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ORIGINAL ARTICLE

Nano-silica sulfuric acid as an efficient catalyst for the synthesis of substituted pyrazoles

Hamideh Emtiazi, Mohammad Ali Amrollahi *, Bi Bi Fatemeh Mirjalili

Department of Chemistry, College of Science, Yazd University, P.O. Box 89195-741, Yazd, Iran

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Abstract A convenient and direct approach has been developed for the preparation of pyrazole derivatives by condensing 1,3-diketones and hydrazines in the presence of nano-silica sulfuric acid. This thermal solvent-free procedure offers some advantages such as short reaction time, simple work-up, high yields, and reusability of the catalyst.

1. Introduction

The synthesis of nitrogen-containing heterocyclic compounds has been a subject of great interest due to their wide application in agrochemical and pharmaceutical fields (Noga et al., 1986; Craig, 1991). Pyrazole derivatives, which belong to this category, fulfill a wide variety of biological functions such as antipyretic (Behr et al., 1967), antibacterial (Mahajan et al., 1991), antipsychotic (Barcelo et al., 2007), antiviral (Larsen et al., 1999), pesticidal (Londershausen, 1996), and insecticidal ones (Windholz, 1976).

Pyrazoles can be synthesized *via* 1,3-dipolar cycloadditions of diazo compounds (Aggarwal et al., 2003), reaction of chalcones and hydrazines (Bhat et al., 2005), a three-component coupling of hydrazine, aldehyde and ethyl acetoacetate (Kumari et al., 2012), reaction of isocyanides, dialkylacetylenedicarboxylates and diacylhydrazines (Adib et al., 2005), and the direct condensation of 1,3-diketones and hydrazines in the presence of an acidic catalyst (Fustero et al., 2008; Katritzky, 1985). The last one is the most straightforward procedure for the synthesis of pyrazole derivatives. This procedure can be carried out in the presence of various catalysts such as H_2SO_4 (Wang and Qin, 2004), polystyrene-supported sulfonic acid (Polshettiwar and Varma, 2008), layered zirconium sulfophenyl phosphonate [a-$Zr(CH_3PO_3)_{1.2}(O_3PC_6H_4SO_3H)_{0.8}$] (Curini et al., 2005), $Sc(OTf)_3$ (Xiong et al., 2009), Y-zeolite (Sreekumar and Padmakumar, 1998), and $Mg(ClO_4)_2$ (Mirjalili et al., 2010).

In recent years, the use of reusable heterogeneous catalysts has received considerable importance in organic synthesis. This is because of their environmental, economic, and industrial benefits. Among these, the application of silica sulfuric acid (SSA) or nano-silica sulfuric acid (nano-SSA), which is a stable and efficient heterogeneous catalyst in organic synthesis, has been widely studied. This catalyst is important from an environmental point of view because it produces little waste. It also has an excellent activity and selectivity even on an industrial scale and, in most cases, can be recovered from reaction mixtures and reused.

* Corresponding author. Tel.: +98 3517256129.
E-mail address: mamrollahi@yazduni.ac.ir (M.A. Amrollahi).

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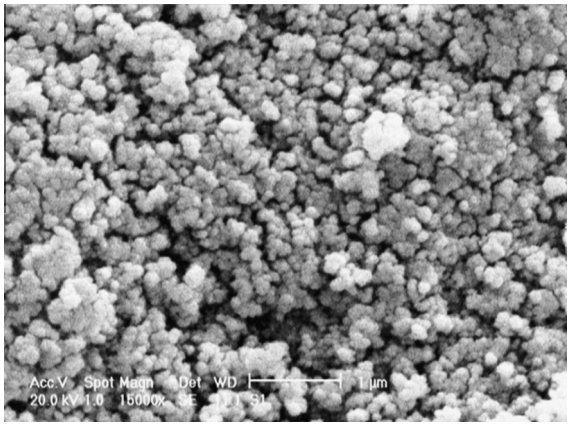


Figure 1 The SEM photograph of nano-SSA.

