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Highly efficient synthesis of benzopyranopyridines via ZrP_2O_7 nanoparticles catalyzed multicomponent reactions of salicylaldehydes with malononitrile and thiols

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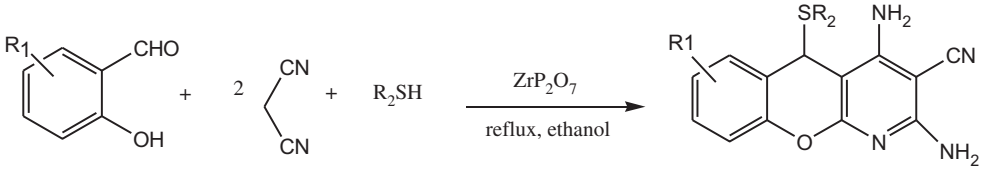
ZrP_2O_7 nanoparticles as an efficient catalyst have been used for the preparation of benzopyrano[2,3-b]pyridines from the four-component condensation reaction of salicylalde-hydes, thiols, and 2 equiv. of malononitrile under reflux conditions in ethanol in excellent yields and short reaction times.



Keywords: ZrP_2O_7 nanoparticles; salicylaldehydes; benzopyrano[2,3-b]pyridine; one-pot; heterocycles

1. Introduction

Benzopyranopyridines are fused heterocyclic compounds that exhibit anti-bacterial,[1,2] anti-proliferative,[3] anti-myopic,[4] anti-rheumatic,[5] anti-asthmatic,[6] and cancer chemopreventive [7] activities. Several compounds derived from libraries were identified as inhibitors of mitogen-activated protein kinase-activated protein kinase 2 and attenuated the production of proinflammatory TNFα,[8] and histamine-stimulated gastric acid secretion in animals.[9] Some other examples of benzopyranopyridine derivatives such as amlexanox and pranoprofen have been reported as anti-allergic and potent NSAIDs, respectively. Therefore, the development of simple methods for the synthesis of benzopyranopyridines is of major interest to modern synthetic organic chemists. In recent years, multicomponent reactions have been extensively utilized to produce heterocyclic compounds with biological activity. Multicomponent reactions are highly flexible, convergent, fruitful and atom-efficient processes of high exploratory power that minimize solvent consumption and maximize atom efficiency.[10–17] Multicomponent reactions are usually designed for the development of environmentally benign synthetic methods. Hence, they are useful from a green chemistry viewpoint.[18,19] A few catalysts such as K_2CO_3,[20] Et_3N

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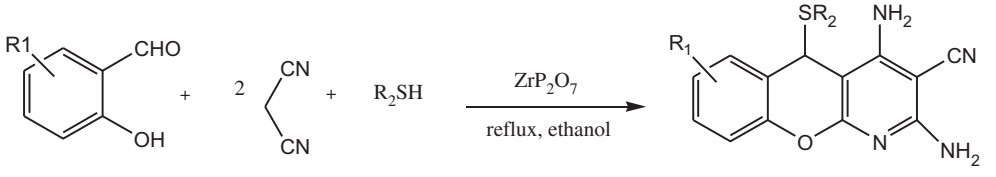
[21] and chitosan [22] have been used for the synthesis of benzopyranopyridine derivatives, but all these methods require a long duration and high temperature. Heterogeneous catalysts have emerged as economically viable due to their unique catalytic properties [23–25] and nanoparticles (NPs) as heterogeneous catalysts have received considerable attention in green chemistry. Metal pyrophosphates are heavily studied materials due to their utility in a variety of applications.[26,27] For example, zirconium pyrophosphate (ZrP_2O_7) NPs were used as an excellent catalyst in many organic reactions. A simple separation and purification process in organic reactions is an advantage of heterogeneous catalytic systems. Due to their high surface-to-volume ratio, which can carry a high payload of catalytically active species, NPs show very high catalytic activity and chemical selectivity under mild conditions.[28–36] Neat processes utilizing eco-friendly and green catalysts that can be simply recycled at the end of reactions has received remarkable attention in recent years. We report herein a simple and facile procedure for the synthesis of benzopyrano[2,3-b]pyridines through one-pot three-component reaction of salicylaldehydes, thiols and 2 equiv. of malononitrile catalyzed by ZrP_2O_7 NPs under reflux conditions in ethanol (Scheme 1).



