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FULL PAPER



Zirconium oxide complex anchored on boehmite nanoparticles as highly reusable organometallic catalyst for C–S and C–O coupling reactions

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Funding information Ilam University Boehmite nanoparticles were prepared by a simple and inexpensive procedure in water using commercially available materials without inert atmosphere. Then, the surface of the boehmite nanoparticles was modified using 3mercaptopropyltrimethoxysilane and subsequently zirconium oxide was supported on the modified surface. Zirconium oxide supported on boehmite nanoparticles (Pr.S-ZrO@boehmite) was characterized using scanning electron microscopy, Fourier transform infrared spectroscopy, thermogravimetric analysis and inductively coupled plasma technique. The catalytic application of Pr. S-ZrO@boehmite was studied in C–O and C–S coupling reactions for synthesis of valuable compounds such as ether and sulfide derivatives. All products were obtained in good to excellent yields and the catalyst could be recovered and reused several times without significant loss of catalytic efficiency. Furthermore, zirconium oxide is rarely used as catalyst for cross-coupling reactions.

KEYWORDS

boehmite nanoparticles, C-O and C-S coupling reactions, ethers, sulfides, zirconium oxide

1 | INTRODUCTION

The recovery and recycling of catalysts are important factors in green chemistry and industrial applications.^[1-3] Recently to develop this issue, homogeneous catalysts have been supported on various solid materials such as magnetic nanoparticles,^[4–6] mesoporous materials,^[7] carbon nanotubes,^[8] ionic liquids,^[9,10] peptide nanofibres,^[11] polymers,^[12] graphene oxide^[13] and heteropolyacids.^[14] Boehmite nanoparticles (BNPs) are a novel solid support that has been rarely used as a catalyst support.^[15] High surface area, high stability in harsh conditions and high density of hydroxyl groups on the boehmite surface are unique properties of BNPs for their catalytic applications.^[16,17] More important, BNPs are a suitable support for heterogenization of catalysts due to the high thermal and chemical stability of BNPs.^[16,18] Because BNPs are prepared with commercial materials

using simple procedures, they are used as absorbents, vaccine adjuvants, coatings, optical material, cosmetic products, composite reinforcement material in ceramics, pillared clays and sweep-flocculation for freshwater treatment, and also rarely used as supports for catalysts.^[19–21] For these reasons, many methods have been reported for the preparation of BNPs, but these reports focused on physical and chemical properties of BNPs.^[22–24]

Therefore in continuation of our studies of the application of boehmite as an insoluble support,^[25,26] here we report a complex of zirconium oxide that is immobilized on BNPs (Pr.S-ZrO@boehmite). This catalyst was used for C–S and C–O coupling reactions. The formation of C–S and C–O bonds are great demand in organic chemistry for the production of several important pharmaceutical and biological compounds and there are several reports for the synthesis of these compounds.^[27–32] Sulfides are used as potent drugs for the WILEY Organometallic Chemistry treatment of HIV, cancer, Alzheimer's disease and Parkinson's disease.^[11] Sulfides are used as important materials for the synthesis of sulfoxide and sulfone

2 | EXPERIMENTAL

2 of 8

derivatives.^[5]

2.1 | Preparation of Catalyst

Initially, BNPs modified using 3mercaptopropyltrimethoxysilane (MPTMS) were prepared according to a newly reported procedure.^[17] For the preparation of Pr.S-ZrO@boehmite, the BNPs modified using MPTMS (1.0 g) were dispersed in ethanol and mixed with 2.5 mmol of $ZrOCl_2 \cdot 8H_2O$. The mixture was stirred at 80 °C for 20 h. The solid product (Pr.S-ZrO@boehmite) was obtained after washing with ethanol and drying at 50 °C.

