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Base-Free Suzuki–Miyaura Coupling Reaction Using Palladium(II) Supported Catalyst in Water

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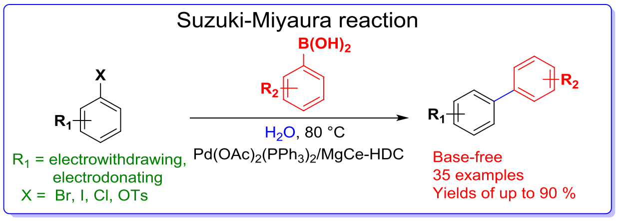
Abstract

The carbon–carbon bond formation via Suzuki–Miyaura reaction was performed in water as green solvent. $Pd(OAc)_2(PPh_3)_2$ supported on magnesium hydroxide and cerium carbonate hydroxide composite was prepared and characterized by various techniques. The cross-coupling reaction of aryl halides carried out in water using mild conditions. The effects of temperature, solvents, the amount of catalyst and leaving groups were studied to find the optimization conditions for cross-coupling reaction. Various aryl halides were smoothly transformed into the biaryls in good yields. In addition, the catalyst also exhibited stability and catalytic performance in the cross-coupling of aryl halides.

Graphical Abstract



A new approach is developed for carbon–carbon bond formation via Suzuki–Miyaura reaction.$_2$ $Pd(OAc)_2(PPh_3)_2$ supported on mixed magnesium hydroxide and cerium carbonate hydroxide were prepared and characterized by XRD, XPS, SEM–EDX techniques. The cross-coupling reaction of aryl halides can be carried out in water and under mild conditions (80 °C).

Keywords Base-free · Suzuki–Miyaura coupling · Water as solvent · Pd(II) catalyzed · Magnesium hydroxide and cerium carbonate hydroxide composite

1 Introduction

The carbon–carbon bond formation is a very interesting topic in organic chemistry. The Suzuki–Miyaura reaction is one of the important reactions in C–C cross coupling reaction [1]. Symmetrical and unsymmetrical biaryl products of an arylboronic acid and aryl halides have good applications in various areas like preparation of materials, natural products, bioactive molecules, herbicides, antifungal, anti-inflammatory, antirheumatic, antitumor, and antihypertensive agents and conducting polymers [2].

Therefore, Suzuki–Miyaura reaction has attracted researchers for development of new methodologies and catalytic routes. On the other hand, it usually proceeds by