Scheme 1. Synthesis of benzopyranopyridines via ZrP_2O_7 NPs catalyzed multicomponent reactions of salicylaldehydes with malononitrile and thiols.

2. Results and discussion

The morphology and particle size of ZrP_2O_7 NPs was investigated by scanning electron microscopy (SEM) as shown in Figure 1. The SEM image shows particles with diameters in the range of nanometers. The X-ray diffraction (XRD) pattern of the ZrP_2O_7 NPs is shown in

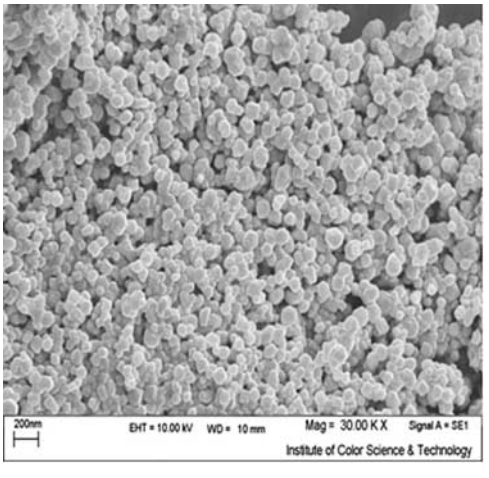


Figure 1. SEM images of ZrP_2O_7 NPs.

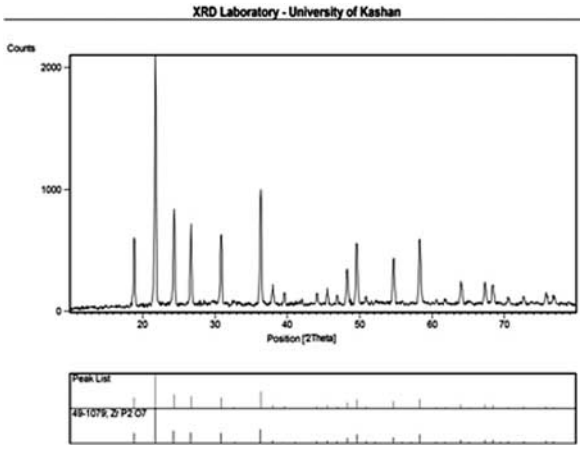


Figure 2. The XRD pattern of ZrP_2O_7 NPs.

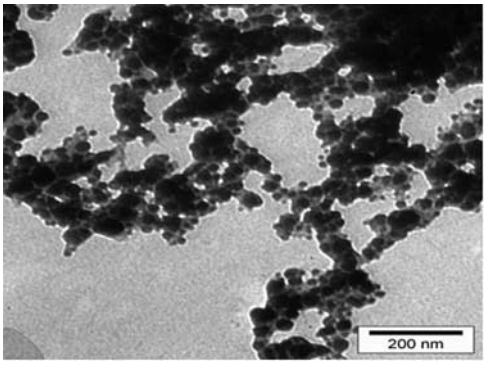


Figure 3. TEM image of ZrP_2O_7 NPs.

Figure 2. The average NP size was estimated from the full-width half-maximum of the peaks with use of the Debye–Sherrer equation. The results show that ZrP_2O_7 NPs were obtained with an average diameter of 11 nm as confirmed by the XRD analysis.

Characterization of nano ZrP_2O_7 showed the same particle size by transmission electron microscopy (TEM) (Figure 3).

In order to achieve optimum conditions, we initially investigated the reaction of salicylaldehydes, thiols and 2 equiv. of malononitrile in the presence of different catalysts such as DBU, $ZnCl_2$, $FeCl_3$, morpholine and ZrP_2O_7 NPs under different conditions (Table 1). Next, we optimized the required amount of ZrP_2O_7 NPs; the optimum amount was found to be 5 mol%.

