The first heterogeneous carbonylative Sonogashira coupling reaction catalyzed by MCM-41-supported bidentate phosphine palladium(0) complex

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ABSTRACT

The first heterogeneous carbonylative Sonogashira coupling reaction of terminal alkynes with aryl iodides under atmospheric pressure of carbon monoxide was achieved in the presence of a catalytic amount of MCM-41-supported bidentate phosphine palladium(0) complex [MCM-41-2P-Pd(0)] in good to high yields. This polymeric palladium catalyst can be reused at least 10 times without any decrease in activity.

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1. Introduction

Synthesis of α,β-alkynyl ketones has attracted considerable interest because of their appearance in a wide variety of biologically active molecules [1] and their utility as synthetic intermediates, particularly for the synthesis of natural products [2–4] and pharmaceutical molecules [5–8]. A common route to α,β-alkynyl ketone involves the acylation of alkynyl organometallic reagents such as silver [9], copper [10], lithium [11], zinc [12], silicon [13], and tin [14] with acid chlorides. The transition metal-catalyzed coupling reaction of alkynes or their metalated derivatives such as alkynyl-stannanes [15,16] and alkynylsilanes [17] with organic halides in the presence of CO provides an alternative approach to the synthesis of α,β-alkynyl ketones under atmospheric conditions with atom economy. Mori and co-worker [18] reported that the palladium-catalyzed carbonylative Sonogashira coupling of terminal alkynes with aryl iodides under atmospheric pressure of carbon monoxide was accomplished by using aqueous ammonia as a base in THF at room temperature with or without CuI. Recently, Yang and co-workers [19] reported that the palladium-catalyzed copper-free carbonylative Sonogashira coupling of terminal alkynes with aryl iodides under atmospheric pressure of carbon monoxide was

achieved by using water as a solvent at room temperature with Et₃N as a base. Although the palladium-catalyzed carbonylative Sonogashira coupling of terminal alkynes with aryl iodides under atmospheric pressure of carbon monoxide has provided a convenient route to α,β-alkynyl ketones, the reaction generally proceeds in the presence of a homogeneous palladium catalyst such as PdCl₂(PPh₃)₂, which makes the recovery of the metal tedious if not impossible and might result in unacceptable palladium contamination of the product. From the standpoint of green chemistry, the development of more environmentally benign conditions for the reaction such as the use of a heterogeneous palladium catalyst would be desirable [20–23].

So far, polymer-supported palladium catalysts have successfully been used for the Heck reaction, the Suzuki reaction, the Sonogashira reaction, etc. [24]. However, to the best of our knowledge, there has been no general study of the carbonylative Sonogashira coupling reaction catalyzed by polymer-supported palladium complex catalyst described to date. Recent developments on the mesoporous material MCM-41 provided a new possible candidate for a solid support for immobilization of homogeneous catalysts [25]. MCM-41 has a uniform pore diameter of *ca.*5 nm and a specific surface area >700 m² g⁻¹ [26]. Its large pore size allows passage of large molecules such as organic reactants and metal complexes through the pores to reach to the surface of the channel [27–29]. To date, a few palladium complexes on functionalized MCM-41 support have been prepared and used in organic reactions [30–34]. In this

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paper, we wish to report the synthesis of the MCM-41-supported bidentate phosphine palladium(0) complex [abbreviated as MCM-41-2P-Pd(0)] and its catalytic properties in the carbonylative Sonogashira coupling reaction of terminal alkynes with aryl iodides under atmospheric pressure of carbon monoxide.

2. Experimental

All chemicals were reagent grade and used as purchased. The mesoporous material MCM-41 was prepared according to the literature procedure [35]. All carbonylative coupling products were characterized by comparison of their spectra and physical data with authentic samples. IR spectra were determined on a PerkinElmer 683 instrument. ^{1}H NMR spectra were recorded on a Bruker AC-P400 (400 MHz) spectrometer with TMS as an internal standard in CDCl$_3$ as solvent. ^{13}C NMR spectra were recorded on a Bruker AC-P400 (100 MHz) spectrometer in CDCl$_3$ as solvent. X-ray powder diffraction patterns were obtained on a Damx-rA diffractometer (Rigaku). X-ray photoelectron spectra were recorded on a Kratos XSAM 800 spectrometer.

