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Zeolite and sulfated zirconia as catalysts for the synthesis of 5-substituted 1H-tetrazoles via [2+3] cycloaddition of nitriles and sodium azide

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ABSTRACT

The [2+3] cycloaddition between various nitriles and sodium azide proceeds smoothly in the presence of zeolite and sulfated zirconia as effective catalysts, in water and DMF/MeOH, to give the corresponding 5-substituted 1H-tetrazoles in good to high yields. The reaction most probably proceeds through the in situ formation of catalyst azide species, followed by a successive [2+3] cycloaddition with the nitriles. This method has the advantages of high yields, simple methodology and easy work-up. The catalyst can be recovered by simple filtration and reused with good yields.

1. Introduction

Tetrazoles are a class of heterocyclic compounds that contain nitrogen and are currently under intensive focus due to their wide range of applications [1], mainly as a result of the roles played by tetrazoles in coordination chemistry, material sciences, pharmaceuticals, explosives, and photography [1–7]. Among them, 5-substituted tetrazoles have received much attention recently, and new preparative methods have appeared [8]. It is desirable to develop a more efficient and convenient method for the synthesis of 5-substituted tetrazoles. The acid-catalyzed cycloaddition between hydrazoic acid and nitriles has long been one of the main routes to 5-substituted tetrazoles. However, this standard procedure needs the direct addition of large excess amounts of dangerous and harmful hydrazoic acid [9]. The conventional method of synthesizing tetrazoles is by addition of azide ions to organic nitriles or cyanamides [10]. Earlier reported methods for the synthesis of 5-substituted tetrazoles suffer from drawbacks such as the use of strong Lewis acids, or expensive and toxic metals, and the in situ-generated hydrazoic acid, which is highly toxic and explosive [11,12]. Several syntheses of 5-substituted tetrazoles have been reported through the [2+3] cycloaddition of nitriles using azides in the presence of catalysts [13]. The development of a catalytic synthetic method for tetrazoles still remains an active research area. An important objective of chemistry is to adapt classical processes so that pollution effects are kept to a minimum, with both reduction in energy and consumption of raw materials. Solid acid catalysts play a prominent role in organic synthesis under heterogeneous conditions.

Nowadays, more and more heterogeneous Bronsted acids, e.g., zeolites, are preferred from an economical perspective as well as from an ecological viewpoint. Due to its high protonic acidity and unique shape-selective behavior, HZSM-5 has been shown to be a highly active and stable catalyst for reactions [14]. Zirconia is attracting considerable interest on account of its potential use as a catalyst support. Among various solid acid catalysts investigated in recent times, sulfate-ion promoted zirconia has received much attention due to its strong acidity, high thermal stability, large specific surface area and ability to perform organic reactions at much lower temperatures. Recent investigations reveal that promoted zirconia is an exceptionally good solid acid catalyst for various organic synthesis and transformation reactions having enormous industrial applications [15].

In continuation of our interest in the development of efficient and environmentally friendly methods for the catalytic synthesis of heterocycles, and the application of heterogeneous reagents for the development of useful synthetic methodologies [16], we report herein the synthesis of 5-substituted 1H-tetrazoles by zeolite and sulfated zirconia catalyzed [3+2] cycloaddition between a wide variety of nitriles and sodium azide in water and DMF/MeOH (Scheme 1).

2. Experimental

All reagents were purchased from Merck and Aldrich and were used without further purification. Products were characterized by

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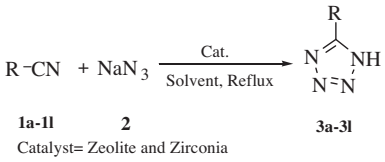


Scheme 1. Zeolite and sulfated zirconia catalyzed synthesis of 5-substituted 1-*H*-tetrazoles.

spectroscopic data (FTIR, ^{1}H and ^{13}C NMR spectra), elemental analysis (CHN) and melting points. A JASCO FT/IR-680 PLUS spectrometer was used to record IR spectra using KBr pellets. NMR spectra were recorded on a Bruker 400 Ultrasheild NMR and DMSO-d_6 was used as the solvent. The reported melting points were determined by the open capillary method using a Galen Kamp melting point apparatus and are uncorrected. Mass spectra were recorded on a Shimadzu Gas Chromatograph Mass Spectrometer GCMS-QP5050A/QP5000 apparatus.