2.2 | General Procedure for Synthesis of Diaryl Sulfides and Diaryl Ethers

A round-bottom flask was charged with 1.2 mmol of thiourea or 1 mmol of phenols, with aryl halide (1 mmol), KOH (6 mmol), Pr.S-ZrO@boehmite (40 mg, 6.2 mol%) and dimethylsulfoxide (DMSO; 3 ml). Then the reaction mixture was stirred at 130 °C. The progress of reaction was observed by TLC. After completion of the reaction, the mixture was extracted with ethyl acetate. The organic layer was dried with anhydrous Na₂SO₄, and organic solvent was evaporated to afford pure products.

2.3 | Selected Spectral Data

Phenyl(*p***-tolyl)sulfane** (Table 2, entry 4). ¹H NMR (300 MHz, CDCl₃, δ, ppm): 2.30 (s, 3H), 6.99–7.07 (m, 2H), 7.14–7.20 (m, 4H), 7.23–7.32 (m, 2H), 7.53–7.56 (m, 1H).

(3-Methoxyphenyl)(phenyl)sulfane (Table 2, entry 9). ¹H NMR (300 MHz, CDCl₃, *δ*, ppm): 3.71 (s, 3H), 6.86–6.89 (m, 2H), 7.16–7.18 (m, 1H), 7.25–7.35 (m, 4H), 7.35–7.37 (m, 2H).

1-Methoxy-3-phenoxybenzene (Table 4, entry 9). ¹H NMR (300 MHz, CDCl₃, δ , ppm): 3.72 (m, 3H), 6.51– 6.55 (m, 1H), 6.56–6.57 (m, 1H), 6.71–9.72 (d, J = 4 Hz, 1H), 6.99–7.02 (m, 2H), 7.11–7.15 (t, J = 6 Hz, 1H), 7.23–7.29 (t, J = 8 Hz, 1H), 7.35–7.40 (m, 2H).

1-Methoxy-4-phenoxybenzene (Table 4, entry 11). ¹H NMR (300 MHz, CDCl₃, *δ*, ppm): 3.71 (s, 3H), 6.53– 6.58 (m, 2H), 6.69–6.72 (m, 1H), 6.89–7.05 (m, 3H), 7.11–7.15 (m, 1H), 7.26–7.40 (m, 2H). In continuation of our studies of development of heterogeneous catalysts,^[25,26] herein we modified BNPs using MPTMS and used them as an efficient insoluble support for zirconium oxide (Scheme 1). This is the first report of the modification of BNPs using MPTMS. Also, zirconium oxide has rarely been applied as a catalyst for the synthesis of sulfide and ether derivatives.



SCHEME 1 Synthesis of Pr.S-ZrO@boehmite



FIGURE 1 SEM image of Pr.S-ZrO@boehmite

Pr.S-ZrO@boehmite was characterized using scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) spectroscopy, thermogravimetric analysis (TGA), CHNS elemental analysis, and inductively coupled plasma (ICP) analysis. SEM was used for evaluation of particles size. As shown in Figure 1, these nanoparticles were formed at a nanometre size (35-50 nm) with quite homogeneous size and shape.

ICP analysis was employed for the determination of the amount of zirconium loaded on the BNPs. According to the ICP analysis, the exact amount of zirconium immobilized on BNPs is 1.55×10^{-3} mol g⁻¹. In order to obtain the ratio of zirconium to sulfur in the catalyst, CHNS analysis was performed. According to this analysis, the amount of sulfur immobilized on BNPs is 5.2×10^{-3} mol g⁻¹. Therefore, the ratio of zirconium to sulfur in the catalyst is 1:3.

DTG/TGA of Pr.S-ZrO@boehmite was used to investigate organic layers coated on BNPs (Figure 2). The TGA diagram of Pr.S-ZrO@boehmite shows three weight losses with a heating rate of 10 °C min⁻¹ in the range 25 800 °C. The first weight loss about of 10% was seen below 100 °C due to the removal of physically adsorbed solvents.^[17] The second weight loss in the TGA diagram (about 20%) between 150 to 700 °C corresponds to the decomposition of organic layers. The final weight loss about 5% may be related to the transformation of thermal crystal phase of boehmite particles that occurs above 700 °C.^[33]