2.1. Preparation of MCM-41-NH$_2$

A solution of γ-aminopropyltriethoxysilane (2.20 g, 10 mmol) in dry chloroform (18 ml) was added to a suspension of the mesoporous support MCM-41 (2.80 g) in dry toluene (180 ml). The mixture was stirred for 48 h at 100 °C. Then the solid was filtered and washed by CHCl$_3$ (2 ml × 20 ml), and dried in vacuum at 160 °C for 5 h. The dried white solid was then soaked in a solution of Me$_3$SiCl (4.36 g, 40 mmol) in dry toluene (150 ml) at room temperature under stirring for 24 h. Then the solid was filtered, washed with acetone (3 ml × 20 ml) and diethyl ether (3 ml × 20 ml), and dried in vacuum at 120 °C for 5 h to give 3.54 g of hybrid material MCM-41-NH$_2$. The nitrogen content was found to be 1.27 mmol/g by elemental analysis.

2.2. Preparation of MCM-41-2P

A Schlenk flask was charged with paraformaldehyde (0.701 g, 23.3 mmol), dry MeOH (20 ml) and diphenylphosphine (4.340 g, 23.3 mmol). The reaction mixture was heated to 60 °C under Ar until the white suspension formed a colorless solution. After removal of MeOH in vacuo the remaining viscous oil was diluted in dry toluene (20 ml). This solution was added to a suspension of MCM-41-NH$_2$ (3.020 g) in dry toluene (60 ml) and the reaction mixture was heated to 105 °C under Ar for 24 h. In the cooler regions of the flask the water–toluene azeotrope separated indicating the reaction progress. After cooling to room temperature the solid product was collected by filtration under Ar, washed with dry toluene (4 ml × 30 ml), CH$_2$Cl$_2$/THF (1/1) (2 ml × 30 ml), CH$_2$Cl$_2$ (2 ml × 30 ml) and dried in vacuo (100 °C) for 5 h to give 4.08 g of the light yellow MCM-41-2P. The nitrogen and phosphine content was found to be 0.76 mmol/g and 1.44 mmol/g, respectively.

2.3. Preparation of MCM-41-2P-Pd(0) complex

To a solution of PdCl$_2$ (0.216 g, 1.22 mmol) in acetone (50 ml) was added the MCM-41-2P (2.01 g). The reaction mixture was refluxed under Ar for 72 h. The product was allowed to cool, and then filtered. The yellow solid was washed with distilled water (3 ml × 30 ml) and acetone (3 ml × 30 ml), and then stirred with hydrazine hydrate (1.6 g) and EtOH (25 ml) at 30 °C under Ar for 5 h. The resulting product was filtered, washed with EtOH (3 ml × 25 ml) and Et$_2$O (3 ml × 25 ml) and dried under vacuum at 60 °C to give 1.93 g of

the brown MCM-41-2P-Pd(0). The nitrogen, phosphine, and palladium contents were 0.58 mmol/g, 1.15 mmol/g and 0.52 mmol/g, respectively.

### 2.4. General procedure for the carbonylative Sonogashira coupling reaction

A 50 ml round-bottomed flask equipped with a gas inlet tube and a magnetic stirring bar was charged with MCM-41-2P-Pd(0) (96 mg, 0.05 mmol Pd). The atmosphere was replaced with carbon monoxide. Aryl iodide (1 mmol) and Et$_3$N (3 ml) were added and a slow stream of CO was allowed to flow into the flask. The mixture was stirred at 25 °C for 30 min, then terminal alkyne (1.2 mmol) was added, and stirring was continued at 25 °C for 25–72 h. The reaction mixture was dissolved in diethyl ether (50 ml). The palladium catalyst was separated from the mixture by filtration, washed with distilled water (2 ml × 10 ml), ethanol (2 ml × 10 ml) and ether (2 ml × 10 ml) and reused in the next run. The ethereal solution was washed with 20% aqueous hydrochloric acid (2 ml × 10 ml), saturated aqueous sodium hydrogen carbonate (10 ml) and distilled water (2 ml × 20 ml). The ether layer was dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (light petroleum–ethyl acetate = 10:1).