Silica sulfuric acid has already gained much interest in the synthesis of substituted pyrroles (Veisi, 2010), β-aminoketones (Wu et al., 2007), N-acylsulfonamides (Massah et al., 2008), triarylmethanes (Mohammad poor-Baltork et al., 2011), imidazo pyridines (Polyakov et al., 2009), and deprotection of oximes to carbonyls (Li et al., 2010), Now, we report an efficient and convenient procedure for the synthesis of substituted pyrazoles using silica sulfuric acid or nano-silica sulfuric acid as a catalyst.

2. Materials and methods

The products were characterized by elemental analysis, IR, ^{1}H-NMR, and ^{13}C-NMR spectra. IR spectra were run on a Bruker, Eqinox 55 spectrometer. ^{1}H-NMR and ^{13}C-NMR spectra were obtained using a Bruker Avance 400 and 500 MHz spectrometers (DRX). The elemental analysis was done by a Costech ECS 4010 CHNS-O analyzer. The melting points were measured by a Buchi melting point B-540 apparatus and the SEM of nano-SSA particles was determined with a VEGA/TESCAN scanning electron microscope.

2.1. Synthesis of nano-SSA

A 500 mL suction flask containing nano-silica gel (60.0 g) was equipped with a constant pressure dropping funnel containing chlorosulfonic acid (23.3 g, 0.2 mol) and gas inlet tube for conducting HCl gas over an adsorbing solution i.e., water. Chlorosulfonic acid was added dropwise over a period of 30 min at room temperature. HCl gas was evolved from the reaction vessel immediately. After the addition was complete, the mixture was shaken for 30 min and a white solid of nano-SSA (76.0 g) was collected. Fig. 1 is the SEM photograph of the product. The SSA was prepared according to the literature (Zolfigol, 2001).

2.2. General procedure for the synthesis of pyrazole derivatives

1,3-Diketone (1 mmol), substituted hydrazine (1 mmol), and silica sulfuric acid (0.06 g) or nano-silica sulfuric acid (0.01 g) were placed in a round bottom flask that was heated at 60 °C. The progress of the reaction was followed by TLC. After the completion of the reaction, the product was dissolved to chloroform and filtered to recover the catalyst. The solvent was evaporated, and the crude mixture was solidified from a mixture of ethanol and water. The pure product was obtained by recrystallization in ethanol. The products were characterized on the basis of spectroscopic data. The ^{1}H-NMR spectrum of product consisted one singlet for pyrazole ring C–H (δ = 6.92), a doublet for 2 ortho protons of 5-phenyl ring (δ = 7.30), a multiplet for 6 para and meta protons of phenyl rings (δ = 7.40–7.50), a doublet for 2 ortho protons of 3-phenyl ring (δ = 8.05), two doublets and one singlet for 3 protons of 2,4-dinitrophenyl ring (δ = 8.15, 8.80, 8.95). The ^{13}C-NMR spectrum exhibited 17 sharp signals in agreement with the proposed structure.

2.3. Some selected spectroscopic data

2.3.1. 1-(2,4-Dinitrophenyl)-3,5-diphenyl-1H-pyrazole (Table 2, entry3)

IR: 1607, 1536, 1491, 1459, 1344, 1076, 832, 762, 691. ^{1}H-NMR (500 MHz, $CDCl_3$): 6.92 (s, 1 H); 7.30 (d, 2 H, J = 6.0 Hz); 7.40–7.50 (m, 6 H); 8.05 (d, 2 H, J = 5.7 Hz); 8.15 (d, 1 H, J = 7.2); 8.80 (d, 1 H, J = 6.8 Hz); 8.95 (s, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): 107.21; 121.47; 126.45; 127.42; 129.19; 129.28; 129.35; 129.57; 129.92; 130.55; 132.27; 138.53; 143.32; 145.61; 146.36; 146.42; 155.34. Anal. calcd. for $C_{21}H_{14}N_4O_4$: C 65.28, H 3.65, N 14.50; Found: C 64.98, H 3.55, N 14.40.

