FULL PAPER



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A new Pd-Schiff-base complex on boehmite nanoparticles: Its application in Suzuki reaction and synthesis of tetrazoles

Arida Jabbari¹ Bahman Tahmasbi² Kohsen Nikoorazm² Arash Ghorbani-Choghamarani²

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¹Department of Chemistry, Islamic Azad University, Qeshm Branch, Qeshm, Iran ²Department of Chemistry, Ilam University, P.O. Box 69315516, Ilam, Iran

Correspondence

Arida Jabbari, Department of Chemistry, Islamic Azad University, Qeshm Branch, Qeshm, Iran. Email: arida_jabbari@yahoo.com

Funding information Ilam University Boehmite nanoparticles are not air or moisture sensitive, therefore have been prepared in water without inert atmosphere using inexpensive materials. Pd-isatin-boehmite was synthesized and used as heterogeneous organometallic catalyst for the synthesis of 5-substituted 1*H*-tetrazoles and C-C band formation. Pd-isatin-boehmite characterized by XRD, TGA, SEM and ICP-OES techniques. This catalyst was reused for several times without significant loss of its catalytic efficiency or palladium leaching. Heterogeneity of this catalyst has been studied using ICP-OES technique.

KEYWORDS

boehmite nanoparticles, organometallic catalyst, palladium, schiff-base, tetrazoles

1 | INTRODUCTION

Recently, immobilized homogeneous catalysts on solid supports have been employed as reusable catalysts in organic functional group transformations.^[1-4] Among various material, boehmite nanoparticles (γ -AlOOH) is great support for heterogenization of homogeneous catalyst because boehmite is stable and producible in water using commercially available and non-expensive materials.^[5,6] Also surface hydroxyl groups and high specific surface area (up to $(>120 \text{ m}^2/\text{g})$ was leads to high catalysts loading capacity on boehmite nanoparticles, which is leads to its high catalytic activity.^[7] Boehmite nanoparticles have several attractive features, such as thermal, mechanical and chemical stability, easily and readily available, high dispersity of the active phases, non-toxicity and ease of surface modification.^[6–10] Several methods were reported for the preparation of boehmite and all of them were focused on chemical and physical properties of boehmite.^[11–13] Modification of boehmite has been rarely reported as heterogeneous support;^[14,15] therefore herein Pd-isatin-boehmite has been reported as new organometallic catalyst for the Suzuki reaction and synthesis of 5-substituted 1H-tetrazole derivatives. Tetrazoles as five

membered heterocyclic compounds are of interest in pharmaceuticals, in synthetic organic chemistry, in coordination chemistry as a ligand, in catalysis technology, in the photographic industry and in organometallic chemistry.^[16–20] Also, tetrazoles and their derivatives have been reported as analgesic, antiviral, anti-inflammatory, anti-proliferative, antibacterial, potential anti HIV drug candidate, antifungal, herbicidal and anticancer agents.^[21–24] Likewise, the C-C band formation has been employed in the synthesis of advanced materials and natural products, pharmaceuticals, agrochemicals and biologically active compounds^[25,26] as well as in engineering materials has attracted enormous interest.^[27,28] The Suzuki cross coupling reaction is one of the most general and powerful tools for the formation C-C bands.^[28,29]

2 | EXPERIMENTAL

2.1 | Preparation of the catalyst

The boehmite nanoparticles were prepared according to the reported procedure and further modified by APTES (n-Pr-NH₂-boehmite).^[4,5] Then, in order to prepare functionalized boehmite-isatin-Schiff base (isatin-

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boehmite); n-Pr-NH₂-boehmite (1 g) was refluxed with 2 mmol of isatin in ethanol for 12 h under N₂ atmosphere. The resulting solid (isatin-boehmite) was filtered, washed with ethanol and dried at room temperature. Finally, for the preparation of Pd-isatin-boehmite, the isatin-boehmite (0.5 g) was mixed with 0.25 g of Pd(OAc)₂ in 20 ml of ethanol. The mixture was stirred at 80 °C for 20 h. Then, the NaBH₄ (0.4 mmol) was added to the reaction mixture and stirred for 2 hours. The solid product was obtained by filtration, washed with ethanol and dried at 60 °C.