2.1. Catalyst preparation

2.1.1. Synthesis of ZSM-5 and HZSM-5

For the synthesis of ZSM-5, hydrated aluminum sulfate and sodium silicate solution were the sources of aluminum and silicon, respectively. Tetrapropylammonium bromide was used as the structure-directing template [17]. ZSM-5 zeolite was synthesized according to the procedure described earlier [13]. The solid phase obtained was filtered, washed with distilled water several times, dried at 120 °C for 12 h and then calcined at 550 °C for 6 h. The Na^+ ions in Na-ZSM-5 were exchanged with NH_4^+ ions using NH_4NO_3 solution. The catalyst was separated, washed with bidistilled water, until all the Cl^- ions were removed, and finally dried at 110 °C overnight. The hydrogen form of the ZSM-5 zeolites was obtained by calcining NH_4-ZSM-5 in air at 530 °C for 4 h [17].

2.1.2. Synthesis of sulfated zirconia

Amorphous hydrated zirconia was synthesized by hydrolysis of $ZrCl_4$ with a concentrated (25%) solution of ammonia, according to the procedure described earlier [18]. The obtained hydrous zirconia sample was dried at 120 °C for 12 h. Sulfated zirconia (SZ) was prepared by suspending ZrO_2 in a solution of 0.5 M H_2SO_4. After 90 min stirring the mixture was filtered and washed with 0.05 M H_2SO_4. The precipitate was dried at 120 °C and calcined for 2 h at 600 °C with subsequent cooling in either a desiccator or under ambient conditions [18].

2.2. General procedure for the synthesis of the tetrazoles

The procedure for the synthesis of the tetrazole **3a** (Scheme 1) is representative. In a round bottom flask benzonitrile (0.2 g, 2 mmol), sodium azide (0.4 g, 6 mmol), catalyst (50 mg) and DMF or water (20 ml) were charged. Then the reaction mixture was stirred in distilled dimethylformamide (DMF) at 110–120 °C, DMF/MeOH at 80 °C or refluxed in water for 24 h. The progress of the reaction (after 2, 4, 6, 12, 18 and 24 h) was followed by HPLC and TLC (75:25 ethyl acetate:*n*-hexane).

After completion of the reaction, the catalyst was separated by centrifugation, washed with doubly distilled water and acetone, and the centrifugate was treated with 6 M HCl (20 mL) while being stirred vigorously. The aqueous solution finally obtained was extracted twice with ethyl acetate. The combined organic phase was washed with water and concentrated to precipitate the crude crystalline solid.

2.2.1. Spectroscopic data

*2.2.1.1. 5-Phenyl-1H-phenyltetrazole (**3a**, Table 2, entry 1).* White powder; M.p.: 212–214 °C. FTIR (KBr, cm^{-1}): 3054, 2981, 2914, 2837, 2794, 2701, 2610, 1608, 726. ^{1}H NMR (DMSO-d_6, 400 MHz) δ ppm: 7.6–8.1 (m, 5H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ ppm: 124.5, 127.4, 129.9, 131.7, 155.8. MS; m/z = 146, 118, 103, 91, 77, 63, 39.

*2.2.1.2. 5-(Thiophen-2yl)-1H-tetrazole (**3b**, Table 2, entry 2).* White solid; M.p.: 201–203 °C. FTIR (KBr, cm^{-1}): 3108, 3093, 3076, 2952, 2890, 2789, 2685, 1505, 1434, 964. ^{1}H NMR (DMSO-d_6, 400 MHz) δ ppm: 7.1 (t, J = 4 Hz, 1H), 7.6 (d, J = 4 Hz, 1H), 7.7 (d, J = 4 Hz, 1H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ ppm: 149, 134, 130, 125. MS; m/z = 154, 152, 124, 109, 97, 69, 45.

*2.2.1.3. 2-(1-H-Tetrazole-5-yl) pyridine (**3c**, Table 2, entry 3).* White solid; M.p.: 208–210 °C. FTIR (KBr, cm^{-1}): 3278, 3181, 2929, 1662, 1578, 1390, 923. ^{1}H NMR (DMSO-d_6, 400 MHz) δ ppm: 7.4 (t, J = 6.4 Hz, 1H), 7.8 (t, J = 6.4 Hz, 1H), 8.0 (t, J = 8.0 Hz, 1H), 8.5 (d, J = 3.2 Hz, 1H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ ppm: 167, 158, 149, 137, 124, 121. MS; m/z = 147, 119, 105, 91, 78, 51.

