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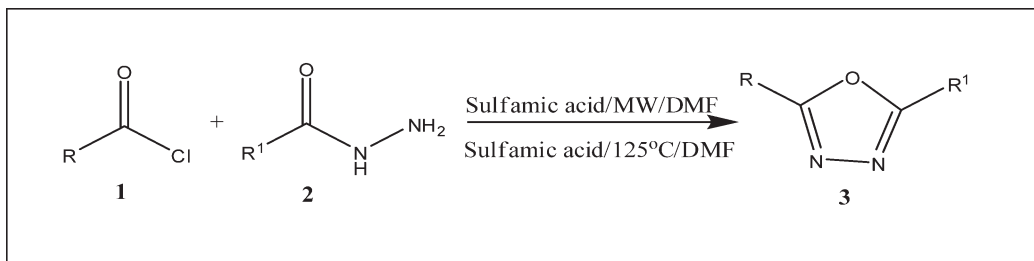
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Sulfamic acid has been found to be an efficient catalyst for the one-pot synthesis of novel 2,5-diaryl-1,3,4-oxadiazoles by condensation of different coumarinoyl hydrazides with various coumarinoyl or quinolinoyl chlorides under microwave irradiation and conventional heating. Some of the advantages of this method are low reaction times, operational simplicity, and high yields.

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INTRODUCTION

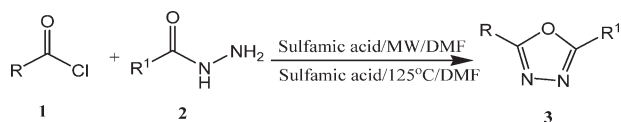
1,3,4-Oxadiazoles and their derivatives have a wide range of biological and therapeutic properties such as antimicrobial [1], antifungal [2], and anti-inflammatory action [3]. Also, vinblastine-like [4] oxadiazole molecules that incorporate a coumarin moiety have been reported as potent antitumor and antibacterial agents [5,6]. Because of these important findings, the synthesis of these kinds of molecules has attracted significant interest. The general synthesis of oxadiazoles can be classified into four main synthetic strategies as follows: (i) cyclization of diacylhydrazines [7–11], (ii) oxidation of acyl hydrazones [12,13], (iii) acid-catalyzed condensation of acyl hydrazides with orthoesters [14–18], and (iv) solid-state synthesis and other methods [19–27]. However, these methods suffer from harsh reaction conditions and long reaction times. Therefore, it was deemed necessary to develop efficient and simple procedures for the synthesis of these biologically important oxadiazole derivatives.

RESULTS AND DISCUSSION

Sulfamic acid (SA) has emerged as an excellent solid acid catalyst for acid-catalyzed reactions such as functional group protection and deprotection [28], and is used in some important organic transformations such as the synthesis of xanthenes [29], Beckmann rearrange-

ment reactions [30], Pechmann [31] and Biginelli [32] condensations. SA is a recyclable and very stable crystalline solid [33], because of its immiscible nature with common organic solvents and its ease in handling. Microwave irradiation accelerated synthesis is emerging as a powerful tool in organic synthesis [34], since this technique affords short reaction times, increased yields, and high purities of reaction products, as well as ease in manipulation. These observations prompted us to investigate an efficient SA-catalyzed one-pot synthesis of novel 2,5-diaryl-1,3,4-oxadiazoles from aryl acid chlorides and aryl hydrazides in dimethylformamide (DMF) utilizing both microwave irradiation and conventional heating (Scheme 1). In the microwave irradiation procedure for the synthesis of 2,5-diaryl-1,3,4-oxadiazoles, the reaction mass containing the aryl acid chloride and the aryl hydrazide was irradiated in a domestic microwave oven at a 300 watt power level over 30 s intervals in an open vessel for 4–5 min. The oxadiazoles were also conventionally prepared at 125°C over 4–5 h. The structures of all newly synthesized 2,5-diaryl-1,3,4-

Scheme 1. Sulfamic acid catalyzed synthesis of 2,5-diaryl-1,3,4-oxadiazoles.



oxadiazoles were confirmed by IR, ¹H NMR and mass spectral data.