3. Results and discussion

It is well known that zero-valent palladium complexes show unique reactivity in various organic reactions. However, it is very difficult to use the Pd(0) complexes as practical catalysts because of their instability to air and moisture. Alper and co-workers first reported the synthesis of polymeric ligands containing *N,N*-bis(diphenylphosphinomethyl)amino-functionality by using the Mannich reaction [36]. Just recently insoluble versions of this chelating phosphine ligand have been applied in Rh-catalyzed hydroformylation and Rh-catalyzed hydrogenation [37,38]. The MCM-41-supported bidentate phosphine palladium(0) complex [MCM-41-2P-Pd(0)] was conveniently synthesized from commercially available and cheap γ-aminopropyltriethoxysilane via immobilization on MCM-41, followed by reacting with diphenylphosphinomethanol which resulted from adduct formation between diphenylphosphine and paraformaldehyde, and palladium chloride and then the reduction with hydrazine hydrate (Scheme 1).

X-ray powder diffraction (XRD) patterns of the parent MCM-41 and the modified materials MCM-41-2P, MCM-41-2P-Pd(0) are displayed in Fig. 1. Small angle X-ray powder diffraction of the parent MCM-41 gave peaks corresponding to hexagonally ordered mesoporous phases. For MCM-41-2P and MCM-41-2P-Pd(0), the (1 0 0) reflection of the parent MCM-41 with decreased intensity remained after functionalization, while the (1 1 0) and (2 0 0) reflections became weak and diffuse, which could be due to contrast matching between the silicate framework and organic moieties which are located inside the channels of MCM-41. These results indicated that the basic structure of the parent MCM-41 was not damaged in the whole process of catalyst preparation.

Elemental analyses and X-ray photoelectron spectroscopy (XPS) were used to characterize the MCM-41-supported bidentate phosphine palladium(0) complex. The phosphine and palladium contents of the MCM-41-2P-Pd(0) were estimated to be 1.15 and 0.52 mmol/g, respectively, and the P: Pd mole ratio of this complex was 2.21. The XPS data for MCM-41-2P, MCM-41-2P-Pd(II), MCM-41-2P-Pd(0) and PdCl$_2$ are listed in Table 1. It can be seen that the binding energies of N$_{1s}$, Si$_{2p}$, and O$_{1s}$ of MCM-41-2P-Pd(II) were similar to those of MCM-41-2P, and the binding energy of Cl$_{2p}$ of
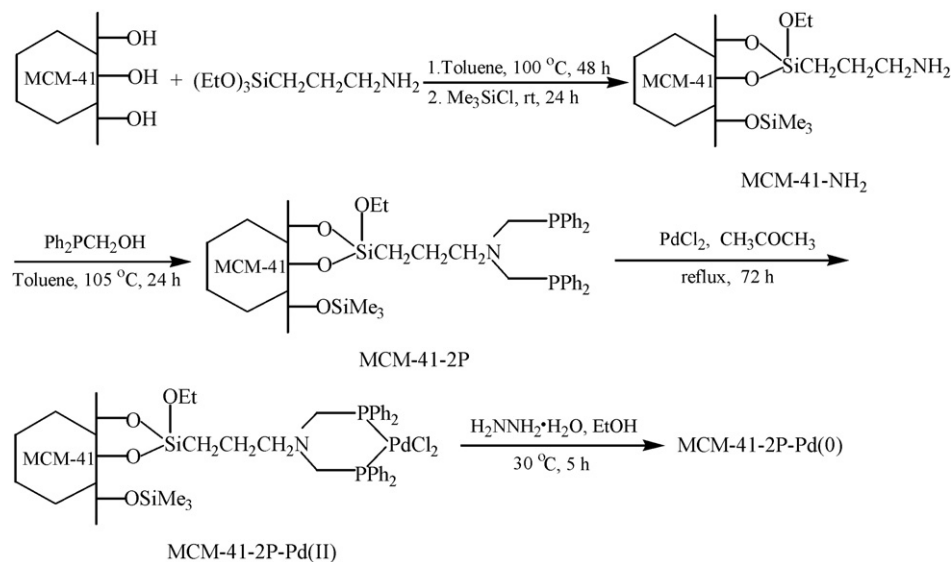
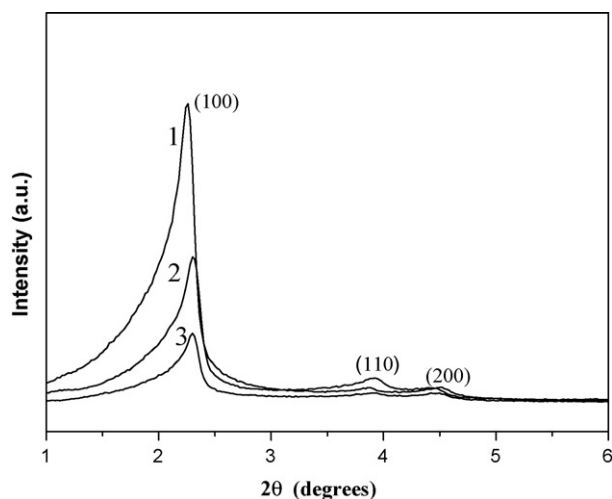**Scheme 1.** Preparation of the MCM-41-2P-Pd(0).