2.2 | General procedure for the synthesis of 5-substituted 1H-tetrazoles

A mixture of sodium azide (1.5 mmol) and nitrile (1 mmol) in the presence of 0.030 g of Pd-isatin-boehmite, was stirred at 120 °C in PEG-400; after completion of the reaction (observed by TLC), the reaction mixture was cooled down, and catalyst was isolated by simple filtration and HCl (4 N, 10 ml) was added to the filtrated solution. The products extracted with ethyl acetate (2×10 ml). The organic solvent was dried over anhydrous sodium sulfate, and concentrated to give the crude solid product.

2.3 | General procedure for the Suzuki reaction

A mixture of aryl halide (1 mmol), phenylboronic acid (1 mmol) or sodium tetraphenyl borate (0.5 mmol), K_2CO_3 (3 mmol), Pd-isatin-boehmite (0.010 g) and 2 ml of PEG-400 were stirred at 80 °C and the progress of reaction was monitored by TLC. After completion of the reaction, catalyst was isolated and washed with diethyl ether. The reaction mixture was extracted with H₂O and diethyl



SCHEME 1 Synthesis of Pd-isatin-boehmite

ether and organic layer dried over anhydrous Na_2SO_4 (1.5 g). Then the solvent was evaporated and pure biphenyl derivatives were obtained in good to excellent yields.



FIGURE 1 SEM image of Pd-isatin-boehmite



FIGURE 2 The XRD patterns of Pd-isatin-boehmite



FIGURE 3 TGA/DTA diagram of Pd-isatin-boehmite

3 | RESULTS AND DISCUSSION

Pd-isatin-boehmite was prepared by the concise route outlined in Scheme 1, and then have been identified by scanning electron microscopy (SEM), X-ray diffraction



SCHEME 2 Synthesis of 5-substituted 1H-tetrazole derivatives in the presence of Pd-isatin-boehmite

(XRD), inductively coupled plasma atomic emission spectroscopy (ICP-OES) and thermogravimetric analysis (TGA).

Figure 1 shows the SEM image of Pd-isatin-boehmite. As shown in this image, the size of Pd-isatin-boehmite is quite homogeneous spherical particles.

Also, the exact amount of palladium loading was calculated by inductively coupled plasma atomic emission spectroscopy (ICP-OES), that the amount of palladium in the Pd-isatin-boehmite was 1.48 mmol g⁻¹.

The XRD patterns of Pd-isatin-boehmite are shown in Figure 2. Which the boehmite phase has been identified

TABLE 1	Optimization of reaction	conditions for synthesis	s of 5-substituted 1H-	tetrazole derivatives in the i	presence of Pd-isatin-boehmite
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$ \begin{array}{c} & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & \\ & $								
Entry	Catalyst (mg)	Solvent	NaN ₃ (mmol)	Temperature (°C)	Time (h)	Yield (%) ^a		
1	30	PEG	1.3	120	8	46		
2	30	PEG	1.4	120	8	49		
3	30	PEG	1.5	120	8	94		
4	35	PEG	1.5	120	7	96		
5	25	PEG	1.5	120	8	55		
6	20	PEG	1.5	120	10	44		
7	30	DMSO	1.5	120	6.7	89		
8	30	DMF	1.5	120	6.4	95		
9	30	H ₂ O	1.5	Reflux	10	33		
10	30	EtOH	1.5	Reflux	10	21		
11	30	Acetone	1.5	Reflux	10	Trace		
12	30	CH_2Cl_2	1.5	Reflux	10	Trace		
13	30	PEG	1.5	100	10	69		

^aIsolated yield.

TABLE 2 Synthesis of 5-substituted 1H-tetrazole derivatives in the presence of Pd-isatin-boehmite

Entry	Nitrile	Time (h)	Yield (%) ^a	Melting point (°C)	Reported melting point (°C) [reference]
1	benzonitrile	8	94	212-214	213-214 ^[25]
2	4-nitrobenzonitrile	4	92	216-218	217-220 ^[5]
3	4-acetylbenzonitrile	32	91	172-175	173-176 ^[5]
4	3-chlorobenzonitrile	10	91	127-129	128-130 ^[25]
5	2-chlorobenzonitrile	4.2	93	180-183	181-183 ^[19]
6	2-hydroxybenzonitrile	8.2	88	222-225	221-224 ^[25]
7	terephthalonitrile	6.7	85	250-254	251-254 ^[25]
8	3-nitrobenzonitrile	9	87	148-150	149-151 ^[19]
9	4-hydroxybenzonitrile	5.3	90	231-234	233-235 ^[5]
10	4-chlorobenzonitrile	7.5	88	261-264	262-265 ^[19]

^aIsolated yield.