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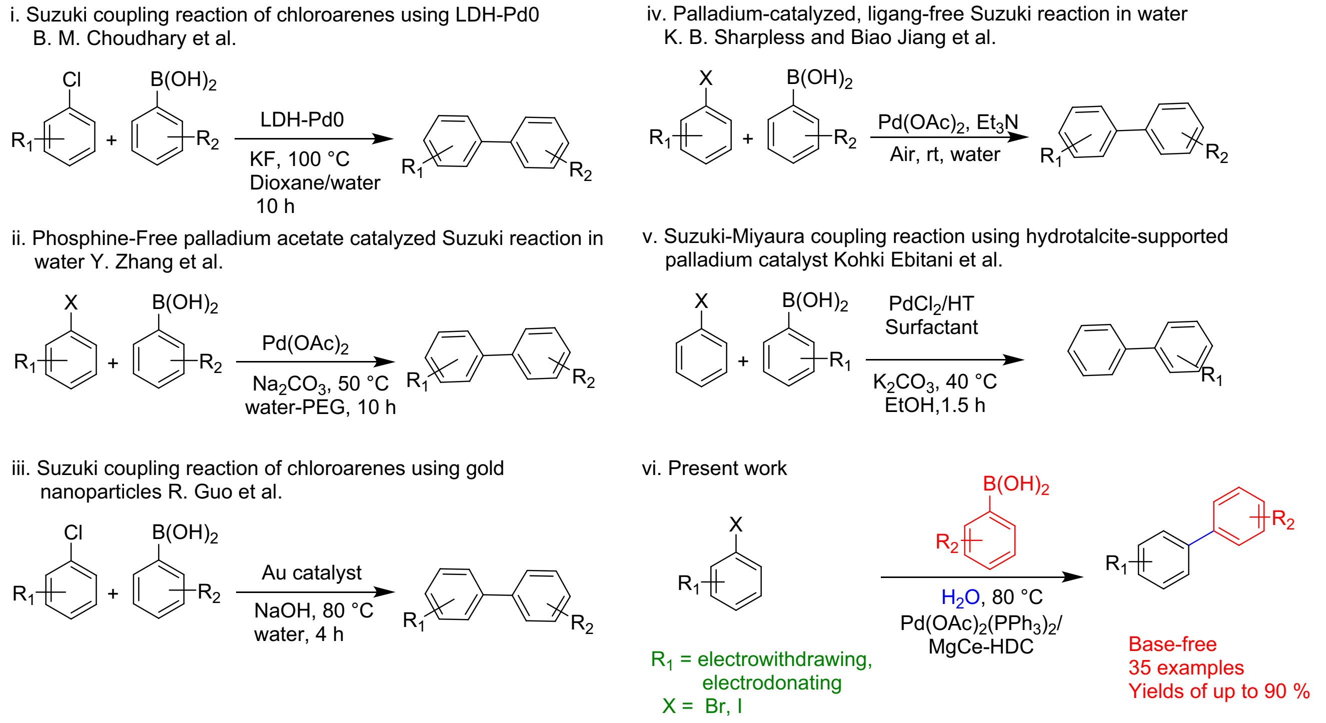
using phosphine-based palladium catalysts, palladium nanoparticles [3–5], microwave technology [6–11], nucleophilic carbene ligands [12–15], ionic liquids [16–21], palladium supported on various species like carbon [22–25], silica [26], zeolites [27], metal–organic frameworks [28], and so on [29, 30]. Choudary and co-workers [31] had reported Suzuki coupling reaction using a nano-palladium catalyst with KF as the base (Scheme 1a). Zhang et al. [32] had also reported phosphine-free Suzuki–Miyaura reaction in water with sodium carbonate, while Biao Jiang et al. [33] showed ligand-free Suzuki–Miyaura reaction in water using triethylamine as base. Recently, Kohki Ebitani and co-workers [34] demonstrated Suzuki–Miyaura coupling using hydrotalcite-supported palladium with potassium carbonate. These methods have used homogeneous bases and various solvents which increase the cost of production. Therefore, any methodology or catalytic route which eliminates the homogenous base and can be performed in the greener solvent, then cost of production will be low.

Palladium(II) salts supported catalysts have been reported many times for different organic transformations like oxidation of alcohols [35, 36], Heck reaction [37–40], Suzuki–Miyaura reaction [41], Sonogashira reaction [42], and so on [43]. Generally, Suzuki–Miyura reaction was reported in DMF [10], aq. DMF [44], Toluene–water [45], aq. EtOH [46], dioxane [47], dioxane–water [31], and so on [32, 34, 48]. For green approaches, Suzuki–Miyaura reaction protocols need some modifications using water as solvents, heterogeneous catalyst, base-free condition.

Recently, magnesium hydroxide and cerium carbonate hydroxide composite eliminated the homogenous base for Henry reaction [49]. We have an ongoing research on the development of a new solid base catalyst for base-free reaction at our laboratory [50–53]. In this study, we have extended the scope of solid base catalyst for greener approach for base-free Suzuki–Miyaura reaction. From green and substantial chemistry points of view, for Suzuki–Miyaura reaction, we have designed our basic aim for the reaction to develop a new catalytic system which employs (i) heterogeneous Pd-salt catalyst, (ii) without any homogenous base, and (iii) without air or oxygen. To realize this aim, $Pd(OAc)_2(PPh_3)_2$ supported on mixed magnesium hydroxide and cerium carbonate hydroxide $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC was synthesized [54], which acted as efficient catalyst for the cross-coupling of aryl halides and phenylboronic acids to obtain the desired symmetric or unsymmetric biaryls at 80 °C under base-free aqueous condition. To the best of our knowledge, it is the first report about the application of MgCe-HDC for Suzuki–Miyaura reaction.