Fig. 1. XRD profiles of the parent MCM-41 (1), MCM-41-2P (2) and MCM-41-2P-Pd(0) (3).

MCM-41-2P-Pd(II) was similar to that of $PdCl_2$. However, the difference of $Pd_{3d5/2}$ binding energies between MCM-41-2P-Pd(II) and $PdCl_2$ was 1.2 eV. The difference of P_{2p} binding energies between MCM-41-2P-Pd(II) and MCM-41-2P was 0.5 eV. These results suggest that a coordination bond between P and Pd was formed in the MCM-41-2P-Pd(II). The binding energy (336.2 eV) of $Pd_{3d5/2}$ of MCM-41-2P-Pd(0) was lower than the binding energy (336.9 eV) of $Pd_{3d5/2}$ of MCM-41-2P-Pd(II). The $Pd_{3d5/2}$ binding energy depends strongly on the nature of the ligands. Consequently, it is impossible to identify the reduced complex as a zero-valent one on the basis

of its $Pd_{3d5/2}$ binding energy only. However, the binding energy of Cl_{2p} in the MCM-41-2P-Pd(0) cannot be detected, the shift (lower) of $Pd_{3d5/2}$ binding energy together with the change in color (from yellow to brown) suggests that the reduction of the starting palladium(II) complex to the lower valent state has taken place. The MCM-41-2P-Pd(0) complex catalyst formed was stable in air but for prolonged storage to be stored under an atmosphere of argon in which case no decomposition and deactivation were noted over the period of 6 months at room temperature.

In order to evaluate the catalytic activity of the MCM-41-supported bidentate phosphine palladium(0) complex [MCM-41-2P-Pd(0)], the carbonylative Sonogashira coupling reactions of terminal alkynes with aryl iodides were studied. Initially, to determine the optimum conditions, the influences of bases, solvents and amounts of the catalyst on the catalytic property of the MCM-41-2P-Pd(0) complex were investigated for the carbonylative Sonogashira coupling reaction of 4-iodoanisole with phenylacetylene. The results are shown in Table 2. Among the bases tested [pyrrolidine, Et_3N, Bu_3N, and aq. NH_3], Et_3N proved to be the most efficient. Among the solvents used [pyrrolidine, H_2O, CH_2Cl_2, toluene, THF, Bu_3N, Et_3N, and CH_3CN], Et_3N was also the best choice. Increasing the amount of palladium catalyst shortened

Table 1
XPS data for MCM-41-2P, MCM-41-2P-Pd(II), MCM-41-2P-Pd(0) and $PdCl_2$[a].

Sample	$Pd_{3d5/2}$	P_{2p}	N_{1s}	Si_{2p}	O_{1s}	Cl_{2p}
MCM-41-2P-Pd(0)	336.2	131.9	399.4	103.2	533.1	
MCM-41-2P-Pd(II)	336.9	131.7	399.2	103.3	533.0	199.3
MCM-41-2P		132.2	399.3	103.2	533.1	
$PdCl_2$	338.1					199.2

[a] The binding energies are referenced to C_{1s} (284.6 eV) and the energy differences were determined with an accuracy of ± 0.2 eV.

Table 2
Carbonylative Sonogashira coupling of 4-iodoanisole with phenylacetylene in the presence of several bases and solvents[a].