*2.2.1.4. 3-(1-H-Tetrazole-5-yl) pyridine (**3d**, Table 2, entry 4).* White solid; M.p.: 238–240 °C. FTIR (KBr, cm^{-1}): 2950, 2890, 2850, 2761, 1480, 1200. ^{1}H NMR (DMSO-d_6, 400 MHz) δ ppm: 9.1 (s, 1H), 8.8 (d, J = 3.8 Hz, 1H), 8.3 (d, J = 3.8 Hz, 1H), 7.6 (1H, m). ^{13}C NMR (DMSO-d_6, 100 MHz) δ ppm: 165, 153, 150, 136, 126, 123. MS; m/z = 147, 119, 92, 78, 50.

Table 1
Effect of catalysts and solvents on the formation of tetrazole.[a]

Entry	Solvent	Benzonitrile:sodium azide	Zeolite	% Conversion [b]	Sulfated zirconia	% Conversion
1	H_2O	1:3				
2	DMF	1:3				
3	DMF/MeOH (9/1)	1:3				
4	H_2O	1:1	50	49	50	62
5	DMF	1:1	50	60	50	67
6	DMF/MeOH (9/1)	1:1	50	70	50	79
7	H_2O	1:3	50	90	50	94
8	DMF	1:3	50	94	50	92
9	DMF/MeOH (9/1)	1:3	50	96	50	98
10	H_2O	1:3	100	78	100	80
11	DMF	1:3	100	83	100	88
12	DMF/MeOH (9/1)	1:3	100	90	100	92
13	H_2O	1:1	20	30	20	35
14	DMF	1:1	20	21	20	25
15	DMF/MeOH (9/1)	1:1	20	39	20	43

[a] Reaction time: 24 h.
[b] Conversion followed by HPLC.

Table 2
Preparation of 5-substituted 1H-tetrazoles in the presence of catalyst by the reaction between sodium azide and nitriles.[a,b]

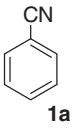
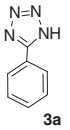
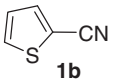
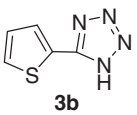
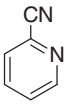
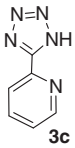
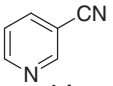
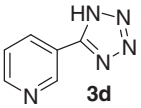
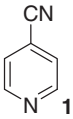
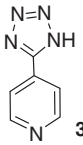
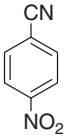
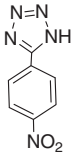
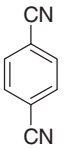
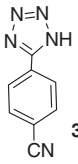
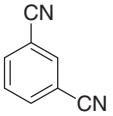
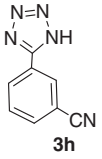
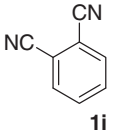
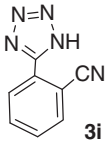
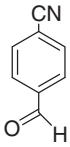
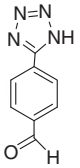
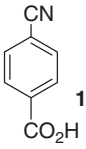
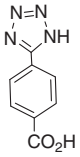
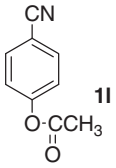
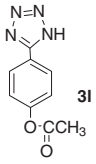
Entry	Substrate	Product	Yields[c]				Ref.
			Zeolite		Sulfated zirconia		
			H_2O	DMF	H_2O	DMF	
1	 **1a**	 **3a**	80	90	82	90	[19,21–28]
2	 **1b**	 **3b**	88	96	80	90	[16b]
3	 **1c**	 **3c**	72	86	74	91	[16b]
4	 **1d**	 **3d**	88	98	74	96	[21,23,26]
5	 **1e**	 **3e**	90	95	80	90	[16b]
6	 **1f**	 **3f**	76	95	91	98	[20–26]
7	 **1g**	 **3g**	88	90	82	95	[21,23,27]
8	 **1h**	 **3h**	83	95	53	95	[16b]
9	 **1i**	 **3i**	43	91	53	95	[16b]
10	 **1j**	 **3j**	55	96	53	56	[24]
11	 **1k**	 **3k**	45	64	57	64	[16b]
12			37	81	30	57	[19]

Table 2 (*continued*)

Entry	Substrate	Product	Yields[c]				Ref.
			Zeolite		Sulfated zirconia		
			H_2O	DMF	H_2O	DMF	
	 **1l**	 **3l**					

[a] The products were characterized by IR, ^{1}H NMR, ^{13}C NMR and mass spectroscopy.
[b] Reaction time: 24 h.
[c] Isolated yields after recrystallization.

*2.2.1.5. 4-(1-H-Tetrazole-5-yl) pyridine (**3e**, Table 2, entry 5).* White solid; M.p.: 254–258 °C. FTIR (KBr, cm^{-1}): 3080, 3060, 3028, 2955, 2917, 2832, 2751, 2689, 1608, 1581, 1492, 1065, 784. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 8.0 (d, J = 7.8 Hz, 2H), 8.8 (d, J = 7.8 Hz, 2H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 165, 149, 134, 121. MS; m/z = 147, 119, 92, 78, 62, 50.