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Applied Organometallic Chemistry by the several peak of 2θ at 14.42, 28.41, 38.52, 46.45, 49.54, 51.93, 56.45, 60.72, 65.10, 65.56, 68.12, and 72.42 from the XRD patterns, which is good agreement with standard boehmite XRD spectrum.^[10,25] It could be seen



SCHEME 3 Mechanism for the synthesis of tetrazoles in the presence of Pd-isatin-boehmite



SCHEME 4 C-C coupling reaction catalyzed Pd-isatin-boehmite

that the boehmite phase has not been destroyed during the modifications. Also the XRD pattern of Pd-isatinboehmite contains a series of characteristic diffraction peaks (39.85°, 45.97° and 66.85°) which are indexed to the Pd indicating the presence of Pd on surface of boehmite nanoparticles.^[10,30]

Quantitative determination of the organic groups supported on the surface of boehmite nanoparticles was determined by TGA (Figure 3). TGA/DTA diagram of Pd-isatin-boehmite showed a three-step weight loss. The first one appearing below 100 °C, shows a weight loss of 5% which is associated to the removal of physically adsorbed solvents on the surface of particles.^[23,29] The second one has appeared in the temperature range of 250-600 °C (about 22 %) that is due to the organic groups which is supported on the surface of boehmite nanoparticles. The third weight loss about 4% between 750 and 850 °C may be associated with the transformation of thermal crystal phase of boehmite particles.

After characterization of Pd-isatin-boehmite, we described the application of Pd-isatin-boehmite as an reusable and inexpensive catalyst for the synthesis of 5-substituted 1H-tetrazole derivatives via [2+3] cycloaddition reaction of nitriles with sodium azide (NaN₃) (Scheme 2).

Pd-isatin-boehmite							
		+					
Enters	Catalyst (ma)	Colvert	Base (2 mm al)	Tomorouture (%C)	Time (min)	Wield (0/)a	
Entry	Catalyst (mg)	Solvent	Base (3 mmol)			y leid (%)	
1	-	PEG	K ₂ CO ₃	80	600		
2	5	PEG	K ₂ CO ₃	80	135	92	
3	7	PEG	K ₂ CO ₃	80	80	96	
4	10	PEG	K ₂ CO ₃	80	50	97	
5	12	PEG	K_2CO_3	80	45	96	
6	10	DMF	K ₂ CO ₃	80	40	95	
7	10	H_2O	K ₂ CO ₃	80	120	88	
8	10	DMSO	K ₂ CO ₃	80	90	91	
9	10	Dioxane	K ₂ CO ₃	80	220	42	
10	10	PEG	NaOEt	80	50	Trace	
11	10	PEG	КОН	80	50	45	
12	10	PEG	Et ₃ N	80	50	69	
13	10	PEG	K ₂ CO ₃	60	100	60	
14	10	PEG	K ₂ CO ₃	40	100	Trace	
15	10	PEG	K ₂ CO ₃	r.t.	180	Trace	

TABLE 3 Optimization of reaction conditions for Suzuki reaction in the presence of Pd-isatin-boehmite

^aIsolated yield,

^bNo reaction.

In order to find the best reaction conditions, the effect of various parameters such as temperature, different solvents (such as dichloromethane, DMF, acetone, DMSO, PEG, H₂O and EtOH), amount of sodium azide and amount of Pd-isatin-boehmite have been examined in the synthesis of 5-phenyl-1H-tetrazole (Table 1). Therefore, 0.03 g of Pd-isatin-boehmite using 1.5 mmol of NaN₃ in PEG-400 at 120 °C was found to be the best reaction conditions for the synthesis of 5-phenyl-1Htetrazole.