Entry	Base	Solvent	MCM-41-2P-Pd(0) (mol%)	Time (h)	Yield[b] (%)
1	Pyrrolidine	Pyrrolidine	5	48	39
2	Pyrrolidine	H_2O	5	48	51
3	Et_3N	H_2O	5	48	46
4	Et_3N	Et_3N	5	36	91
5	Et_3N	Toluene	5	48	35
6	Et_3N	CH_3CN	5	48	41
7	Et_3N	CH_2Cl_2	5	48	33
8	Bu_3N	Bu_3N	5	48	75
9	NH_3 (0.5 M)	H_2O	5	48	0
10	NH_3 (0.5 M)	THF	5	48	16
11	Et_3N	Et_3N	7	30	89
12	Et_3N	Et_3N	10	24	88

[a] All reactions were performed using 1.0 mmol of 4-iodoanisole, 1.2 mmol of phenylacetylene, 3.0 mmol of base in 3.0 ml of solvent at 25 °C under 1 atm of CO.
[b] Isolated yield based on the 4-iodoanisole used.
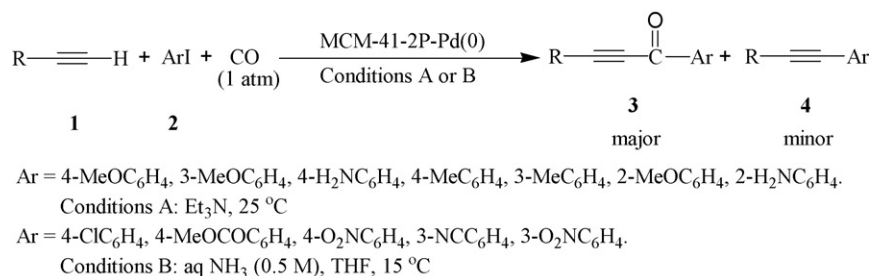
Scheme 2. Carbonylative Sonogashira coupling of aryl iodides with terminal alkynes catalyzed by MCM-41-2P-Pd(0).

the reaction time, but did not increase the yield of 1-phenyl-2-(4-methoxybenzoyl)-ethyne (entries 11, 12). Taken together, an excellent result was obtained when the carbonylative Sonogashira coupling reaction was carried out with 5 mol% of the catalyst using Et$_3$N as the base and the solvent at 25 °C (entry 4).

To examine the scope for this carbonylative coupling reaction, a variety of terminal alkynes were reacted with various aryl iodides in the presence of a catalytic amounts of MCM-41-2P-Pd(0) under atmospheric pressure of carbon monoxide in Et$_3$N at room temperature (Scheme 2). The experimental results are summarized in Table 3. As shown in Table 3, the carbonylative Sonogashira coupling reactions of the electron-rich aryl iodides with a variety of terminal alkynes proceeded smoothly in Et$_3$N at 25 °C in the presence of 5 mol% of MCM-41-2P-Pd(0) complex, giving the corresponding carbonylative coupling products in good to high yields. Only traces of the direct Sonogashira coupling products of the electron-rich aryl iodides with terminal alkynes were formed under such conditions. However, the carbonylative Sonogashira coupling reaction of iodobenzene with 1-hexyne under the same conditions afforded the carbonylative coupling product in 45% yield, along with 47% yield of noncarbonylative coupling product due to higher reactivity than the electron-rich aryl iodides (entry 5). The carbonylative

Sonogashira coupling reaction of the *ortho*-substituted electron-rich aryl iodides with terminal alkynes gave the corresponding carbonylative coupling products in moderate yields owing to the steric hindrance of the *ortho*-substituents (entries 7, 9, 17, 18). Interestingly, the unprotected *o*- or *p*-amino-phenyl iodides can participate in the reaction (entries 6–9), and good results can be obtained for *p*-amino-phenyl iodide (entries 6, 8). Unfortunately, the carbonylative Sonogashira coupling reaction of the electron-deficient aryl iodides with terminal alkynes was unsuccessful under the same conditions; only traces of carbonylative coupling products were formed and the direct Sonogashira coupling products were generated in good yields because of high reactivity of the electron-deficient aryl iodides.