The catalyst was characterized by X-ray diffraction (XRD) patterns (see Figure S1, in the Supporting Information) confirming the phase of magnesium hydroxide and cerium carbonate hydroxide in Pd(II) supported catalyst. Figure S3

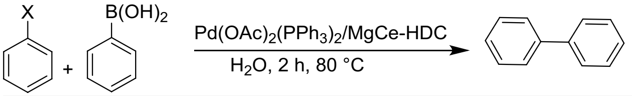


Scheme 1 Previous reports versus present work

(Supporting Information) showed the XRD pattern of catalyst after 5th run. SEM–EDX was also performed and shown in Figure S2(i) (see Supporting Information). Figure S2(i) represents: (a) low magnified SEM images of the MgCe-HDC (b) high magnified SEM images of the MgCe-HDC, (c) high magnified SEM image of $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC and (d–i) elemental mapping images of six elements: (d) C K edge, (e) Ce L edge, (f) Mg K edge, (g) O K edge, (h) P K edge, (i) Pd L edge. Figure S2(ii) showed the SEM image of catalyst after 5th run. We can conclude that palladium (II) species distributed in uniform patterns does not alter the porosity of material after loading of Pd(II) species. Figure S4 (see Supporting Information) represents the XPS spectrum of $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC and MgCe-HDC. As shown in Figure S4 (Supporting Information), the signals centered at about 332.2 and 337.4 eV for supported Pd(II) on MgCe-HDC as assigned to Pd 3d 5/2 and Pd 3d 3/2 regions, respectively, indicative of the existence of Pd(II) species [55]. The binding energy values 130, 282.3, 286.3 and 530 correspond to P 2p, C 1s(C–C), C 1s(C–O) and O 1s spectrum [56–59]. Figure S10 (see Supporting Information) represents the XPS spectrum of $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC after 5th Run. The TGA (Figure S9, Supporting Information) analysis shows a two-step weight loss in the material. The first weight loss (7.13 wt% and 0.2638 mg) corresponds to the decomposition of $Mg(OH)_2$ to MgO and water, whereas the second weight loss (14.51 wt% and 0.4985 mg) indicates a loss of water and carbon dioxide from $CeCO_3OH$. The pH of catalyst in water was 10.44.

$Pd(OAc)_2(PPh_3)_2$/MgCe-HDC was employed and compared with a variety of partners, including aryl halides (iodo-, chloro-, fluoro-) and aryl tosylate for phenylboronic acid in the Suzuki–Miyaura reaction (Table 1). The aryl iodide was found to react smoothly with phenylboronic acid while aryl bromide and aryl tosylate have shown moderate reactive behavior (Table 1, entries 1–3).

Table 1 Performance of different leaving groups



Entry	Leaving group (X)	Yield[a] (%)
1	I	90.8
2	Br	55.5
3	OTs	24.6
4	Cl	19.5

Reactions conditions: aryl-X (0.5 mmol), phenyl boronic acid (0.6 mmol), H_2O (4 mL), catalyst (100 mg), 80 °C, 2 h

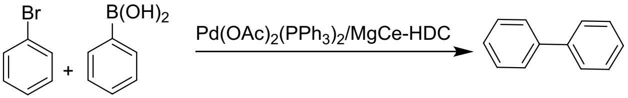
[a]Isolated yield

The aryl chloride has shown less reactivity towards phenylboronic acid in this simple catalytic system at 80 °C without other additives in pure water as a solvent (Table 1, entry 4). Thus, $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC can be used as cheap and greener catalyst for aryl halide in the Suzuki–Miyura reaction in water.

Inspired by these stimulating results, various reaction parameters, including the amount of catalyst, temperature, and solvents, were screened broadly order to optimize the reaction condition for the coupling of boronic acid and aryl bromide. For solvents optimization, we have carried out three solvents i.e. DMF, DMSO, and water in present work (Table 2, entries 1, 2 and 4). The aryl bromide showed better yield with phenylboronic acid in water as compared to other solvents. Next, the amount of catalyst for cross-coupling reaction was investigated (Table 1). When the catalyst was increased to 100 mg, biaryl was obtained with an isolated yield of 55.5% after reaction for 2 h. by increasing the amount of catalyst to 200 and 300 mg, only 38.4 and 32.9% yield of biaryl were obtained (Table 2, entries 5 and 6). Even with a very low amount of catalyst (50 mg), a 20% of the yield of biaryl was still obtained (Table 2, entry 50). Effect of temperature was observed from room temperature to 373 K (Table 2). The yield first increased till 353 K; after 353 K, the yields gradually decreased till 373 K. At 343 and 353 K, aryl bromide was coupled with phenylboronic acid in quantitative yield (Table 2, entries 4 and 9).

