; ultrasound: 50 W) for the indicated time (monitored by TLC)

[a] Isolated yields

[b] Added HCl and reacted for 5 min

[c] Added HCl and reacted for 10 min

Encouraged by these results, we tried to explore the CMUI approach for synthesizing other substituted 1,3,4-thiadiazoles. Based on the literature [38–41], we found that 1,3,4-thiadiazoles could be obtained via a one-pot three-component reaction of an aldehyde, a phenylisothiocyanate, and hydrazine hydrate in water under CMUI (Table 4). Therefore, we next investigated the application of our previous protocol applying a variety of aldehydes **5** containing electron-withdrawing (Table 4, entries 2–5) or electron-donating (Table 4, entries 6–13) substituents. To our satisfaction, all reactions worked well and provided the desired products **8a–8m** in good yields.

In conclusion, we have developed an efficient, simple, and green procedure for the synthesis of 5-aryl-*N*-arylidene-1,3,4-thiadiazol-2-amines and 5-aryl-*N*-phenyl-1,3,4-thiadiazol-2-amines employing a $FeCl_3$-promoted process under combined microwave and ultrasound irradiation (CMUI) in water. The CMUI provides extremely efficient dielectric heating along with intensive mass transport in these heterogeneous systems, making it an excellent approach for achieving environmentally friendly and efficient organic synthesis.

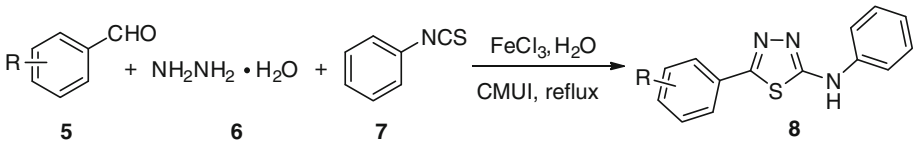
Experimental

All solvents and reagents were purchased from commercial sources and were used without prior purification. All combined microwave and ultrasound irradiation experiments were carried out in a professional TCMC-102 microwave apparatus (Nanjing Lingjiang Technological Development Company, Nanjing, China) operating at a frequency of 2.45 GHz with continuous irradiation power from 0.0 to 500 W, and a FS-250 professional ultrasound apparatus (Shanghai S.X. Ultrasonics, Shanghai, China) operating at a frequency of 20 kHz with controllable irradiation power from 10 to 100 W. The reactions were carried out in a 15 cm^3 two-necked Pyrex flask placed in the microwave cavity with the tip of the detachable horn immersed just under the liquid surface. TLC analysis was performed on aluminum-backed plates SIL G/UV254. The products were purified by filtration and were identified by ^1H NMR (DMSO-d_6, 400 MHz) and MS (EI). All new products were identified by ^1H and ^{13}C NMR (DMSO-d_6, 400 MHz) and high-resolution mass spectra (EI). Melting points were measured with a digital melting-point apparatus (WRR, Shanghai Precision and Scientific Instruments, Shanghai, China).

General experimental procedure for the synthesis of 5-aryl-1,3,4-thiadiazol-2-amines 2

A mixture of 1-arylidenethiosemicarbazide **1** (1 mmol), $FeCl_3$ (2 mmol), and 6 cm^3 water was subjected to microwave–ultrasound activation conditions. The ultrasound and microwave sources were switched on successively (power levels: ultrasound 50 W, microwave 200 W). The mixture was irradiated simultaneously with microwaves and ultrasound for 3 min. The suspension was

Table 4 Synthesis of 5-aryl-*N*-phenyl-1,3,4-thiadiazol-2-amines



Entry	R	Product **8**	Time/min	Reported yield/%	Yield[a]/%
1	H	**8a**	4	85 [40]	79
2	4-CN	**8b**	3	–	75
3	3-NO_2	**8c**	3.5	Not mentioned [42]	76
4	2,4-di-Cl	**8d**	4	72 [43]	74
5	3-F	**8e**	4	–	74
6	3-OCF_3	**8f**	2.5	–	83
7	4-OCH_3	**8g**	3.5	85 [44]	71
8	3,5-di-OCH_3	**8h**	4	–	76
9	3,4,5-tri-OCH_3	**8i**	3.5	78 [30]	75
10	4-CH_3	**8j**	3.5	64 [44]	73
11	4-$CH(CH_3)_2$	**8k**	3.5	–	85
12	4-OH	**8l**	4	87 [45]	85
13	2-OH	**8m**	4	75 [46]	84

A mixture of **5** (1.0 mmol), **6** (1.0 mmol), **7** (1.0 mmol), $FeCl_3$ (2.0 mmol), and 6 cm^3 water was brought to reflux upon CMUI (microwave: 200 W; ultrasound: 50 W) for the indicated time (monitored by TLC)

[a] Isolated yields

filtered and the residue was washed with water and ethanol, after which the residue was recrystallized from ethanol and dried under vacuum to obtain the products **2**.

