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Short communication

Synthesis of biaryl compounds via Suzuki homocoupling reactions catalyzed by metal organic frameworks encapsulated with palladium nanoparticles

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ABSTRACT

Heterogeneous homocoupling reactions of phenylboronic acids were greatly accelerated via Suzuki homocoupling reactions. In this work, a tandem route was designed which firstly one part of phenylboronic acids reacted with iodine to form iodobenzenes, then another part of phenylboronic acids coupled with iodobenzenes to produce biaryl compounds. The tandem reaction were catalyzed by a bifunctional heterogeneous catalyst of metal organic frameworks encapsulated with palladium nanoparticles (Pd@MOFs). This strategy for forming symmetric C-C bond between benzene rings has obvious advantages such as high efficiency, easy separation, good recyclability and no addition of toxic halogenated benzene.

1. Introduction

Homocoupling reaction of phenylboronic acids is an important strategy for forming symmetric C-C bond between benzene rings [1-3]. Symmetrical biaryls are of great significance in versatile applications of drugs, dyes, agrochemicals, semi-conductor and optically active ligands [4-7]. Homocoupling reaction of phenylboronic acids catalyzed by different catalysts has been extensively reported such as Pd(OAc)₂ [8], Cu(BDC) [9], the clay encapsulated Cu(OH)_x [10], gold nanoparticles [2,11], Fe₃O₄ nanoparticle-supported Cu(II)-β-cyclodextrin complex [12], and so on. They can be divided into two types. One is a homogeneous catalyst such as Pd(OAc)₂ reported by Dwivedi et al. [8], which is difficult to separate and recycle and easy to cause metal pollution. The other is a heterogeneous catalyst such as Cu(BDC) reported by Puthiaraj et al. [9], which is not very stable and efficient.

Heterogeneous Suzuki cross-coupling catalysts has been extensively reported [13-18], due to their highly efficiency and stability. A typical Suzuki reaction is phenylboronic acid coupled with halogenated benzene, catalyzed by zero-valent palladium complex [19]. Among all of the halogenated benzenes, iodobenzene is the most suitable for Suzuki reaction [20]. More importantly, iodobenzenes can be synthesized from phenylboronic acids catalyzed by various forms of Cu(II) [21,22].

Therefore, as long as there are two catalytic sites of Pd(0) and Cu(II), phenylboronic acids could synthesize biaryl compounds via a Iodination-Suzuki tandem reaction.

Metal organic frameworks (MOFs) composed of metal ions and organic linkers have received extensive attention in recent years [23–26]. Owing to their ordered porous structures, large specific surface areas and certain chemical and thermal stability, MOFs were widely used in heterogeneous catalysis, gas storage and separation, drug delivery and so on [27-32]. Especially, there are several typical MOFs with excellent performance and wide application, such as MIL-101(Cr) [33], UiO-66(Zr) [34], HKUST-1(Cu) [35], ZIF-8(Zn) [36]. All of them are common carriers for Pd nanoparticles. Pd@HKUST-1 is a ideal bifunctional heterogeneous catalyst that both palladium nanoparticles and copper ions are evenly and stably distributed on it [31,37]. Porous structure materials often have better catalytic performance, because the catalysts with porous structure have larger contact areas with the reactants, more exposed catalytic sites, and certain adsorption, as the same conclusion of Chen and Hou et al. [38,39].

Tandem reactions catalyzed by metal organic frameworks have been reported widely [40-43]. Based on this, we have designed a new homocoupling route of phenylboronic acid, using Pd@HKUST-1 as a bifunctional heterogeneous catalyst. This route is divided into two steps.

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Scheme 1. Reaction route.



Fig. 1. PXRD patterns of simulated (black), HKUST-1 (magenta), Pd@ HKUST-1 (red). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

The first step is Cu(II) to catalyze one part of phenylboronic acid with I_2 to synthesize iodobenzene. The second step is Pd(0) to catalyze the another part of phenylboronic acid coupled with iodobenzene to synthesize biphenyl. Both of them are catalyzed by the same heterogeneous catalyst. Compared to the conventional homogeneous catalysis homocoupling reaction of phenylboronic acids, this strategy for forming symmetric C-C bond between benzene rings has obvious advantages of separating catalysts. Compared to the Suzuki reaction to form symmetric C-C bond between rings, this strategy has advantages of no

adding toxic halogenated benzene (see Scheme 1).