2.3.2. 4-Chloro-1-(2,4-dinitrophenyl)-3,5-dimethyl-1H-pyrazole (Table 2, entry 4)

IR: 1607, 1529, 1480, 1344, 1104, 1029, 903, 848, 834, 795. ^{1}H-NMR (500 MHz, $CDCl_3$): 2.30 (s, 3 H); 2.47 (s, 3 H); 8.10 (d, 1 H, J = 8.8 Hz); 8.57 (dd, 1 H, J = 8.8 Hz, J = 2.4 Hz); 8.83 (d, 1 H, J = 2.4 Hz). ^{13}C-NMR (125 MHz, $CDCl_3$): 10.61; 11.87; 112.73; 121.67; 127.98; 129.73; 137.54; 137.97; 142.82; 145.64; 149.92. Anal. calcd. for $C_{11}H_9ClN_4O_4$: C 44.53, H 3.06, N 18.89; Found: C 44.60, H 3.30, N 18.70.

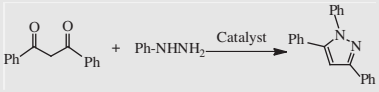
2.3.3. 1-(4-Bromophenyl)-4-chloro-3,5-dimethyl-1H-pyrazole (Table 2, entry 8)

IR: 1588, 1500, 1470, 1401, 1380, 1366, 1099, 1070. 1037, 1008, 831, 810, 795. ^{1}H-NMR (500 MHz, $CDCl_3$): 2.33 (s, 3 H); 2.65 (s, 3 H); 7.54 (d, 2H, J = 8.4 Hz); 7.85 (d, 2H, J = 8.4 Hz). ^{13}C-NMR (125 MHz, $CDCl_3$): 10.83; 11.35; 121.31; 125.58; 125.85; 132.34; 132.39; 138.72; 146.51. Anal. calcd. for $C_{11}H_{10}BrClN_2$: C 46.26, H 3.53, N 9.81; Found: C 46.50, H 3.32, N 10.05.

2.3.4. 3,5-Diphenyl-1-(4-methylphenyl)-1H- pyrazole (Table 2, entry 11)

IR (ATR, neat): 1604, 1545, 1511, 1480, 1361, 972, 822, 760, 691. ^{1}H-NMR (400 MHz, $CDCl_3$): 2.38 (s, 3 H); 6.83 (s, 1 H); 7.16 (d, 2 H, J = 8.4 Hz); 7.30 (m, 6H); 7.44 (t, 2 H, J = 7.6 Hz); 7.93 (dd, 2 H, J = 7.8 Hz, J = 1.2 Hz), 8.10 (d, 2 H, J = 8.0 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): 21.11; 105.22; 125.29; 125.83; 128.05; 128.39; 128.55; 128.79; 128.92; 129.57; 130.69; 133.10; 137.52; 137.73; 142.55; 149.32. Anal. calcd. for $C_{22}H_{18}N_2$: C 85.13, H 5.58, N 9.03; Found: C 85.20, H 5.81, N 8.75.

Table 1 Condensation of 1, 3-diphenyl-1, 3-propanedione with phenylhydrazine under different reaction conditions[a]