*2.2.1.6. 5-(4-Nitrophenyl)-1H-tetrazole (**3f**, Table 2, entry 6).* White solid; M.p.: 218–220 °C. FTIR (KBr, cm^{-1}): 3103, 2914, 2853, 2752, 2621, 1605, 1526, 1487, 861. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 8.1 (d, J = 8 Hz, 2H), 8.2 (d, J = 8 Hz, 2H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 156, 130, 128, 124. MS; m/z = 191, 163, 149, 134, 90, 63.

*2.2.1.7. 4-(1H-Tetrazole-5-yl)benzonitrile (**3g**, Table 2, entry 7).* White solid; M.p.: 258–260 °C. FTIR (KBr, cm^{-1}): 3100, 2848, 2750, 2250, 1480, 781. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 8.0 (d, J = 7.8 Hz, 2H), 8.2 (d, J = 7.8 Hz, 2H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 160, 135, 132, 130, 126, 114. MS; m/z = 171, 143, 129, 103, 62.

*2.2.1.8. 3-(1H-Tetrazole-5-yl)benzonitrile (**3h**, Table 2, entry 8).* White solid; M.p.: 214–216 °C. FTIR (KBr, cm^{-1}): 3113, 2981, 2780, 2442, 2237, 1476, 870, 780. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 7.7–8.1 (5H, m). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 164, 134, 133, 132, 131, 129, 115. MS; m/z = 171, 143, 102, 62.

*2.2.1.9. 2-(1H-Tetrazole-5-yl)benzonitrile (**3i**, Table 2, entry 9).* White solid; M.p.: 208–210 °C. FTIR (KBr, cm^{-1}): 3096, 2531, 2110, 2023, 1632, 1436, 845. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 7.6 (t, J = 6.8 Hz, 1H), 7.7 (t, J = 6.8 Hz, 1H), 7.8 (m, 2H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 166, 140, 136, 133, 132, 128, 126, 118. MS; m/z = 171, 143, 129, 115, 88, 76, 62, 57.

*2.2.1.10. 4-(1H-Tetrazole-5-yl)benzaldehyde (**3j**, Table 2, entry 10).* White solid; M.p.: 180–182 °C. FTIR (KBr, cm^{-1}): 3015, 2924, 2854, 2713, 2612, 1667, 1440, 776. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 7.9 (d, J = 7.2 Hz, 2H), 8.0 (d, J = 7.2 Hz, 2H), 9.1 (s, 1H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 188, 156, 138, 131, 129, 128. MS; m/z = 174, 146, 130, 116, 102, 90, 57, 43.

*2.2.1.11. 4-(1H-Tetrazole-5-yl) benzoic acid (**3k**, Table 2, entry 11).* White solid; M.p.: 248–250 °C. FTIR (KBr, cm^{-1}): 3600–3000 (br), 2500, 1760, 1500, 1480, 780. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 7–8 (m, 4H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 188, 166, 138, 131, 129, 128. MS; m/z = 190, 174, 146, 130, 116, 102, 90, 75, 57.

*2.2.1.12. 4-(1H-Tetrazole-5-yl)phenyl acetate (**3l**, Table 2, entry 12).* White solid; M.p.: 212–214 °C. FTIR (KBr, cm^{-1}): 3097, 2925, 2865, 2700, 2625, 1678, 1580, 1269, 843. ^{1}H NMR (DMSO-d$_6$, 400 MHz) δ ppm: 7.9 (d, J = 7.4 Hz, 2H), 7.7 (d, J = 7.4 Hz, 2H), 2.59 (s, 3H). ^{13}C NMR (DMSO-d$_6$, 100 MHz) δ ppm: 170, 164, 152, 131, 128, 124, 22. MS; m/z = 204, 189, 173, 160, 145, 130, 102, 90.

3. Results and discussion

In continuation of our recent work on the applications of heterogeneous reagents for the development of synthetic methodologies, we report a new protocol for the preparation of 5-substituted 1H-tetrazoles from a wide variety of nitriles using zeolite and sulfated zirconia as a solid acid catalyst (Scheme 1).