To study the efficiency of a number of acidic catalysts compared with SA in the synthesis of the 2,5-diaryl-1,3,4-oxadiazoles, we conducted a model reaction between 3-coumarinoyl chloride and 3-coumarinoyl hydrazide in the presence of either SA, silica sulfuric acid, *p*-toluenesulfonic acid, or sulfuric acid (all at 4 mol %) under both microwave irradiation (Method A) and conventional heating (Method B) conditions (Table 1). In this study it was found that, compared with the other acid catalysts utilized, SA was a more effective catalyst with respect to reaction time and yield of the resulting 1,3,4-oxadiazole (Table 1).

A variety of symmetrical and unsymmetrical 2,5-diaryl-1,3,4-oxadiazoles were also synthesized *via* the above microwave procedure, all of which were obtained in good yields (Table 2), illustrating the versatility of the method.

CONCLUSION

In conclusion, an efficient, facile and flexible one-pot synthesis of 2,5-diaryl-1,3,4-oxadiazoles *via* SA-catalyzed microwave irradiation has been developed. We consider this methodology to be superior to existing methodologies for the synthesis of 2,5-disubstituted-1,3,4-oxadiazoles.

EXPERIMENTAL

All the melting points were determined in open capillary in liquid paraffin bath and are uncorrected. The purity of the compounds was checked by TLC. IR spectra (KBr) were recorded on Shimadzu FTIR model 8010 spectrometer and the ¹H NMR spectra on Varian Gemini 200 MHz Spectrometer using TMS as an internal standard. The C, H, and N analysis of the compounds was done on a Carlo Erba model EA1108. Mass spectra were obtained on a Jeol JMS D-300 Spectrometer. For the microwave irradiation experiments (BPL 800T) domestic microwave oven was used.

General procedure for the synthesis of 2,5-diaryl-1,3,4-oxadiazoles (3a-m) *via* methods A and B.

Method A. The coumarinoyl or quinolinoyl chloride (1.1 mmol), coumarinoyl hydrazide (1.5 mmol), and SA (0.04 mmol) were added to DMF (2 mL). The reaction mass was irradiated in a microwave oven (BPL, 800 model) at a power level of 300 W over 30 s intervals in an open vessel for 4–5 min (Table 2). Completion of the reaction was determined by TLC monitoring. The reaction mass was cooled to room temperature, poured over crushed ice, and stirred for 15 min at 0–5°C. The precipitated product was collected by filtration, washed with cold water and air-dried. Pure product was obtained by recrystallization from ethanol.

Method B. The coumarinoyl or quinolinoyl chloride (1.1 mmol), coumarinoyl hydrazide (1.5 mmol), and SA (0.04 mmol) were added to DMF (3 mL). The reaction mixture was

stirred at 125°C for 4–5 h (Table 2). The progress of the reaction was monitored by TLC. After completion of the reaction, the contents were cooled to room temperature, poured over crushed ice and stirred for 15 min at 0–5°C. The precipitated product was collected by filtration, washed with cold water and air-dried. Pure product was obtained by recrystallization from ethanol.

Spectral data and combustion analyses for 2,5-diaryl-1,3,4-oxadiazoles.

3-(5-(2-Oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-2H-chromen-2-one (3a). IR (KBr, cm⁻¹): 1745 (—C=O), 1610 (—C=N), 1567 (—C=C—), 1122 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 7.30–7.90 (m, 8H), 8.78 (s, 2H); EIMS, 70 E.V, *m/z*: 358 (M⁺); Anal. Calcd. for C₂₀H₁₀N₂O₅: C, 67.04; H, 2.81; N, 7.82; Found: C, 67.08; H, 2.84; N, 7.79.