General experimental procedure for the synthesis of 5-aryl-N-arylidene-1,3,4-thiadiazol-2-amines 4

A mixture of 5-aryl-1,3,4-thiadiazol-2-amines **2** (1 mmol), 4-hydroxy-benzaldehyde **3** (1 mmol), and 6 cm^3 water was subjected to microwave-ultrasound activation conditions. The ultrasound and microwave sources were switched on successively (power level: ultrasound 50 W, microwave 200 W). The mixture was irradiated simultaneously with microwaves and ultrasound for 3 min. The suspension was filtered and the residue was washed with water and ethanol, after which the residue was recrystallized from ethanol and dried under vacuum to obtain the products **4**.

4-[[5-(4-Fluorophenyl)-1,3,4-thiadiazol-2-ylimino]-methyl]phenol (**4b**, $C_{15}H_{10}FN_3OS$)
Yellow solid; m.p.: 261.2–262.0 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.94$ (d, $J = 8.4$ Hz, 2H), 7.41 (t, $J = 8.4$ Hz, 2H), 7.93 (d, $J = 8.8$ Hz, 2H), 8.00–8.03 (m, 2H), 8.88 (s, 1H), 10.63 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 116.7$, 117.2, 126.2, 127.3, 130.2, 133.2, 163.5, 164.5, 165.4, 169.1, 174.8 ppm; HRMS (EI): *m/z* calc'd for $C_{15}H_{10}FN_3OS$ (M + H) 299.0529, found 299.0529.

4-[[5-(4-Chlorophenyl)-1,3,4-thiadiazol-2-ylimino]-methyl]phenol (**4d**, $C_{15}H_{10}ClN_3OS$)
Yellow solid; m.p.: 251.3–252.4 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.94$ (d, $J = 8.4$ Hz, 2H), 7.63 (d, $J = 8.4$ Hz, 2H), 7.93 (d, $J = 8.8$ Hz, 2H), 7.98 (d, $J = 8.4$ Hz, 2H), 8.89 (s, 1H), 10.65 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 116.3$, 126.2, 128.4, 130.0, 133.3, 134.4, 136.3, 163.8, 164.5, 169.2, 175.0 ppm; HRMS (EI): *m/z* calc'd for $C_{15}H_{10}ClN_3OS$ (M + H) 315.0233, found 315.0236.

4-[[5-(2,4-Dichlorophenyl)-1,3,4-thiadiazol-2-ylimino]-methyl]phenol (**4e**, $C_{15}H_9Cl_2N_3OS$)
Yellow solid; m.p.: 255.1–256.6 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.95$ (d, $J = 8.8$ Hz, 2H), 7.65 (m, 1H), 7.92–7.95 (m, 3H), 8.21 (d, $J = 8.4$ Hz, 1H), 8.96 (s, 1H), 10.64 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 116.7$, 126.3, 128.8, 130.7, 132.7, 132.3, 133.3, 135.0, 136.6, 156.0, 163.6, 169.4, 176.3 ppm; HRMS (EI): *m/z* calc'd for $C_{15}H_9Cl_2N_3OS$ (M + H) 348.9842, found 348.9848.

4-[[5-(4-Methylphenyl)-1,3,4-thiadiazol-2-ylimino]-methyl]phenol (**4f**, $C_{16}H_{13}N_3OS$)
Yellow solid; m.p.: 249.7–250.8 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 2.38$ (s, 3H), 6.94 (d, $J = 8.4$ Hz, 2H), 7.37 (d, $J = 8.0$ Hz, 2H), 7.84 (d, $J = 8.0$ Hz, 2H), 7.92 (d, $J = 8.4$ Hz, 2H), 8.87 (s, 1H), 10.62 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 21.8$, 116.7, 126.3, 127.6, 127.9, 130.5, 133.1, 141.8, 163.4, 165.7, 168.9, 174.3 ppm; HRMS (EI): *m/z* calc'd for $C_{16}H_{13}N_3OS$ (M + H) 295.0779, found 295.0760.

4-[[5-(Benzo[d][1,3]dioxol-5-yl)-1,3,4-thiadiazol-2-ylimino]-methyl]phenol (**4h**, $C_{16}H_{11}N_3O_3S$)
Yellow solid; m.p.: 250.2–251.1 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 6.15$ (s, 2H), 6.94 (d, $J = 8.8$ Hz, 2H), 7.09 (d, $J = 8.0$ Hz, 1H), 7.46 (m, 1H), 7.51 (d, $J = 8.2$ Hz, 1H), 7.92 (d, $J = 8.4$ Hz, 2H), 8.85 (s, 1H), 10.60 (s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 102.5$, 107.1, 109.5, 116.7, 123.0, 124.6, 126.3, 133.1, 148.7, 150.3, 163.4, 165.3, 168.7, 174.1 ppm; HRMS (EI): *m/z* calc'd for $C_{16}H_{11}N_3O_3S$ (M + H) 325.0521, found 325.0513.