In the reaction between benzonitrile **1a–l** and sodium azide **2**, we investigated the effect of the benzonitrile:sodium azide ratio and catalyst (Table 1). Attempts to carry out these reactions in the absence of catalyst in water, DMF and MeOH did not yield any products, indicating that the sulfated zirconia and zeolite are acting as a catalyst (Table 1, entries 1 and 2). First, we optimized the amount of catalyst required in the reaction between benzonitrile and sodium azide. Different amounts of zeolite as the catalyst (20, 50 and 100 mg) were tried and it was found that 50 mg of catalyst gave the maximum yield of the products. An increase in the amount of catalyst did not improve the results to any great extent. However on using 20 mg of catalyst the reaction was complete after 76 h. In addition the amount of sulfated zirconia was optimized using different amounts (10, 20 and 50 mg) of this catalyst. It was observed that 20 mg of catalyst gave the best results. Again an increase in the value of catalyst did not improve the yield, and with values lower than 20 mg the reaction was only completed after long reaction times (72 h).

One of the most important advantages of heterogeneous catalysis over the homogeneous counterpart is the possibility of reusing the catalyst by simple filtration, without loss of activity. In a typical experiment, after the reaction was completed, the catalysts were recovered from the reaction mixtures by simple filtration and they were purified by washing the solid residues with deionized water and acetone followed by drying in an oven at 100 °C for 40 min. From each experiment, more than 98% of the catalyst was recovered. The recovered catalyst was reused three times without any loss of activity (Table 1, entries 16–18) (see Fig. 1).

Next we examined the effect of solvent. In the present study we used DMF–methanol and water as the solvent. Not many organic solvents are stable at the high temperatures necessary for cycloaddition reactions (sometimes as high as 130 °C), and for this reason DMF is most commonly used for this purpose [3,21,27]. However the use of water as a clean, inexpensive, and universal solvent combines features of both economic and environmental advantages. As can be seen from Table 1, the best results were obtained from a DMF–MeOH mixture as the solvent. Under these conditions and using the molar ratio 1:3 benzonitrile:sodium azide, the conversion

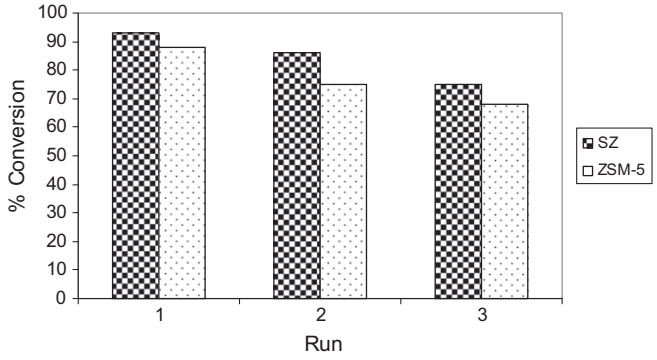


Fig. 1. The results obtained from catalyst reuse, zeolite (black bars) and sulfated zirconia (white bars) in the tetrazole formation.

was found to be 96% and 98% using zeolite and sulfated zirconia as the catalyst, respectively. These results are comparable with the application of other heterogeneous and homogeneous catalysts [28–32]. In addition it was found that the best results were obtained by using a 1:3 benzonitrile:sodium azide ratio (Table 1 entries 7–12).

Several substituted nitriles reacted with sodium azide to give the corresponding tetrazoles in good yields. The nature of the substituents on the nitriles had a significant effect on the tetrazole formation reaction (Table 2). Reactions of electron poor aromatic and heteroaromatic nitriles, such as 2-, 3-, 4-cyanopyridines and 4-nitrobenzonitrile were completed within a few hours (Table 2, entries 3–6). Some electron rich nitriles required higher temperatures, therefore it seems that the more electron-poor a nitrile, the faster it reacts. Interestingly, the best percentage conversions were observed for nitriles with electron withdrawing substituents. However nitriles with electron donating groups (e.g. OH and NH_2) were the least reactive ones. With acetylation of 4-hydroxy benazonitrile a better yield was obtained, but for 4-amino benzonitrile and 2-amino benzonitrile even on heating for 72 h no product was formed.

4. Conclusion

In conclusion, we have developed a simple, ecofriendly and efficient method for the preparation of 5-substituted 1H-tetrazoles using zeolite and sulfated zirconia as effective and reusable catalysts. Various nitriles reacted with NaN_3 in water and DMF/MeOH to yield the corresponding 5-substituted 1H-tetrazoles. The significant advantages of this methodology are high yields, simple methodology, easy work-up and elimination of dangerous and harmful hydrazoic acid, a simple work-up procedure and easy preparation and handling of the catalyst. The catalyst can be recovered by filtration and reused. This methodology may find widespread use in organic synthesis for the preparation of 5-substituted 1H-tetrazoles.

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