HKUST-1 was synthesized by the hydrothermal method. Pd@HKUST-1 was prepared via a simple capillary impregnationreduction method. The catalyst has been characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscopy (TEM), energy-dispersive spectra (EDS), thermogravimetric analysis (TGA). The specific surface areas and pore volumes of the catalyst were determined by N₂ physisorption measurements. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) was used to determine the content of Pd in Pd@HKUST-1. The conversions and yields of the catalytic reactions were analyzed by GC. Proton NMR spectra (¹H NMR, 300 MHz, CDCl₃) was used to further verify the structure of the tandem reaction product.

2. Experimental

The experimental section is described in detail in Supplementary information.

3. Results and discussion

3.1. Characterization of catalysts

The powder XRD patterns of HKUST-1 and Pd@HKUST-1 are displayed in Fig. 1. The PXRD pattern of as-synthesized HKUST-1 matched well with the already reported PXRD patterns [44], which suggested that HKUST-1 was obtained. After loading of Palladium nanoparticles, there was no apparent loss of crystallinity in PXRD patterns (Fig. 1), which demonstrated that the frameworks of HKUST-1 are mostly maintained, similar to the results reported previously [45]. Scanning electron microscope (SEM) was performed to observe the morphologies of Pd@HKUST-1 before and after catalysis. As shown in Fig. 2 (a) and (b),



Fig. 2. (a) SEM image of as-synthesized Pd@HKUST-1 before catalysis reaction; (b) SEM image of Pd@HKUST-1 after catalysis reaction.



Fig. 3. (a) TEM image of as-synthesized Pd@HKUST-1 before catalysis reaction; (b) corresponding size distribution of Pd nanoparticles before catalysis reaction; (c) EM image of Pd@HKUST-1 after catalysis reaction; (d) corresponding size distribution of Pd nanoparticles after catalysis reaction.



Fig. 4. (a) $\rm N_2$ adsorption isotherms at 77 K of as-synthesized HKUST-1 and Pd@HKUST-1. (b) Pore size distribution of as-synthesized HKUST-1 and Pd@HKUST-1.

crystals of regular octahedron could be observed before and after catalysis, indicating that the structure of Pd@HKUST-1 was stable. Transmission electron microscopy (TEM) was performed to observe the loading of palladium nanoparticles. Fig. 3 (a) demonstrated that palladium nanoparticles were successfully uniformly loaded on HKUST-1. Fig. 3 (c) showed that palladium nanoparticles were stable after catalysis. Fig. 3 (b) and (d) showed that the size of palladium nanoparticles was mainly at \sim 1.8 nm before and after catalysis. As shown in Fig. S1 the EDS further confirmed the presence of Cu and Pd in the catalyst. The loading amount of Pd was 0.97 wt% determined by ICP-AES. N2 adsorption isotherms and pore size distribution of as-synthesized HKUST-1 and Pd@HKUST-1 are showed in Fig. 4. The N₂ sorption properties of samples was analyzed by the Brunauer-Emmett-Teller (BET) method at 77 K. It showed that HKUST-1 has a large BET specific surface area of 971 m^2/g . After the Pd on the load, the BET specific surface area is significantly reduced to 461 m²/g. The total pores volumes of HKUST-1 and Pd@HKUST-1 are 0.54 mL/g and 0.41 mL/g, respectively. The average pores diameters (4 V/A by BET) of HKUST-1 and Pd@HKUST-1 are 2.2 nm and 3.6 nm, respectively. In addition, as shown in Fig. S2, TG analysis showed that the catalysts were of high thermal stability below 350 °C. After the catalyst was cycled five times, the reaction supernatant was taken and no leakage of Pd was detected by ICP-AES.



Fig. 5. Product analysis of phenylboronic acid reaction. (The solvent was acetonitrile and the temperature was 343 K.)

Table 1

Reaction of different conditions.