After optimization of reaction conditions, the catalytic activity of Pd-isatin-boehmite was extended for wide range of substrates (Table 2). Various substrates with both electron-donating and electron-withdrawing functional groups on the benzonitriles have been converted to corresponding tetrazoles, and all products were obtained in good yields. As shown in Table 2, terephthalonitrile was afforded the monoaddition product (Table 2, entry 7).

Based on reported mechanism in the literatures, initially, Pd-isatin-boehmite reacts with nitrile to produce the intermediate I; which is convert to intermediate II through [3+2] cycloaddition reaction of C=N bond with azide. The protonolysis of intermediate II by HCl gives III, which is rearranges to stable product IV and catalyst was regenerated (Scheme 3).

Also, we examined the catalytic activity of Pd-isatinboehmite in the C-C coupling of aryl halides with phenylboronic acid or sodium tetraphenyl borate (Scheme 4).

In order to found the best reaction conditions, the effect of different parameters such as amount of catalyst,

TABLE 4 Suzuki reaction in the presence of Pd-isatin-boehmite

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base, solvent and temperature were studied in the coupling of iodobenzene and phenylboronic acid as a model reaction. Results of this studies is summarized in Table 3. As shown, the best results were obtained by 10 mg of Pd-isatin-boehmite at 80 °C. Also, the coupling of iodobenzene with phenylboronic acid was carried out in various solvents and bases. The best results were obtained in PEG using 3 mmol of K₂CO₃.

In order to extend of catalytic activity of Pd-isatinboehmite, we examined the coupling of other aryl halide including electron-donor and electron-withdrawing functional groups with phenylboronic acid (Table 4, entries 1-11). All aryl iodides (Table 4, entries 1-3), aryl bromides (Table 4, entries 4-9) and aryl chlorides (Table 4, entries 10 and 11) were successfully coupled



SCHEME 5 Mechanism for the Suzuki reaction in the presence of Pd-isatin-boehmite

Entry	Aryl halide	Phenylatig reagent	Time (min)	Yield (%) ^a	Melting point (°C)	Reported melting point (°C) [reference]
1	Iodobenzene	PhB(OH) ₂	50	97	62-64	62-65 ^[31]
2	4-Iodoanisole	PhB(OH) ₂	55	92	82-85	82-84 ^[29]
3	4-Iodotoluene	PhB(OH) ₂	90	93	43-45	42-44 ^[31]
4	4-Bromochlorobenzene	PhB(OH) ₂	60	95	70-72	70-71 ^[10]
5	4-Bromotoluene	PhB(OH) ₂	60	96	43-45	42-44 ^[31]
6	4-Bromobenzonitrile	PhB(OH) ₂	200	90	82-84	83 ^[10]
7	4-Bromonitrobenzene	PhB(OH) ₂	140	94	111-114	111-112 ^[29]
8	4-Bromoanisole	PhB(OH) ₂	420	95	82-85	82-84 ^[29]
9	Bromobenzene	PhB(OH) ₂	110	88	62-64	62-65 ^[31]
10	Chlorobenzene	PhB(OH) ₂	250	87	62-64	62-65 ^[31]
11	4-Chlorobenzonitrile	PhB(OH) ₂	400	89	82-84	83 ^[10]
12	Iodobenzene	NaB(Ph) ₄	30	91	62-64	62-65 ^[31]
13	4-Iodoanisole	NaB(Ph) ₄	40	92	82-85	82-84 ^[29]
14	4-Bromonitrobenzene	NaB(Ph) ₄	70	97	111-114	111-112 ^[29]

^aIsolated yield.

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and corresponding biphenyls were obtained in good to excellent yields.

We also performed coupling of aryl halides with sodium tetraphenyl borate in the presence of Pd-isatinboehmite (Table 4, entries 12-14) and the corresponding biphenyls were obtained in the good to excellent yields.



FIGURE 4 Recyclability of Pd-isatin-boehmite in the synthesis of 5-(2-chlorophenyl)-1H-tetrazole

Therefore, these results shown that this procidure is effective for a wide range of substrates.