FT-IR spectra of BNPs, modified BNPs using MPTMS (Pr.SH@boehmite) and Pr.S-ZrO@boehmite are shown in Figure 3. The FT-IR spectra show several stretching vibration at 480, 630 and 740 cm⁻¹ which correspond to the vibration of Al-O bonds in the structure of BNPs.^[26] Vibrations of hydrogen bonds and nitrate impurity were indicated by two bands at 1080 and 1634 cm⁻¹, respectively. Symmetric and asymmetric vibrations of the surface hydroxyl groups were observed above 3000 cm^{-1.[17]} C-H stretching vibration bands were observed at 2880-2980 cm⁻¹ in the FT-IR spectra of Pr.SH@boehmite and Pr.S-ZrO@boehmite.

After synthesis and characterization of the catalyst, the application of this catalyst was studied in C-S and C-O coupling reactions (Scheme 2). In order to determine the best reaction conditions, various parameters were examined in the coupling of iodobenzene with thioacetamide as the source of sulfur. Therefore, the sample reaction was examined in the presence of various amounts of Pr.S-ZrO@boehmite (Table 1, entries 5-8) and in various solvents such as DMSO, dimethylformamide (DMF), poly(ethylene glycol) (PEG), toluene, and water (Table 1, entries 1-5). As evident from Table 1, the best results were obtained in DMSO using 0.04 g (6.2 mol%) of catalyst. Also, the sample reaction was examined at various temperatures (Table 1, entries 8-12) and with different amounts of KOH (Table 1, entries 12-14). The best results were obtained using 6 mmol of KOH at 130 °C.

After optimization of the reaction conditions, the investigation of the catalytic activity of Pr.S-ZrO@boehmite was extended to a wide range of aryl halides including those electron-donating and electron-withdrawing functional groups. Also arvl iodides, aryl bromides and aryl chlorides were employed for preparation of corresponding diaryl sulfides. Results of these studies are summarized in Table 2. All products were obtained in good to excellent yields.

40.00 3.400 95.00 40.00 30.00 ZR.S 20.00 90.00 20.00 3.200 DTG ug/min 10.00 0.00 N 3.000 an 85.00 DTA 0.00 -20.00 2.800 E 80.00 -10.00 -40.00 75.00 -20.00 2.600 -60.00 -30.00 70.00 2.400 -80.00 -40.00 65.00 100.0 200.0 300.0 400.0 500.0 600.0 700.0 800.0 Temp Cel

FIGURE 2 TGA/DTA curves of Pr.S-ZrO@boehmite





FIGURE 3 FT-IR spectra of (a) BNPs, (b) Pr.S@boehmite and (c) Pr.S-ZrO@boehmite

Also, in order to optimize the reaction conditions for the synthesis of diaryl ethers, we examined the C–O coupling of iodobenzene with phenol in the presence of Pr.S-ZrO@boehmite as a model reaction. Different amounts of catalyst and various solvents were examined (Table 3). In the C–O coupling of iodobenzene with phenol, the best results were obtained in DMSO using 40 mg (6.2 mol%) of Pr.S-ZrO@boehmite (Table 3, entry 5). Effects of temperature, type of base and amount of base were examined, with 130 °C and 6 mmol of KOH being selected as optimum.

In order to extend the scope of catalytic activity of Pr.S-ZrO@boehmite, we examined the C—O coupling reactions for various aryl halides. The results are summarized in Table 4. Various aryl iodides, bromides and chlorides (Table 4) were successfully converted to diaryl ethers in short reaction times and in high yields.