The reaction works well for salicylaldehyde and 5-bromo-2-hydroxybenzaldehyde and different thiols. All the reactions reached completion within 40–55 min to afford good yields of products. These products precipitate from refluxing ethanolic solutions and are isolated by easy filtration. The yields of recrystallized benzopyranopyridines are given in Table 2.

2.1. *Proposed mechanism*

A plausible mechanism for the preparation of benzopyranopyridines using ZrP_2O_7 NPs is shown in Scheme 2.

Table 1. Optimization of reaction condition using different catalysts[a].

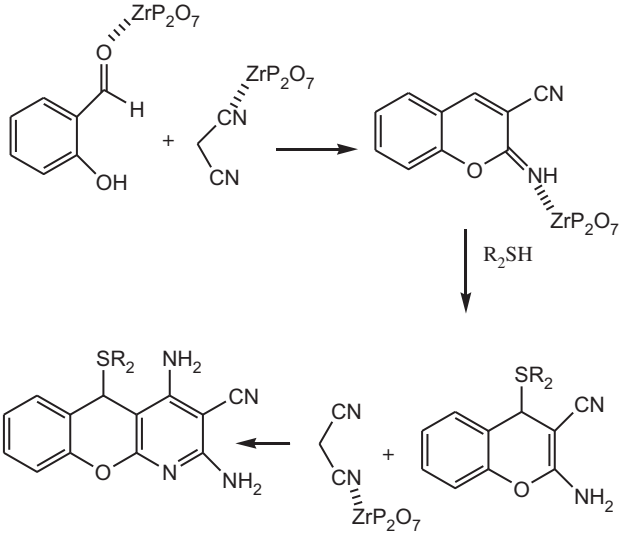
Entry	Catalyst	Mol (%)	Time (min)	Yield (%)[b]
1	DBU	30	200	40
2	$ZnCl_2$	10	100	18
3	$FeCl_3$	10	100	30
4	Morpholine	10	90	55
5	ZrP_2O_7 NPs	2	50	80
6	ZrP_2O_7 NPs	5	50	90
7	ZrP_2O_7 NPs	8	50	90

[a]Reaction conditions: salicyladehyde (1.5 mmol), malononitrile (3 mmol) and benzenethiol (1.5 mmol).
[b]Isolated yields.

Table 2. Synthesis of benzopyranopyridine derivatives using ZrP_2O_7 NPs[a].

Entry	R_1	R_2	Product	Time (min)	Yield[b]	m.p. (°C)	Lit. m.p. (°C)
1	H	Ar	1a	42	90	221–223	(220–222)[19]
2	H	4-Me-Ar	2a	47	87	222–224	(223–225)[19]
3	H	Ar-CH_2	3a	55	82	210–212	–
4	H	2-furyl-methyl	4a	48	85	230–234	–
5	Br	Ar	1b	40	91	215–217	–
6	Br	4-Me-Ar	2b	46	88	213–215	–
7	Br	Ar-CH_2	3b	57	83	206–208	–
8	Br	2-furyl-methyl	4b	46	87	225–227	–

[a]All the reactions were carried out under reflux conditions in ethanol.
[b]Isolated yields.



Scheme 2. Proposed reaction pathway for the synthesis of benzopyranopyridines by ZrP_2O_7 NPs.

3. Conclusions

In this research, ZrP_2O_7 NPs were used for mild preparation of benzopyranopyridine derivatives under reflux conditions in ethanol for the first time. The advantages offered by this method are satisfactory yields of products using a green and recyclable nanocatalyst, easy workup, short reaction time and mild reaction conditions.

4. Experimental

All organic materials were purchased commercially from Sigma-Aldrich and Merck and were used without further purification. All melting points are uncorrected and were determined in a capillary tube on a Boetius melting point microscope. FT-IR spectra were recorded with KBr pellets using a Magna-IR, spectrometer 550 Nicolet. NMR spectra were recorded on a Bruker 400 MHz spectrometer with DMSO as solvent and TMS as an internal standard. Powder XRD was carried out on a Philips diffractometer of X'pert Company. Microscopic morphology of the products was visualized by SEM (LEO 1455VP). TEM images were obtained on a Philips EM208 TEM with an accelerating voltage of 100 kV.