6-Bromo-3-(5-(6-bromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-2H-chromen-2-one (3b). IR (KBr, cm⁻¹): 1719 (—C=O), 1610 (—C=N), 1568 (—C=C—), 1120 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 7.54 (t, 2H), 7.65–7.80 (m, 4H), 8.76 (s, 2H); EIMS, 70 E.V, *m/z*: 516 (M⁺); Anal. Calcd. for C₂₀H₈Br₂N₂O₅: C, 46.54; H, 1.56; N, 5.43; Found: C, 46.57; H, 1.52; N, 5.40.

6,8-Dibromo-3-(5-(6,8-dibromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-2H-chromen-2-one (3c). IR (KBr, cm⁻¹): 1745 (—C=O), 1620 (—C=N), 1575 (—C=C—), 1135 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 7.55 (d, 2H), 7.70 (d, 2H), 8.78 (s, 2H); EIMS, 70 E.V, *m/z*: 673 (M⁺); Anal. Calcd. for C₂₀H₆Br₄N₂O₅: C, 35.65; H, 0.90; N, 4.16; Found: C, 35.63; H, 0.92; N, 4.19.

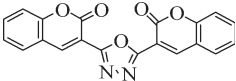
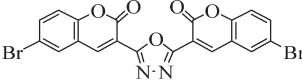
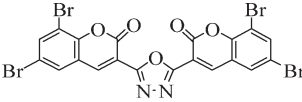
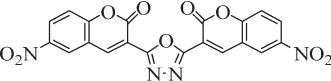
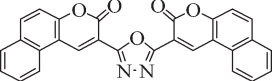
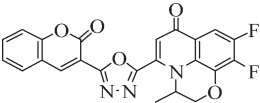
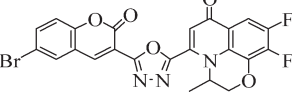
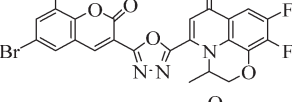
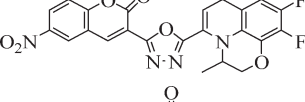
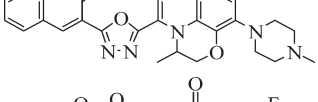
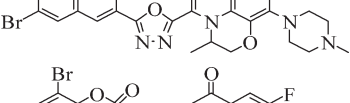
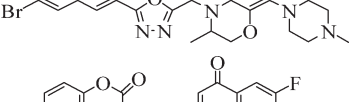
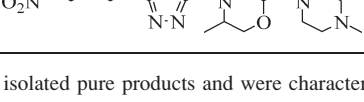
6-Nitro-3-(5-(6-nitro-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-2H-chromen-2-one (3d). IR (KBr, cm⁻¹): 1730 (—C=O), 1618 (—C=N), 1570 (—C=C—), 1130 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 7.60 (m, 4H), 7.70 (d, 2H), 8.75 (s, 2H); EIMS, 70 E.V, *m/z*: 448 (M⁺); Anal. Calcd. for C₂₀H₈N₄O₉: C, 53.58; H, 1.80; N, 12.50; Found: C, 53.57; H, 1.82; N, 12.53.

2-(5-(3-Oxo-3H-benzof[f]chromen-2-yl)-1,3,4-oxadiazol-2-yl)-3H-benzof[f]chromen-3-one (3e). IR (KBr, cm⁻¹): 1720 (—C=O), 1600 (—C=N), 1565 (—C=C—), 1120 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 7.45–8.30 (m, 12H), 8.70 (s, 2H); EIMS, 70 E.V, *m/z*: 458 (M⁺); Anal. Calcd. for C₂₈H₁₄N₂O₅: C, 73.36; H, 3.08; N, 6.11; Found: C, 73.38; H, 3.04; N, 6.13.

9,10-Difluoro-2,3-dihydro-3-methyl-5-(5-(2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3f). IR (KBr, cm⁻¹): 1718 (lactone C=O), 1683 (—C=O), 1615 (C=N), 1565 (—C=C—), 1131 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.59 (d, 3H), 4.35 (d, 1H), 4.50 (d, 1H), 4.76 (d, 1H), 6.90 (d, 2H), 7.51 (d, 2H), 7.81 (t, 1H), 8.72 (s, 1H), 8.90 (s, 1H); EIMS, 70 E.V, *m/z*: 449 (M⁺); Anal. Calcd. for C₂₃H₁₃F₂N₃O₅: C, 61.48; H, 2.92; N, .35; Found: C, 61.46; H, 2.96; N, .37.