Pr.S-ZrO@boehmite

DMSO, KOH, 130 °C

Pr.S-ZrO@boehmite

DMSO, KOH, 130 °C

) + _{H3}C

NH₂

The recoverability and reusability of Pr.S-ZrO@boehmite was studied in C—O and C—S coupling reactions. To investigate this issue, iodobenzene was selected as sample substrate. After completion of the reactions, the catalyst was recovered by centrifugation and was used again. As shown in Figure 4, Pr.S-ZrO@boehmite can be reused up to five times without any significant loss of its catalytic activity in the synthesis of diphenyl sulfide and diphenyl ether. TABLE 1 Optimizing reaction conditions for synthesis of sulfide derivatives in the presence of Pr.S-ZrO@boehmite

Entry	Solvent	Catalyst (mg (mol%))	Amount of KOH (mmol)	Temperature (°C)	Yield (%) ^a
1	H_2O	40 (6.2)	6	Reflux	b
2	Toluene	40 (6.2)	6	Reflux	b
3	DMF	40 (6.2)	6	130	b
4	PEG	40 (6.2)	6	130	b
5	DMSO	40 (6.2)	6	130	94
6	DMSO	20 (3.1)	6	130	40
7	DMSO	30 (4.6)	6	130	80
8	DMSO	50 (7.7)	6	130	94
9	DMSO	40 (6.2)	6	100	Trace
10	DMSO	40 (6.2)	6	80	b
11	DMSO	40 (6.2)	6	60	b
12	DMSO	40 (6.2)	6	40	b
13	DMSO	40 (6.2)	5	130	83
14	DMSO	40 (6.2)	4	130	77

^aIsolated yield after 60 min.

^bNo reaction.

TABLE 2	C-S coupling	reaction for	synthesis of	f sulfides	catalysed l	oy Pr.S-2	ZrO@boehmite
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Entry	Aryl halide	Time (min)	Yield (%) ^a	Melting point (°C)	Reported melting point (°C)
1	Iodobenzene	60	92	Oil	Oil ^[34]
2	Bromobenzene	180	87	Oil	Oil ^[34]
3	Chlorobenzene	24 h	27	Oil	Oil ^[34]
4	4-Iodotoluene	130	60	Oil	Oil ^[35]
5	4-Bromotoluene	500	50	Oil	Oil ^[35]
6	4-Bromonitrobenzene	15	49	157–159	150–161 ^[34]
7	4-Chloronitrobenzene	30	40	157–159	150–161 ^[34]
8	4-Bromochlorobenzene	155	50	Oil	Oil ^[35]
9	3-Bromoanisole	140	80	Oil	Oil ^[35]
10	2-Iodoanisole	330	87	Oil	Oil ^[34]
11	1-Bromo-3-(trifluoromethyl)benzene	70	73	Oil	Oil ^[34]
12	4-Iodoanisole	300	75	Oil	Oil ^[34]
13	2-Iodotoluene	125	65	Oil	Oil ^[36]

^aIsolated yield.

An SEM image of the recovered catalyst is shown in Figure 5. Particles of the recovered catalyst were observed with sizes between 35 and 50 nm with homogeneous nature and cubic orthorhombic structures. Therefore, the size and shape of catalyst particles have not been changed after recycling.

Also, the amounts of zirconium leaching after recycling of Pr.S-ZrO@boehmite were studied by

determining the zirconium loading amount before and after recycling of the catalyst using ICP analysis. Based on such analysis, the amounts of zirconium in fresh and reused catalyst are 1.55 and 1.41 mmol g⁻¹, respectively, which shows that zirconium leaching from Pr.S-ZrO@boehmite is very low.

Applied Organometallic– Chemistry 5 of 8

6 of 8 WILEY Organometallic Chemistry

TABLE 3 Optimizing reaction conditions for synthesis of diaryl ether derivatives in the presence of Pr.S-ZrO@boehmite