4.1. *Preparation of ZrP_2O_7 NPs*

The catalyst was prepared via a sonochemical method (worked at 20 kHz frequency and 80 W powers) using $ZrOCl_2$ as the zirconium source. The stoichiometric amount of $ZrOCl_2/8H_2O$ was first added to 20 mL of distilled water and dissolved with the aid of sonication. Then, H_3PO_4 (85%) was added dropwise in 20 min and the mixture was sonicated until the precipitation of solids was finished. When the reaction was completed, a dispersed white precipitate was obtained. The solid was filtered and washed with distilled water and ethanol several times. Subsequently, the catalyst was dried at 100°C for 8 h and calcined at 500°C for 1 h to obtain pure nano zirconium pyrophosphate.

4.2. *General procedure for the preparation of benzopyranopyridines*

To a mixture of a selected salicylaldehyde (1.5 mmol), malononitrile (3 mmol) and a desired thiol (1.5 mmol) in 5 mL of anhydrous ethanol was added ZrP_2O_7 NPs (5 mol%) in 2 mL ethanol at room temperature. The resulting mixture was refluxed for 40–50 min and then allowed to cool to room temperature. The formed precipitate was isolated by filtration. The product was dissolved in DMF (3 mL) and the catalyst was filtered. Then, 4 mL water was added to the filtrate which resulted in the crystallization of the product. The resulting crystalline structure was filtered and dried with a vacuum pump. The structures of the products were fully established on the basis of their ^{1}H NMR, ^{13}C NMR and FT-IR spectra.

4.3. *2,4-Diamino-5-phenylsulfanyl-5H-chromeno[2,3-b]pyridine-3-carbonitrile (1a)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}): 3355, 3429, 2203, 1400–1623; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 5.73$(s, 1H), 6.50 (2H, s), 6.74–6.78(3H, m), 6.94 (s, 2H), 7.05–7.11(3H, m), 7.15–7.30 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 160.9$, 159.7, 156.4, 150.9, 137.7, 134.2, 129.7,129.5, 129.2, 128.64, 128.60, 123.9, 121.5, 116.5, 116.01, 86.90, 70.8, 43.12. Anal. calcd for $C_{19}H_{14}N_4OS$: C 65.88, H 4.074, N 16.17. Found C 65. 82, H 4.09, N 16.07.MS (EI) (m/z): 346 (M^+).

4.4. *2,4-Diamino-5-(4-methylphenylsulfanyl)-5H-chromeno[2,3-b]pyridine-3-carbonitrile (2a)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}): 3457, 3349, 2198, 1400–1623. ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 2.19$ (s, 3H), 5.66 (s, 1H), 6.46 (br s, 2H), 6.61 (d, $J = 7.9$ Hz, 2H), 6.79 (d, $J = 7.9$ Hz, 1H), 6.90 (m, 4H), 7.11; (d, $J = 6.8$ Hz, 1H), 7.19 (d, $J = 7.3$ Hz, 2H).^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 160.25$, 160.08, 156.3, 151.33, 138.3, 136.9, 129.8, 128.6, 128.2, 127.0, 123.6,

121.8, 116.4, 115.7, 86.3, 70.8, 43.11, 21.2. Anal. calcd for $C_{20}H_{16}N_4OS$: C 66.64, H 4.47, N 15.54. Found C 66. 59, H 4.51, N 15.48.MS (EI) (m/z): 360 (M^+).

4.5. *2,4-Diamino-5(benzylthio)-5H-chromeno[2,3-b]pyridine-3-carbonitrile(3a)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}): 3377, 3439, 2200, 1400, 1605; ^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.49$ (ABq, 2H, $J = 12$ Hz), 5.48 (1H), 6.55 (bs, 2H), 6.83 (bs, 2H), 7.03–7.21 (m, 7H), 7.33 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 160.2$, 159.9, 152.9, 143.7, 136.6, 133.3, 133.2, 129.6, 129.4, 124.3, 124.2,122.8, 119.3, 118.3, 117.6, 87.01, 70.90, 44.01, 37.02. Anal. calcd for $C_{20}H_{16}N_4OS$: C 66.64, H 4.47, N 15.54. Found C 66. 48, H 4.58, N 15.39.MS (EI) (m/z): 360 (M^+).