5-(5-(6-Bromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-9,10-difluoro-2,3-dihydro-3-methyl-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3g). IR (KBr, cm⁻¹): 1719 (lactone C=O), 1681 (—C=O), 1621 (C=N), 1567 (—C=C—), 1135 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.60 (d, 3H), 4.41 (d, 1H), 4.52 (d, 1H), 4.82 (d, 1H), 6.91 (d, 1H), 7.53 (d, 2H), 7.82 (t, 1H), 8.75 (s, 1H), 8.95 (s, 1H); EIMS, 70 E.V, *m/z*: 528 (M⁺); Anal. Calcd. for C₂₃H₁₂BrF₂N₃O₅: C, 52.29; H, 2.29; N, 7.95; Found: C, 52.32; H, 2.27; N, 7.97.

Table 1
2,5-Diaryl-1,3,4-oxadiazoles (3a–m) synthesized by using sulfamic acid as catalyst.

Entry	Product	Method A		Method B		mp (°C)
		Time (min)	Yield (%) ^a	Time (h)	Yield (%) ^a	
1		5.0	96	4.0	93	180
2		5.0	95	4.5	91	179
3		4.5	96	4.5	91	250
4		5.0	95	5.0	90	140
5		5.0	87	5.0	90	165
6		4.0	96	5.0	93	280
7		4.0	97	5.0	94	255
8		4.5	98	5.0	93	250
9		5.0	96	5.0	94	260
10		4.5	97	5.0	95	220
11		4.0	97	4.0	93	210
12		5.0	98	5.0	95	259
13		5.0	96	5.0	93	190

^a Yields refer to isolated pure products and were characterized by NMR, IR, and mass spectral data.

Table 2

Effect of five acid-catalysts on the reaction of 3-coumarinoyl chloride with 3-coumarinyl hydrazide under conventional and microwave heating.

Catalyst	Method A		Method B	
	Time (min)	Yield (%)	Time (h)	Yield (%)
Sulfamic acid	5	96	4	93
Silicaulfuric acid	5	88	4	82
<i>p</i> -TsOH	5	63	4	56
H ₂ SO ₄	5	32	4	28

5-(5-(6,8-Dibromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-9,10-difluoro-2,3-dihydro-3-methyl-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3h). IR (KBr, cm⁻¹): 1717(lactone C=O), 1683 (C=O), 1623(C=N), 1565 (—C=C—), 1181 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.61 (d, 3H), 4.44 (d, 1H), 4.56 (d, 1H), 4.86 (d, 1H), 6.91 (d, 1H), 7.55 (d, 1H), 7.82 (t, 1H), 8.76 (s, 1H), 8.95 (s, 1H); EIMS, 70 E.V, *m/z*: 607 (M⁺); Anal. Calcd. for C₂₃H₁₁Br₂F₂N₃O₅: C, 45.50; H, 1.83; N, 6.92; Found: C, 45.48; H, 1.86; N, 6.94.

9,10-Difluoro-2,3-dihydro-3-methyl-5-(5-(6-nitro-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3i). IR (KBr, cm⁻¹): 1718 (lactone C=O), 1683 (—C=O), 1632 (—C=N), 1572 (—C=C—), 1115 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.49 (d, 3H), 4.47 (d, 1H), 4.67 (d, 1H), 5.01 (d, 1H), 7.10 (t, 1H), 7.77–7.87 (m, 2H), 7.95 (t, 1H), 8.52 (s, 1H), 8.70 (s, 1H); EIMS, 70 E.V, *m/z*: 494 (M⁺); Anal. Calcd. for C₂₃H₁₂F₂N₄O₇: C, 55.88; H, 2.45; N, 11.33; Found: C, 55.84; H, 2.47; N, 11.35.