Subsequently, we carried out the carbonylative Sonogashira coupling reaction of the electron-deficient aryl iodides with terminal alkynes using 2 equiv. of ammonia (0.5 M) as a base in THF at room temperature [18]. Interestingly, as previously reported [18], a mixture of carbonylative and noncarbonylative coupling products were obtained when the electron-deficient aryl iodide was used as the electrophile in the coupling reaction, presumably because the alkyne reacts too rapidly with the electro-deficient arylpalladium complex derived from the oxidative addition of the electro-deficient aryl iodide to palladium without insertion of CO. Thus, to get the desired carbonylative coupling products, we eventually carried out the reaction at 15 °C, and products 3s-w were obtained in 45–70% yields (entries 19–23).

It is noteworthy that the carbonylative coupling reaction of aryl iodides with the alkynes bearing alkyl substituents was achieved by running the reaction without CuI as a cocatalyst. We reasoned that in the absence of CuI, the less reactive alkyne (compared with its corresponding alkynyl copper ate complex) would easily react with the electron-deficient acylpalladium (II) rather than its precursor palladium complex (I) derived from the oxidative addition of aryl iodide to palladium; thus, the desired carbonylative coupling reaction could be secured. A plausible mechanism is shown in Scheme 3. When the electron-rich aryl iodide is used as the substrate, the insertion reaction of CO to less reactive arylpalladium complex (I) (compared with arylpalladium complex (I) produced by oxidative addition of the electron-deficient aryl iodide to palladium(0)) can proceed smoothly, giving the corresponding acylpalladium complex (II), which reacts with terminal alkyne in the presence of Et$_3$N to afford the carbonylative coupling product. However, when the electron-deficient aryl iodide is used as the substrate in the presence of Et$_3$N, the electron-deficient arylpalladium complex (I) reacts too rapidly with terminal alkyne without insertion of CO, affording the direct Sonogashira coupling products in good yields.

The MCM-41-supported bidentate phosphine palladium(0) catalyst can be easily recovered by simple filtration. We also examined the reuse of the catalyst by using the carbonylative Sonogashira coupling reaction of 4-iodoanisole with phenylacetylene. In general, the continuous recycle of resin-supported palladium catalysts is difficult owing to leaching of the palladium species from the
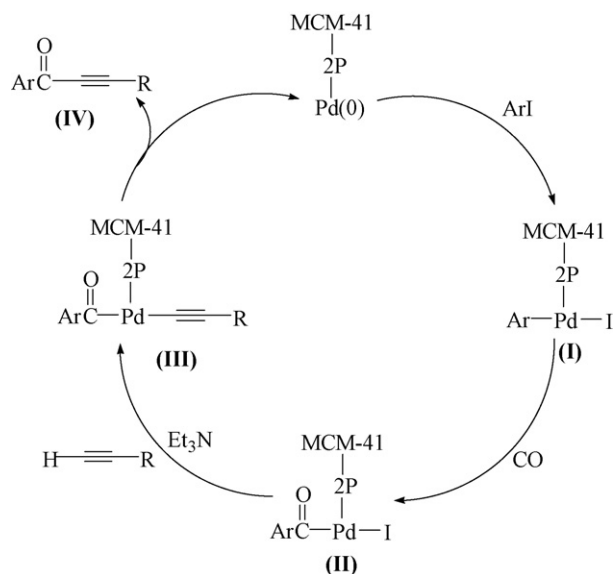
Table 3
Heterogeneous carbonylative Sonogashira coupling of aryl iodides with terminal alkynes catalyzed by MCM-41-2P-Pd(0)[a].