Catalytic cycle for Suzuki reaction in the presence of Pd-isatin-boehmite is outlined in Scheme 5.^[32,33]

The reusability of Pd-isatin-boehmite was studied for the synthesis of 5-(2-chlorophenyl)-1H-tetrazole under optimized conditions. The catalyst has been reused up to five times without any significant loss of its catalytic activity (Figure 4). Palladium leaching of Pd-isatinboehmite was described by ICP technique. Which amount of palladium in fresh catalyst and the recovered catalyst is 1.48 mmol g⁻¹ and 1.35 mmol g⁻¹, respectively, which shows the practical reusability of this catalyst

In order to examine leaching of palladium in reaction mixture and heterogeneity of Pd-isatin-boehmite, hot filtration test was performed for the synthesis of biphenyl with coupling of iodobenzene and phenylboronic acid. In this experiment we obtained the yield of product in half time of the reaction that it was 59%. Then the same reaction was repeated and in half time of the reaction, the catalyst was separated and the filtered solution was allowed to react for 50 min. The yield of reaction in this stage was 62%. We found that, after this hot filtration, no further reaction was observed, this result confirmed the leaching of palladium is negligible.

In order to confirm the efficiency and advantages of Pd-isatin-boehmite than previous reported catalysts, synthesis of 5-phenyl-1H-tetrazole and biphenyl in the presence of Pd-isatin-boehmite was compared with previous catalysts in the literatures (Table 5). Beter catalytic activity of Pd-isatin-boehmite led to short

TABLE 5 Comparison results of Pd-isatin-boehmite with other catalysts for synthesis of 5-phenyl-1H-tetrazole and biphenyl

Entry	Catalyst	Substrate	Product	Time (min)	Yield (%)	Ref.
1	Pd/SH-SiO ₂	Iodobenzene	Biphenyl	12 h	95	[34]
2	Pd-MPA@MCM-41	Iodobenzene	Biphenyl	100	95	[35]
3	Pd@MTiO ₂	Iodobenzene	Biphenyl	180	99	[36]
4	CA/Pd(0)	Iodobenzene	Biphenyl	120	94	[37]
5	PdCl ₂	Iodobenzene	Biphenyl	120	95	[38]
6	PANI-Pd	Iodobenzene	Biphenyl	240	91	[39]
7	Pd-SBTU@Fe ₃ O ₄	Iodobenzene	Biphenyl	450	92	[40]
8	Pd-isatin-boehmite	Iodobenzene	Biphenyl	50	97	This work
9	PdCl ₂	Benzonitrile	5-phenyl-1H-tetrazole	8 h	81	[41]
10	(NH ₄)Ce(NO ₃) ₆	Benzonitrile	5-phenyl-1H-tetrazole	6 h	97	[42]
11	Amberlyst-15	Benzonitrile	5-phenyl-1H-tetrazole	12 h	91	[43]
12	CuFe ₂ O ₄	Benzonitrile	5-phenyl-1H-tetrazole	12 h	82	[44]
13	FeCl ₃ -SiO ₂	Benzonitrile	5-phenyl-1H-tetrazole	12	79	[45]
14	MZHSs	Benzonitrile	5-phenyl-1H-tetrazole	36 h	86	[46]
15	Pd-isatin-boehmite	Benzonitrile	5-phenyl-1H-tetrazole	8 h	94	This work

reaction times and good yields of products than other catalysts that were reported in previous literatures. Also this new catalyst is comparable in terms of price, non-toxicity, stability and easy separation. In addition, recovered and recycled of this catalyst is more rapid and easier than other catalysts.

CONCLUSIONS 4

Pd-isatin-boehmite as a new type of reusable heterogeneous nanocatalyst was reported and has been identified using XRD, TGA, ICP-OES and SEM techniques. This catalyst showed good reusability, high catalytic activity and excellent stability for synthesis of 5-substituted 1H-tetrazole derivatives and Suzuki reaction. The advantages of this protocol are commercially available materials, the operational simplicity and good yields, and more importance the catalyst can be synthesized from commercially inexpensive and available starting materials. Heterogeneity of this catalyst has been studied by ICP-OES technique.

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ORCID

Arida Jabbari D http://orcid.org/0000-0002-0559-9978 Bahman Tahmasbi D http://orcid.org/0000-0002-3605-2852 Mohsen Nikoorazm D http://orcid.org/0000-0002-4013-0868 Arash Ghorbani-Choghamarani D http://orcid.org/0000-0002-7212-1317

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