	$B(OH)_2$ + I_2 Cataly	st C	∕I + [B(OH)2	Catalyst	
1a	2	3a		1a		4a
Entry	catalyst	Base	T(°C)	solvent	Yield ^{3a} (%)	Yield ^{4a} (%)
1	Cu(NO3)2·3H2O	/	60	acetonitrile	84.2	0
2	HKUST-1	/	60	acetonitrile	7.1	0
3	Cu(NO ₃) ₂ ·3H ₂ O	K ₂ CO ₃	60	acetonitrile	99.9	0
4	HKUST-1	K ₂ CO ₃	60	acetonitrile	42.5	0
5	None	K ₂ CO ₃	60	acetonitrile	25.1	0
6	Pd@HKUST-1	/	60	acetonitrile	16.7	0
7	Pd@HKUST-1	K ₂ CO ₃	60	acetonitrile	59.3	26.3
8	Pd@HKUST-1	K ₂ CO ₃	70	acetonitrile	78.4	20.6
9	Pd@HKUST-1	K ₂ CO ₃	70	Ethanol	18.5	80.5
10	Pd@HKUST-1	K ₂ CO ₃	70	DMF	55.1	25.4
11	Pd@HKUST-1	K ₂ CO ₃	70	DMSO	56.5	42.6
12	Pd@HKUST-1	K_2CO_3	70	Toluene	17.2	81.8
13 ⁿ	Pd@HKUST-1	K_2CO_3	70	Ethanol	0	5.4

Reaction conditions: 1a (2 mmol phenylboronic acid), 2 (0.5 mmol I_2), ⁿ (no I_2), catalyst, K_2CO_3 (0.7 mmol), solvent (5 mL), N_2 (1 bar), 7 h, Yields was determined by GC with internal standard (1 mmol n-dodecane).

3.2. Catalytic tests

A series of catalytic tests were carried out to explore the best conditions for this tandem reaction. As shown in Fig. 5, in a typical phenylboronic acid reaction it was detected that the yield of iodobenzene increased at first and then decreased, meanwhile the yield of biphenyl gradually increased. The step of phenylboronic acid to iodobenzene was the focus of discussion. Firstly, consistent with previous reports [24,28], copper ions had good catalytic activity (Entry. 1, Table 1) as a homogeneous catalyst. The mechanism of the iodination reaction of phenylboronic acid catalyzed by Cu(II) is illustrated in Scheme 2 [46]. HKUST-1 as a Cu-MOF had poor catalytic effect (Entry. 2, Table 1), maybe because of steric hindrance. However, when the Suzuki reaction cocatalyst inorganic base was added, the catalytic effects of copper ions and HKUST-1 were both improved (Entry. 3 and 4, Table 1). Under individual effect of the inorganic base, somewaht iodobenzene was formed (Entry. 5, Table 1). Pd@HKUST-1 had better catalytic efficiency than HKUST-1 (Entry. 6 and 2, Table 1). When all factors including Pd, Cu-MOFs and inorganic bases were combined, the catalyst could achieve the conversion of a large amount of phenylboronic acid (Entry. 7,



Scheme 2. Mechanism of Cu catalyzed iodination reaction of phenylboronic acid.



Scheme 3. Mechanism of Pd catalyzed Suzuki coupling reaction.

Table 1), but the conversion of phenylboronic acid had not reached 100%. So increasing the temperature from 60 °C to 70 °C, and it was monitored that the conversion of phenylboronic acid reached almost 100%. (Entry. 8, Table 1) Different solvents were tested, and ethanol and toluene were more conducive to the formation of biphenyl among the solvents investigated (Entry. 8–12, Table 1). Besides, in the absence of iodine, homocoupling reaction of phenylboronic acid catalyzed by Pd@HKUST-1 had a very low yield (Entry. 13, Table 1). The mechanism of the Suzuki reaction see Scheme 3.