With the optimal conditions in hand, the reaction conditions were then applied to the coupling of a variety of the substrates (Table 3). The electron-rich and electron-withdrawing groups containing boronic acids were coupled with

Table 2 Optimization of the reaction conditions for Suzuki reaction

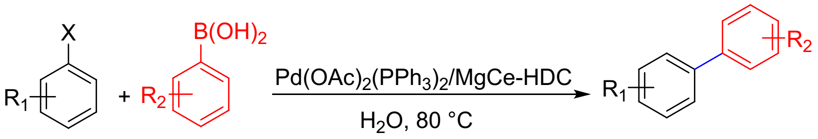


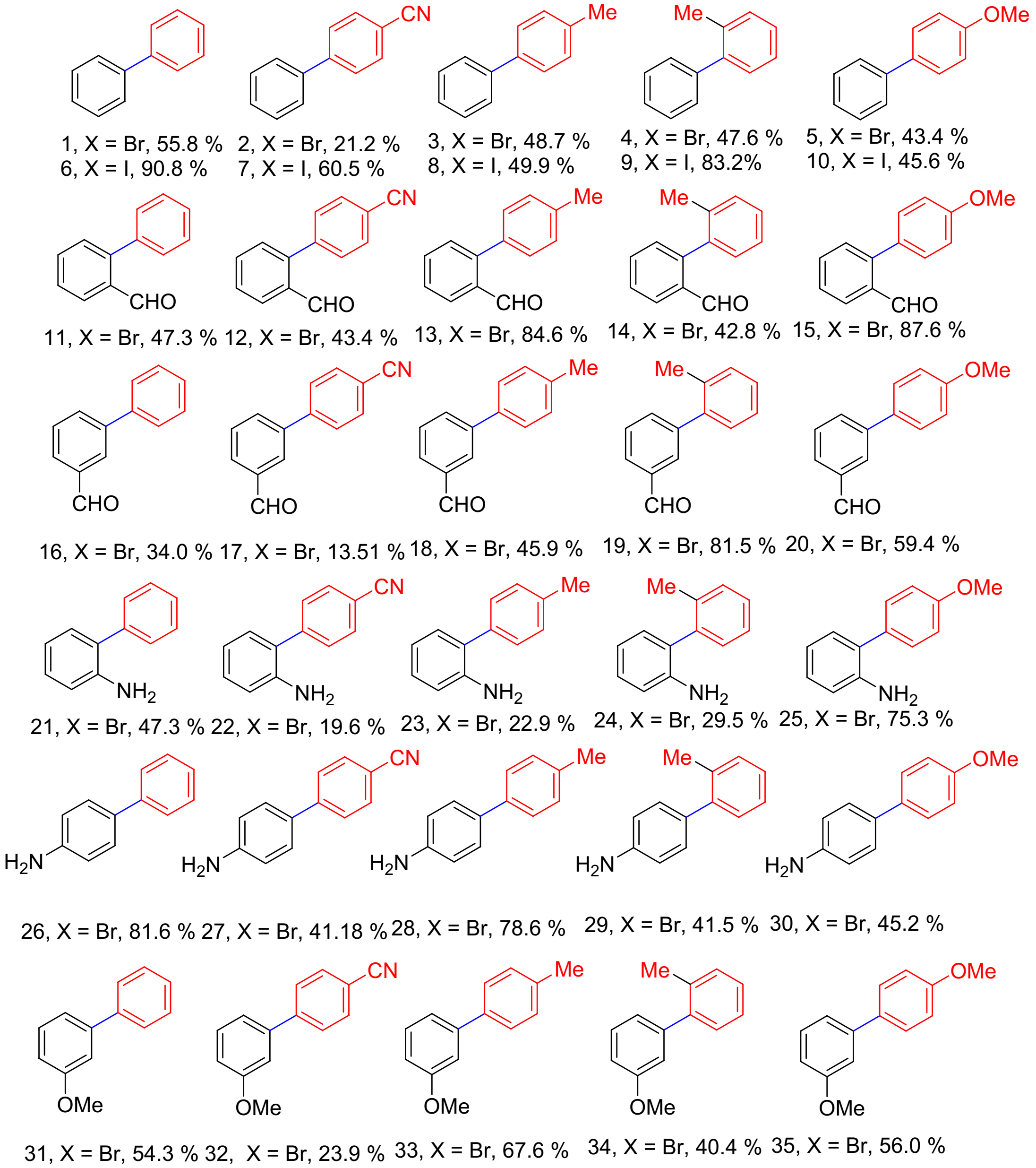
Entry	Solvent	Catalyst (mg)	Temperature (K)	Yield[a] (%)
1	DMF	100	353	33.7
2	DMSO	100	353	25.9
3	H_2O	50	353	20.8
4	H_2O	100	353	55.8
5	H_2O	200	353	38.4
6	H_2O	300	353	32.9
7	H_2O	100	rt	36.3
8	H_2O	100	333	38.9
9	H_2O	100	343	45.4
10	H_2O	100	363	25.9
11	H_2O	100	373	23.3