Entry	Catalyst (g)	Solvent	Conditions	Time (h)	Yield (%)	Ref.
1	–	Solvent-free	60 °C	2	25	–
2	SSA(0.02)	Solvent-free	r.t	0.5	40	–
3	SSA(0.02)	Solvent-free	60 °C	0.5	68	–
4	SSA(0.04)	Solvent-free	r.t	0.5	45	–
5	SSA(0.04)	Solvent-free	60 °C	0.5	78	–
6	SSA(0.06)	Solvent-free	r.t	0.5	52	–
7	SSA(0.06)	Solvent-free	60 °C	0.5	86	–
8	SSA(0.08)	Solvent-free	r.t	0.5	52	–
9	SSA(0.08)	Solvent-free	60 °C	0.5	88	–
10	Nano-SSA(0.002)	Solvent-free	60 °C	0.5	68	–
11	Nano-SSA(0.006)	Solvent-free	60 °C	0.5	82	–
12	Nano-SSA(0.01)	Solvent-free	60 °C	0.5	93	–
13	Nano-SSA(0.014)	Solvent-free	60 °C	0.5	95	–
14	Nano-SSA(0.01)	Water	60 °C	5	30	–
15	Nano-SSA(0.01)	Ethanol	60 °C	1	80	–
16	Nano-SSA(0.01)	Acetonitrile	60 °C	3	75	–
17	Nano-SSA(0.01)	Ethyl acetate	60 °C	3	70	–
18	Nano-SSA(0.01)	Chloroform	60 °C	3	60	–
19	Nano-SSA(0.01)	Dichloromethane	60 °C	3	55	–
20	Nano-SSA(0.01)	*n*-Hexane	60 °C	5	40	–
21	SSA(0.01), 2nd run	Solvent-free	60 °C	0.5	76	–
22	H_2SO_4 (0.1 drop)	Solvent-free	r.t	1	86	Wang and Qin (2004)
23	Polystyrene supported sulfonic acid (0.1 mL of 20% PSSA solution)	Solvent-free	r.t	0.04	92	Polshettiwar et al. (2008)
24	$[a\text{-}Zr(CH_3PO_3)_{1.2}(O_3PC_6H_4SO_3H)_{0.8}](0.025)$	Solvent-free	40 °C	2	95	Curini et al. (2005)
25	$Sc(OTf)_3$ (2 mol%)	Solvent-free	r.t	0.35	94	Xiong et al. (2009)
26	Y-Zeolite (1)	Ethylene dichloride	r.t 2		84	Sreekumar and Padmakumar (1998)

[a] 1, 3-Diphenyl-1, 3-propanedione (1 mmol) and Phenylhydrazine (1 mmol) were applied.

2.3.5. 3,5-Diphenyl-1-(4-tolosulfono)-1H-pyrazole (Table 2, entry 16)

IR: 1594, 1557, 1484, 1458, 1379, 1191, 1174, 1101, 942, 759, 684, 658. ^{1}H NMR (400 MHz, $CDCl_3$): 2.20 (s, 3 H); 6.81 (s, 1H); 7.40–7.60 (m, 8H); 7.80 (m, 4 H); 8.13 (d, 2 H, J = 7.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): 21.52; 106.46; 127.32; 127.55; 128.34; 128.72; 129.10; 129.43; 129.68; 130.45; 130.59; 132.73; 133.96; 144.51; 146.22; 146.78. Anal. calcd. for $C_{22}H_{18}N_2O_2S$: C 70.57, H 4.85, N 7.48; Found: C 70.25, H 5.10, N 7.70.

2.3.6. 3-Methyl-5-phenyl-1-(4- tolosulfono)-1H-pyrazole (Table 2, entry 17)

IR: 1593, 1563, 1459, 1375, 1294, 1278, 1190, 1122, 1076, 811, 767, 688, 670. ^{1}H-NMR (400 MHz, $CDCl_3$): 2.32 (s, 3 H); 2.40 (s, 3 H); 6.40 (s, 1 H); 7.32 (d, 2 H, J = 8.2 Hz); 7.43 (m, 3 H); 7.81 (d, 2 H, J = 6.8 Hz); 7.90 (d, 2 H, J = 8.2 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$): 10.58; 23.42; 105.43; 126.49; 127.31; 127.95; 128.53; 128.92; 131.31; 132.12; 135.25; 143.58; 149.43. Anal. calcd. for $C_{17}H_{16}N_2O_2S$: C 65.36, H 5.16, N 8.97; Found: C 65.25, H 5.10, N 7.27.