9-Fluoro-2,3-dihydro-3-methyl-10-(4-methyl piperazin-1-yl)-5-(5-(2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3j). IR (KBr, cm⁻¹): 1745 (lactone C=O), 1683 (—C=O), 1610 (C=N), 1567 (—C=C—), 1122 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.30 (d, 3H), 2.81 (s, 3H), 3.24 (s, 4H), 3.54 (s, 4H), 4.41 (d, 1H), 4.55 (d, 1H), 4.85 (d, 1H), 7.43 (d, 2H), 7.56 (d, 2H), 7.74 (t, 1H), 8.73 (s, 1H), 8.87 (s, 1H); EIMS, 70 E.V, *m/z*: 529(M⁺); Anal. Calcd. for C₂₈H₂₄FN₅O₅: C, 63.51; H, 4.57; N, 13.23; Found: C, 63.54; H, 4.54; N, 13.27.

5-(5-(6-Bromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-9-fluoro-2,3-dihydro-3-methyl-10-(4-methylpiperazin-1-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3k). IR (KBr, cm⁻¹): 1745 (lactone C=O), 1687 (—C=O), 1615 (C=N), 1580 (—C=C—), 1135 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.32 (d, 3H), 2.72 (s, 3H), 3.35 (s, 4H), 3.58 (s, 4H), 4.43 (d, 1H), 4.58 (d, 1H), 4.90 (d, 1H), 7.54 (d, 2H), 7.56 (d, 1H), 7.77 (t, 1H), 8.70 (s, 1H), 8.87 (s, 1H); EIMS, 70 E.V, *m/z*: 608 (M⁺); Anal. Calcd. for C₂₈H₂₃BrFN₅O₅: C, 55.27; H, 3.81; N, 11.51 Found: C, 55.29; H, 3.84; N, 11.47.

5-(5-(6,8-Dibromo-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-9-fluoro-2,3-dihydro-3-methyl-10-(4-methylpiperazin-1-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3l). IR (KBr, cm⁻¹): 1745 (lactone C=O), 1685 (—C=O), 1620 (C=N), 1580 (—C=C—), 1130 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.35 (d, 3H), 2.65 (s, 3H), 3.36 (s, 4H), 3.55 (s, 4H), 4.58 (d, 1H), 4.65 (d, 1H), 4.90 (d, 1H), 7.56 (d, 1H),

7.59 (d, 1H), 7.77 (t, 1H), 8.75 (s, 1H), 8.90 (s, 1H); EIMS, 70 E.V, *m/z*: 687 (M⁺); Anal. Calcd. for C₂₈H₂₂Br₂FN₅O₅: C, 48.93; H, 3.23; N, 10.19; Found: C, 48.91; H, 3.25; N, 10.20.

9-Fluoro-2,3-dihydro-3-methyl-10-(4-methylpiperazin-1-yl)-5-(5-(6-nitro-2-oxo-2H-chromen-3-yl)-1,3,4-oxadiazol-2-yl)-[1,4]oxazino[2,3,4-ij]quinolin-7-one (3m). IR 1740 (lactone C=O), 1680 (—C=O), 1618 (C=N), 1585 (—C=C—), 1130 (—C—O—C—); ¹H NMR (300 MHz, DMSO-d₆): δ = 1.30 (d, 3H), 2.70 (s, 3H), 3.30 (s, 4H), 3.55 (s, 4H), 4.35 (d, 1H), 4.50 (d, 1H), 4.80 (d, 1H), 7.57 (d, 2H), 7.65 (d, 1H), 7.80 (m, 1H), 8.67 (s, 1H), 8.80 (s, 1H); EIMS, 70 E.V, *m/z*: 574 (M⁺); Anal. Calcd. for C₂₈H₂₃FN₆O₇: C, 58.54; H, 4.04; N, 14.63; Found: C, 58.58; H, 4.07; N, 14.59.

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