Reactions conditions: aryl-X (0.5 mmol), phenyl boronic acid (0.6 mmol), solvent (4 mL), 2 h

[a]Isolated yield

Table 3 Suzuki–Miyaura reaction of aryl halide derivatives and boronic acids





1, X = Br, 55.8 %
6, X = I, 90.8 %
2, X = Br, 21.2 %
7, X = I, 60.5 %
3, X = Br, 48.7 %
8, X = I, 49.9 %
4, X = Br, 47.6 %
9, X = I, 83.2%
5, X = Br, 43.4 %
10, X = I, 45.6 %

11, X = Br, 47.3 % 12, X = Br, 43.4 % 13, X = Br, 84.6 % 14, X = Br, 42.8 % 15, X = Br, 87.6 %

16, X = Br, 34.0 % 17, X = Br, 13.51 % 18, X = Br, 45.9 % 19, X = Br, 81.5 % 20, X = Br, 59.4 %

21, X = Br, 47.3 % 22, X = Br, 19.6 % 23, X = Br, 22.9 % 24, X = Br, 29.5 % 25, X = Br, 75.3 %

26, X = Br, 81.6 % 27, X = Br, 41.18 % 28, X = Br, 78.6 % 29, X = Br, 41.5 % 30, X = Br, 45.2 %

31, X = Br, 54.3 % 32, X = Br, 23.9 % 33, X = Br, 67.6 % 34, X = Br, 40.4 % 35, X = Br, 56.0 %

Reactions conditions: aryl-X (0.5 mmol), boronic acid derivatives (0.6 mmol), H_2O (4 mL), catalyst (100 mg), 80 °C, 2 h

Isolated yield

different aryl halides derivatives using $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC in water at 80 °C for 2 h. The iodobenzene was coupled with phenylboronic acid, 4-cyanoboronic acid, 4-methylboronic acid, 4-methoxyboronic acid, and 2-Methylboronic acid (Table 3, entries 1–5). The iodobenzene seemed to be less reactive towards 4-methyl, and 2-methoxy with yields of 49.9% and 45.6% as compared to other boronic acids. The bromobenzene was tolerated well and engaged in reactions with these boronic acids except 4-cyanoborononic acid. Electron-rich group at *para*-position encouraged the reaction with 2-bromobenzaldehyde while –CN, –H, and –Me showed yield range of 42.0–47.3%. 3-Bromobenzaldehyde also coupled with boronic acids, out of them cross-coupling 2-methylboronic acid gave a good yield.

4-Cyanoboronic acid was less compatible with the optimal reaction conditions. 2-bromoaniline gave moderate yield with phenylboronic acid. On the other hand, it gave effective yield with 4-methoxyboronic acid while it gave comparatively low yield with cyano-, methyl- group containing boronic acids. However, surprisingly these results were opposite when 4-bromoaniline coupled with boronic acids. Phenylboronic acid and 4-methylboronic acid gave good yield with 4-bromoaniline. 3-Methoxyaniline also coupled with five boronic acids, except 4-cynoboronic acid other all have moderate yields. In these cases, however, biphenyl was formed as a byproduct from self- coupling of phenyl boronic acid as confirmed by ^{1}H-NMR (Table 3, entries 11, 21, and 31).