Entry	Solvent	Catalyst (mg (mol%))	Base	Amount of base (mmol)	Temperature (°C)	Time (min)	Yield (%) ^a
1	DMF	40 (6.2)	КОН	6	130	120	b
2	PEG	40 (6.2)	КОН	6	130	120	b
3	Toluene	40 (6.2)	КОН	6	Reflux	120	b
4	H_2O	40 (6.2)	КОН	6	Reflux	120	b
5	DMSO	40 (6.2)	КОН	6	130	120	90
6	DMSO	20 (3.1)	КОН	6	130	120	58
7	DMSO	30 (4.6)	КОН	6	130	120	72
8	DMSO	50 (7.7)	КОН	6	130	120	90
9	DMSO	40 (6.2)	K_2CO_3	6	130	120	b
10	DMSO	40 (6.2)	Na ₂ CO ₃	6	130	120	b
11	DMSO	40 (6.2)	Et ₃ N	6	130	120	b
12	DMSO	40 (6.2)	КОН	5	130	120	88
13	DMSO	40 (6.2)	КОН	4	130	120	70
14	DMSO	40 (6.2)	КОН	3	130	120	58
15	DMSO	40 (6.2)	КОН	6	100	120	Trace
16	DMSO	40 (6.2)	КОН	6	80	120	b
17	DMSO	40 (6.2)	КОН	6	60	120	b
18	DMSO	40 (6.2)	КОН	6	40	120	b

^aIsolated yield.

^bNo reaction.

 TABLE 4
 C—O coupling reaction for synthesis of diaryl ethers catalysed by Pr.S-ZrO@boehmite

Entry	Aryl halide	Time (min)	Yield (%) ^a	Melting point (°C)	Reported melting point (°C)
1	Iodobenzene	160	90	Oil	Oil ^[37]
2	Bromobenzene	450	85	Oil	Oil ^[37]
3	Chlorobenzene	24 h	25	Oil	Oil ^[37]
4	4-Bromonitrobenzene	30	75	55–57	57-59 ^[37]
5	4-Bromochlorobenzene	60	50	Oil	Oil ^[37]
6	4-Iodotoluene	420	82	Oil	Oil ^[38]
7	4-Chloronitrobenzene	40	71	55–57	57-59 ^[37]
8	4-Bromotoluene	510	80	Oil	Oil ^[38]
9	3-Bromoanisole	370	72	Oil	Oil ^[38]
10	4-Iodoanisole	12 h	75	Oil	Oil ^[37]
11	4-Bromoanisole	55	70	Oil	Oil ^[37]

^aIsolated yield.

The efficiency of Pr.S-ZrO@boehmite for C–S coupling reactions was compared with previous literature for the coupling of iodobenzene with sulfur. The results of this comparison are presented in Table 5. Pr.S-ZrO@boehmite is better than other catalysts in

terms of price, non-toxicity, activity, stability and ease of separation. Also this catalyst can be reused and the average isolated yield for five successive runs is more than 90%, which clearly demonstrates the practical recyclability of this catalyst.



FIGURE 4 Recycling experiments of Pr.S-ZrO@boehmite in synthesis of (a) diphenyl sulfide and (b) diphenyl ether



FIGURE 5 SEM image of Pr.S-ZrO@boehmite after recycling

TABLE 5 Comparison of Pr.S-ZrO@boehmite for synthesis ofdiphenyl sulfide with previously reported procedures

Entry	Catalyst	Time (h)	Yield (%) ^a	Ref.
1	IMes-Cu-Cl	6	81	[32]
2	Cu ^{II} -ZSM5	17	95	[39]
3	Cu ^I -ZSM5	15	91	[39]
4	Ni–NHC	16	99	[40]
5	CuI	16	99	[41]
6	PdNP-PNF	5	92	[11]
7	Pr.S-ZrO@boehmite	1	92	This work

^aIsolated yield.

4 | CONCLUSIONS

In summary, a new complex of zirconium supported on modified BNPs (Pr.S-ZrO@boehmite) is reported as a novel, highly reusable and stable catalyst for C—S and C—O coupling reactions. This catalyst can be recovered and reused for several cycles without loss of its catalytic activity. The advantage of this catalyst is its simple preparation using commercially available, eco-friendly and cheap materials. Also, nano-boehmite is new support for heterogenization of homogeneous catalysts. Diaryl sulfides and diaryl ethers were obtained in high yields in the presence of Pr.S-ZrO@boehmite.

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7 of 8

Applied

Organometallic

8 of 8 WILEY Organometallic Chemistry

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