4.6. *5-((Furan-2-yl) methylthio)-2,4-diamino-5H-chromeno[2,3-b]pyridine-3-carbonitrile (4a)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}): 3386, 3440, 2203, 1397, 1612;^{1}H NMR (400 MHz, DMSO-d_6): $\delta = 3.51$ (ABq, 2H, $J = 12$ Hz), 5.46(1H), 5.99 (1H), 6.23 (s, 1H), 6.55 (bs, 2H), 6.79 (bs, 2H), 7.10(d, 1H, $J = 8$ Hz), 7.17 (t, 1H, $J = 8$ Hz), 7.24(s, 1H), 7.31(t, 2H, $J = 8$ Hz); ^{13}C NMR (100 MHz, DMSO-d_6): $\delta = 160.10$, 160.3, 157.1, 151.2, 149.4, 142.7, 133.8, 129.7, 129.0, 116.9, 116.5, 111.0, 107.8, 87.9, 71.0, 35.03, 25.9. Anal. calcd for $C_{18}H_{14}N_4O_2S$: C 61.70, H 4.03, N 15.99. Found C 61.62, H 4.10, N 15.91. MS (EI) (m/z): 350(M^+).

4.7. *2,4-Diamino-5-(benzylthio)-7-bromo-5H-chromeno[2,3-b]pyridine-3-carbonitrile (3b)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}): 3315, 3441, 2189, 1403, 1653; ^{1}H NMR (400 MHz, DMSO-d_6) $\delta = 4.06$–4.45 (ABq, 2H, $J = 12$ Hz), 4.67 (1H), 6.48 (s, 1H), 6.88–6.90 (m, 5H), 7.37 (m,6H); ^{13}C NMR (100 MHz, DMSO-d_6) $\delta = 160.80$, 160.44, 152.75, 148.22, 137.96, 137.53, 131.05, 129.79, 128.60, 124.41,121.60, 120.90, 118.79, 116.46, 87.22, 35.78, 35.4, 33.34. Anal. calcd for $C_{20}H_{15}N_4OSBr$: C 54.68, H 3.44, N 12.75. Found C 54.55, H 3.52, N 12.61. MS (EI) (m/z): 439(M^+).

4.8. *5-((Furan-2-yl)methylthio)-2,4-diamino-7-bromo-5H-chromeno[2,3-b]pyridine-3-carbonitrile (4b)*

Yellow solid, IR (KBr) (ν_{max}/cm^{-1}):3311, 3442, 2194, 1404, 1655;^{1}H NMR (400 MHz, DMSO-d_6) $\delta = 4.14$–4.56 (ABq, 2H, $J = 12$ Hz), 4.89 (1H), 6.34 (s, 1H), 6.46 (d, $J = 8.3$ Hz,1H), 6.78–6.95 (m, 6H), 7.39 (d, 1H, $J = 9$ Hz), 7.62 (d, 1H);^{13}C NMR (100 MHz, DMSO-d_6) $\delta =$ 160.5, 155.24, 150.72, 148.21, 143.26, 131.89, 131.12, 125.50, 124.14, 119.41, 118.87, 116.50, 111.49, 108.77, 84.86, 54.27, 36.31, 28.64. Anal. calcd for $C_{18}H_{13}N_4O_2SBr$: C 50.39, H 3.05, N 13.06. Found C 50.35, H 2.97, N 13.01.MS (EI) (m/z): 429 (M^+).

4.9. *Catalyst recovery*

In the recycling procedure of ZrP_2O_7 NPs, DMF was added to dilute the reaction mixture after terminating the reaction. The catalyst was insoluble in the solvent and was separated by simple filtration. The recovered ZrP_2O_7 NPs was washed with ethanol and dried at 70°C for 2 h. The separated catalyst was used several times with a slightly decreased activity as given in Table 3.

Table 3. Recycling of ZrP_2O_7 NPs as catalyst.

Recycle	1	2	3	4	5
Yield (%)	94	92	90	88	85

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