Under the same conditions, yields of iodobenzenes and biphenyls with different groups at 7 h were displayed. Ethanol was selected as an efficient and environmentally friendly organic solvent for the tandem reaction. Almost all phenylboronic acids with different groups smoothly reacted to provide the desired products. For unsubstituted phenylboronic acid, the yield of biphenyl was 81% (Entry. 1, Table 2). Firstly, the case of electron-withdrawing group was discussed. The yield of 2,2-difluorodiphenyl acid was 69% (Entry. 2, Table 2). When the group of meta- phenylboronic acids was a -F or -Cl, the yields of products were raised to 91.4% and 92.8% (Entry. 3 and 5, Table 2). When the group of

Table 2

	R + I_2 1 2	$\frac{Pd@HKUST-1}{R} + R + R + R + R + R + R + R + R + R +$	$\frac{Pd@HKUST-1}{R} \xrightarrow{4}$		
Entry	Reactant	Product	Yield ³ (%)	Yield ⁴ (%)	
1	B(OH) ₂ la		18.0	81.0	
2	F 1b	F 4a	30.1	68.9	
3	F lc	F 4b	7.6	91.4	
4	F B(OH) ₂	F 4c	9.7	89.3	
5	B(OH) ₂ CI le	F 4d	6.2	92.8	
6	ct B(OH) ₂		7.7	91.3	
7	B(OH) ₂ lg	dr 4f	17.4	15.3	
8	B(OH) ₂ lh	4g	40.6	58.4	
9	B(OH) ₂ li	4h	22.7	76.3	
10	B(OH) ₂ lj	4i	71.0	28.0	
11	B(OH) ₂	4j	50.8	48.2	

Reaction conditions: 1 (2 mmol phenylboronic acid), 2 (0.5 mmol elemental iodine), catalyst (1 mmol % Pd), base (0.7 mmol), solvent (5 mL ethanol), N_2 (1 bar), 70 °C, 7 h, Yields was determined by GC with internal standard (1 mmol n-dodecane).



Fig. 6. The recyclability studies of Pd@HKUST-1 in tandem reactions. Reactant was 3-chlorophenylboronic acid, and yield of biaryl compounds was showed.

para-phenylboronic acids was a -F or -Cl, the yields of products were raised to 89.3% and 91.3% (Entry. 4 and 6, Table 2). Therefore phenylboronic acids with electron-withdrawing groups had higher product vield, except ortho-substituted 2-fluorobenzeneboronic acid probably because of steric hindrance effect. Then, the case of electron-donating group was discussed. The conversion of 2-methylbenzeneboronic acid and the yield of its product were very poor (Entry. 7, Table 2). When the group of *meta*-phenylboronic acids was a methyl or phenyl, the yields of products were reduced to 58.4% and 28.0% (Entry. 8 and 10, Table 2). When the group of *para*-phenylboronic acids was a methyl or phenyl, the yields of products were reduced to 76.3% and 48.2% (Entry. 9 and 11, Table 2) Therefore phenylboronic acids with electron-donating groups had lower product selectivity, probably because electron-donating groups had negative effects on the the Suzuki reaction [47,48]. In summary, group of phenylboronic acids was an electron-withdrawing group which was beneficial to the formation of biaryl compounds, and vice versa. And different groups has greater steric hindrance at the ortho position than at the meta and para position. The result is consistent with Tang et al. [49].

3-chlorophenylboronic acid was selected to conduct recycle tests and the yields of its products are shown in Fig. 6. The catalyst of Pd@HKUST-1 retained high catalytic activity after 5 cycles of reuse. As shown in Fig. 1, PXRD patterns indicated the structure of Pd@HKUST-1 was retained after 5 runs. It is proved that the bifunctional heterogeneous catalyst is recyclable.

4. Conclusions

An Iodination-Suzuki tandem reaction was developed to accelerate homocoupling reactions of phenylboronic acids. The tandem reaction was catalyzed by a bifunctional heterogeneous catalyst of Pd@HKUST-1, which has excellent performances because of special porous structure and high dispersion. The catalyst demonstrated high efficiency, especially when solution was ethanol at 70 °C under N₂ atmosphere for 7 h. Moreover, the ortho position of phenylboronic acids with any groups is not conducive to the tandem reaction because of steric hindrance. Electron-withdrawing group at the meta and para position was beneficial to the formation of biaryl compounds, and vice versa. The catalyst of Pd@HKUST-1 show well recyclability after reusing 5 times, indicating its potential practical application.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2020.108368.

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H. Tang et al.

Inorganic Chemistry Communications 123 (2021) 108368

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