2.3.7. 1,3,5-Triphenyl-4,5-dihydro-1H-pyrazole (Scheme 2)

IR: 1596, 1503, 1393, 1325, 1268, 1124, 874, 758, 745, 692. H NMR (400 MHz, $CDCl_3$): 3.16 (dd, 1H, J = 12.4 and 7.6 Hz), 3.86 (dd, 1H, J = 14.4 and 12.4 Hz), 5.29 (dd, 1H, J = 14.4 and 7.6 Hz), 7.00–7.50 (m, 12H), 7.80 (brs, 2H). ^{13}C-NMR (100 MHz, $CDCl_3$): 115.43; 118.48; 125.14; 126.52; 127.51; 127.97; 128.32; 129.22; 129.63; 130.50; 130.91; 131.65; 135.32; 136.83; 140.72.

2.3.8. Ethyl 3-(2-(2,4-dinitrophenyl)hydrazono) butanoate

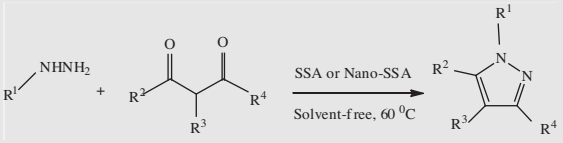
IR: 1726, 1614, 1506, 1420, 1342, 1314, 1104, 834. ^{1}H-NMR (500 MHz, $CDCl_3$): 1.35 (t, J = 7.1 Hz, 3H); 2.21 (s, 3H); 3.52(s, 2H); 4.27 (q, 2H, J = 7.1 Hz); 8.01 (d, 1H, J = 9.5 Hz); 8.36 (dd, 1H, J = 9.5 and 2.5 Hz); 9.17 (d, 1H, J = 2.5 Hz); 11.13 (s, NH). ^{13}C-NMR (125 MHz, $CDCl_3$): 14.62; 16.65; 44.97; 61.93; 116.91; 123.82; 130.57; 132.54; 138.67; 145.45; 151.12; 169.67.

3. Results and discussion

In order to optimize the reaction conditions, we studied the condensation of 1,3-diphenyl-1, 3-propanedione (1 mmol) with phenylhydrazine (1 mmol) in various reaction conditions. The results are summarized in Table 1.

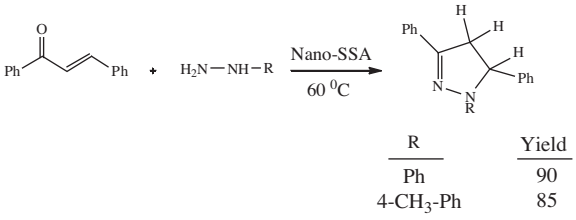
Initially, the reaction was performed with different amounts of SSA in various conditions. It was found that 0.06 g of SSA as a catalyst would be sufficient and an excessive amount of that would not increase the yield remarkably (Table 1, entry

Table 2 The condensation of 1, 3-diketones and hydrazines in the presence of silica sulfuric acid or nano-silica sulfuric acid[a]