To examine the scope of recycling of the heterogeneous catalyst, the catalyst was used 5 times with no loss in catalytic activity as shown in Figure S11 (see Supporting Information). The catalytic activity, however, decreased after the 2nd run, which could either be due to temporary poisoning by organic impurities or loss in recovery of the catalyst material.

In conclusion, we developed a simple, efficient procedure for the Suzuki–Miyaura cross-coupling in water. $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC catalyst was prepared and investigated in the synthesis of symmetrical or unsymmetrical biaryls under the base-free condition without using any promoter. Furthermore, it can acquire good to excellent yields (up to 90.8%) with a wide substrate scope (35 examples). This methodology can be extended to the synthesis of nitrogen, oxygen-based heterobiaryls. Further applications of these base-free systems in other transformations are currently under examination in our laboratory.

2 Experimental Section

2.1 Preparation of Mixed Magnesium Hydroxide and Cerium Carbonate Hydroxide

A typical preparation procedure for mixed magnesium hydroxide and cerium carbonate hydroxide, using our previous method [49], is as follows: $Na_2CO_3 \cdot 10H_2O$ (30 mmol, 8.6 g), NaOH (70 mmol, 2.8 g) and water (60 mL) were taken into a 200 mL beaker. $Mg(NO_3)_2 \cdot 6H_2O$ (30 mmol), $Ce(NO_3)_3 \cdot 6H_2O$ (10 mmol) and water (100 mL) were added in another 200 mL beaker salts were let dissolve completely and dropped into the 200 mL of beaker while pH was kept to be 10. Gel was obtained and aged for 4 h at 338 K, filtered and washed with water (~4 L), thoroughly up to neutral pH. The obtained semi-solid was dried in an oven overnight at 373 K then solid was crushed to convert it to powder form.

2.2 Preparation of Supported $Pd(OAc)_2(PPh_3)_2$ Over Mixed Magnesium Hydroxide and Cerium Carbonate Hydroxide Composite

$Pd(OAc)_2(PPh_3)_2$ supported on mixed magnesium hydroxide and cerium carbonate hydroxide ($Pd(OAc)_2(PPh_3)_2$/MgCe-HDC) were prepared according to literature with a modified procedure [54] (synthesis of $Pd(OAc)_2$ on hydrotalcite). A typical preparation procedure is as follows: 112 mg of $Pd(OAc)_2$ and 216 mg of PPh_3 were dissolved in 10 mL of toluene. 1 g of magnesium hydroxide and cerium carbonate hydroxide were added to this solution and the mixture was stirred at 80 °C for 1 h. After the reaction, the solution was filtered and washed with toluene (40 mL). The residue was dried at 100 °C for 30 min and finally grained to obtain the desired $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC.

2.3 General Procedure for Suzuki Reaction

A typical procedure for Suzuki reaction is as follows: aryl halide (0.5 mmol) was weighed in 25 mL round bottom flask containing boronic acids (0.6 mmol), and $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC (100 mg). The reactants and catalyst were dispersed in water (4 mL), the RB was mounted on an oil bath at 353 K and the mixture was stirred for 2 h. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the catalyst was separated by filtration. Then the solution was extracted with ethyl acetate and brine solution. The organic phase was dried over anhydrous Na_2SO_4 and concentrated under vacuum. The crude product was purified by column chromatography on silica gel to afford pure product. The pure product was analyzed using ^{1}H- and ^{13}C-NMR techniques and compared with literature NMR data.

2.4 General Procedure for Reuse of the Catalyst

First, run of the Suzuki reaction catalyzed by $Pd(OAc)_2(PPh_3)_2$/MgCe-HDC was performed by the above-described procedure. Recovered catalyst was washed with toluene (20 mL) and water (20 mL). Afterwards, the catalyst was dried at 100 °C for 30 min and the obtained catalyst was ready for reuse.

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