General experimental procedure for the synthesis of 5-aryl-N-phenyl-1,3,4-thiadiazol-2-amines ***8***
A mixture of aldehyde **5** (1 mmol), hydrazine hydrate **6** (1 mmol), phenylisothiocyanate **7** (1 mmol), $FeCl_3$ (2 mmol), and 6 cm^3 water was subjected to microwave–ultrasound activation conditions. The ultrasound and microwave sources were switched on successively (power levels: ultrasound 50 W, microwave 200 W). The mixture was irradiated simultaneously by microwaves and ultrasound for 3 min. The suspension was filtered and the residue was washed with water and ethanol, after which the residue was recrystallized from ethanol and dried under vacuum to obtain the products **8**.

4-[5-(Phenylamino)-1,3,4-thiadiazol-2-yl]benzonitrile (**8b**, $C_{15}H_{10}N_4S$)
Light yellow solid; m.p.: 242.3– 243.5 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 7.06$ (t, $J = 7.2$ Hz, 1H), 7.39 (t, $J = 7.6$ Hz, 2H), 7.67 (d, $J = 7.9$ Hz, 2H), 7.98 (d, $J = 8.7$ Hz, 2H), 8.05 (d, $J = 8.3$ Hz, 2H), 10.71 (br s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 112.6$, 118.1, 122.8, 127.8, 129.6, 133.6, 140.6, 156.4, 165.5 ppm; HRMS (EI): *m/z* calc'd for $C_{15}H_{10}N_4S$ (M + H) 278.0626, found 278.0623.

5-(3-Fluorophenyl)-N-phenyl-1,3,4-thiadiazol-2-amine (**8e**, $C_{14}H_{10}FN_3S$)
White solid; m.p.: 212.2–213.8 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 7.03$ (t, $J = 7.0$ Hz, 1H), 7.34–7.37 (m, 4H), 7.65 (d, $J = 8.1$ Hz, 2H), 7.93 (t, $J = 6.4$ Hz, 2H), 10.54 (br s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 117.0$, 117.9, 122.6, 127.4, 129.6, 140.8, 156.9, 162.2, 164.5 ppm; HRMS (EI): *m/z* calc'd for $C_{14}H_{10}FN_3S$ (M + H) 271.0579, found 271.0577.

N-Phenyl-5-[3-(trifluoromethoxy)phenyl]-1,3,4-thiadiazol-2-amine (**8f**, $C_{15}H_{10}F_3N_3OS$)
White solid; m.p.: 193.8–195.6 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 7.04$ (t, $J = 7.0$ Hz, 1H), 7.38 (t, $J = 7.6$ Hz, 2H), 7.51 (d, $J = 9.3$ Hz, 2H), 7.66 (d, $J = 8.1$ Hz, 2H), 8.01 (d, $J = 9.3$ Hz, 2H), 10.58 (br s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 118.1$, 122.2, 122.7, 129.2, 129.6, 130.0, 140.8, 149.7, 156.5, 164.9 ppm; HRMS (EI): *m/z* calc'd for $C_{15}H_{10}F_3N_3OS$ (M + H) 337.0497, found 337.0490.

5-(3,5-Dimethoxyphenyl)-N-phenyl-1,3,4-thiadiazol-2-amine (**8h**, $C_{16}H_{15}N_3O_2S$)
White solid; m.p.: 192.3–194.2 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.82$ (s, 6H), 6.64 (s, 1H), 6.98–7.05 (m, 3H), 7.37 (t, $J = 7.0$ Hz, 2H), 7.64 (d, $J = 8.1$ Hz, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 56.0$, 102.6, 105.2, 117.9, 122.6, 129.6, 132.5, 140.8, 157.9, 161.3, 164.5 ppm; HRMS (EI): *m/z* calc'd for $C_{16}H_{15}N_3O_2S$ (M + H) 313.0885, found 313.0880.

5-(4-Isopropylphenyl)-N-phenyl-1,3,4-thiadiazol-2-amine (**8k**, $C_{17}H_{17}N_3S$)
White solid; m.p.: 178.2–180.0 °C; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 1.23$ (d, $J = 6.8$ Hz, 6H), 2.91–2.98 (m, 1H), 7.02 (t, $J = 7.2$ Hz, 1H), 7.37 (t, $J = 8.6$ Hz, 4H), 7.66 (d, $J = 8.0$ Hz, 2H), 7.77 (d, $J = 8.0$ Hz, 2H), 10.50 (br s, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 24.1$, 33.6, 117.9, 122.4, 127.3, 127.6, 128.5, 129.6, 140.8, 151.2, 158.1, 164.1 ppm; HRMS (EI): *m/z* calc'd for $C_{17}H_{17}N_3S$ (M + H) 295.1143, found 295.1140.

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