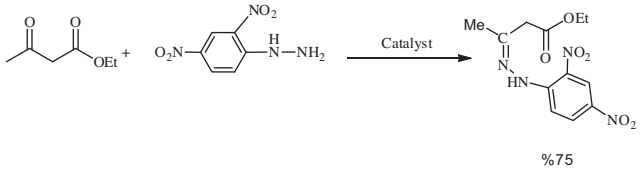
Entry	R^1	R^2	R^3	R^4	Yield (%) Nano-SSA/SSA	Time (min)	Mp (°C) [m.p. reported]	Ref.
1	2,4- O_2N-C_6H_3	CH_3	H	CH_3	85/79	15	122–124	Xiong et al. (2009)
2	2,4- O_2N-C_6H_3	C_6H_5	H	CH_3	92/93	20	127–129	Curini et al. (2005)
3	2,4- O_2N-C_6H_3	C_6H_5	H	C_6H_5	92/90	20	149–150	Mirjalili et al. (2010)
4	2,4- O_2N-C_6H_3	CH_3	Cl	CH_3	89/80	25	166–168	Mirjalili et al. (2010)
5	C_6H_5	C_6H_5	H	C_6H_5	93/86	30	137–138	Sreekumar and Padmakumar (1998)
6	C_6H_5	CH_3	Cl	CH_3	93/86	20	Oil	Polshettiwar et al. (2008)
7	H	C_6H_5	H	CH_3	87/87	25	202–204	Mirjalili et al. (2010)
8	4- Br-C_6H_4	CH_3	Cl	CH_3	94/90	15	87–88	Mirjalili et al. (2010)
9	4- Br-C_6H_4	C_6H_5	H	C_6H_5	90/85	40	119–120	Mirjalili et al. (2010)
10	4- Br-C_6H_4	C_6H_5	H	CH_3	92/88	25	178–180	Mirjalili et al. (2010)
11	4- Me-C_6H_4	C_6H_5	H	C_6H_5	87/94	40	104–105	Mirjalili et al. (2010)
12	4- Me-C_6H_4	C_6H_5	H	CH_3	89/87	15	82–84	Mirjalili et al. (2010)
13	4-OMe-C_6H_4	C_6H_5	H	C_6H_5	74/78	30	oil	Mirjalili et al. (2010)
14	4-OMe-C_6H_4	C_6H_5	H	CH_3	88/88	20	oil	Mirjalili et al. (2010)
15	4- Me-$C_6H_4SO_2$	CH_3	H	CH_3	83/80	20	94–95	Xiong et al. (2009)
16	4- Me-$C_6H_4SO_2$	C_6H_5	H	C_6H_5	83/85	25	101–103	Mirjalili et al. (2010)
17	4- Me-$C_6H_4SO_2$	C_6H_5	H	CH_3	84/85	35	86–87	Mirjalili et al. (2010)

[a] Phenylhydrazine (1 mmol), 1, 3-Diphenyl-1, 3-propanedione (1 mmol) silica sulfuric acid (0.06 g) or nano-silica sulfuric acid (0.01 g).



R	Yield
Ph	90
4-CH_3-Ph	85

Scheme 1



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Scheme 2

7). We repeated the above mentioned reaction with various amounts of nano-SSA with the finding that the activity of nano-SSA is six times more, and only 0.01 g of it would be sufficient (Table 1, entry 12). Among the various reaction conditions (Table 1, entries 1–20), the most effective condition in terms of reaction yield and rate was found to be created by SSA (0.06 g) and nano-SSA (0.01 g) in solvent- free media at 60 °C. The nano-SSA is a more effective activation agent because of the vide surface of it and can accelerate the overall reaction rate by a little amount of it.

To examine the reusability of nano-SSA under a solvent-free condition, after each run, the product was dissolved to $CHCl_3$ and filtered. The catalyst residue was washed with acetone and reused. As a matter of fact, treatment with acetone removes the tar from the catalyst surface more efficiently (Table 1, entry 21). The catalyst was reusable although a gradual decline was observed in its activity.

The general efficiency of this protocol was then studied for the synthesis of a variety of pyrazoles (Table 2). As it can be seen in Table 2, various hydrazines reacted efficiently with 1,3-diketones to afford the desired pyrazoles in good yields. Investigation was made of a series of aromatic hydrazines bearing either electron-donating or electron-withdrawing groups on the aromatic ring. The substitution group on the phenyl ring seemed to affect the reaction significantly neither in the product yield nor in the reaction rate.

The reactions of chalkon with hydrazines in the presence of nano-silica sulfuric acid were examined and the corresponding products were produced in high yields (Scheme 1).

Ethylacetoacetate was utilized as substrate and the pyrazole ring was not formed (Scheme 2).

4. Conclusions

This paper reports the development of an efficient procedure for the synthesis of substituted pyrazoles using nano-silica sulfuric acid as a reusable, eco-friendly and efficient heterogeneous catalyst. The major advantages of this procedure include easy work-up, high yields, clean reactions, and low catalyst loading.

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