| Entry	R	Ar	Conditions	Time (h)	Yield[b] (%)	
					3	4
1	Ph	4-MeOC$_6$H$_4$	A	36	91 (3a)	0 (4a)
2	Ph	3-MeOC$_6$H$_4$	A	36	87 (3b)	2 (4b)
3	n-C$_4$H$_9$	4-MeOC$_6$H$_4$	A	36	90 (3c)	0 (4c)
4	n-C$_4$H$_9$	3-MeOC$_6$H$_4$	A	36	88 (3d)	0 (4d)
5	n-C$_4$H$_9$	Ph	A	25	45 (3e)	47 (4e)
6	Ph	4-H$_2$NC$_6$H$_4$	A	36	89 (3f)	0 (4f)
7	Ph	2-H$_2$NC$_6$H$_4$	A	48	60 (3g)	7 (4g)
8	n-C$_4$H$_9$	4-H$_2$NC$_6$H$_4$	A	36	87 (3h)	2 (4h)
9	n-C$_4$H$_9$	2-H$_2$NC$_6$H$_4$	A	48	63 (3i)	9 (4i)
10	Ph	4-MeC$_6$H$_4$	A	36	88 (3j)	0 (4j)
11	Ph	3-MeC$_6$H$_4$	A	36	86 (3k)	3 (4k)
12	n-C$_4$H$_9$	4-MeC$_6$H$_4$	A	36	86 (3l)	2 (4l)
13	n-C$_4$H$_9$	3-MeC$_6$H$_4$	A	36	83 (3m)	6 (4m)
14	n-C$_6$H$_{13}$	4-MeOC$_6$H$_4$	A	36	89 (3n)	0 (4n)
15	n-C$_6$H$_{13}$	4-MeC$_6$H$_4$	A	36	85 (3o)	3 (4o)
16	n-C$_6$H$_{13}$	3-MeC$_6$H$_4$	A	36	82 (3p)	5 (4p)
17	Ph	2-MeOC$_6$H$_4$	A	72	49 (3q)	8 (4q)
18	n-C$_4$H$_9$	2-MeOC$_6$H$_4$	A	72	51 (3r)	9 (4r)
19	Ph	4-ClC$_6$H$_4$	B	48	53 (3s)	34 (4s)
20	Ph	4-MeOCOC$_6$H$_4$	B	72	50 (3t)	35 (4t)
21	Ph	4-O$_2$NC$_6$H$_4$	B	36	45 (3u)	38 (4u)
22	Ph	3-NCC$_6$H$_4$	B	40	70 (3v)	21 (4v)
23	Ph	3-O$_2$NC$_6$H$_4$	B	38	64 (3w)	25 (4w)

[a] All the reactions were run with terminal alkyne (1.2 mmol), aryl iodide (1 mmol), CO (1 atm), and palladium catalyst (0.05 mmol). A: Et$_3$N (3 ml), 25 °C. B: aq. NH$_3$ (0.5 M, 4 ml), THF (8 ml), 15 °C.

[b] Isolated yield based on the aryl iodide 2 used.

Scheme 3. A plausible mechanism for carbonylative Sonogashira coupling of aryl iodides with terminal alkynes catalyzed by MCM-41-2P-Pd(0).

Table 4
Carbonylative Sonogashira coupling reaction of 4-iodoanisole with phenylacetylene catalyzed by recycled catalyst.
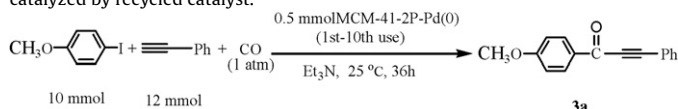
Entry	Catalyst cycle	Isolated yield (%)	TON
1	1st	91	18.2
2	10th	89	17.8
3	1st to 10th consecutive	av 90	Total of 180

polymer supports, which often reduces their activity within a five-recycle run. However, when the carbonylative coupling reaction of 4-iodoanisole with phenylacetylene was performed with 5 mol% of MCM-41-2P-Pd(0), the catalyst was recycled 10 times without any loss of activity. The 10th run promoted by the recycled catalyst gave 3a in 89% yield (Table 4, entry 2). The average yield of 3a in consecutive runs from the 1st to the 10th promoted by the recycled catalyst was 90% (entry 3). The high stability and excellent reusability of the catalyst should result from the chelating action of bidentate phosphine ligand on palladium and the mesoporous structure of MCM-41. The result is important from a practical point of view. The high catalytic activity, excellent reusability and the easy accessibility of the MCM-41-2P-Pd(0) make them a highly attractive supported palladium catalyst for the parallel solution phase synthesis of diverse libraries of compounds.

4. Conclusion

We have described the first heterogeneous carbonylative Sonogashira coupling reaction catalyzed by an MCM-41-supported bidentate phosphine palladium(0) complex. The MCM-41-2P-Pd(0) complex not only has high activity for the carbonylative Sonogashira coupling reactions of aryl iodides with terminal alkynes under atmospheric pressure of carbon monoxide without CuI as a cocatalyst but also offers practical advantages such as easy handling, separation from the products and reuse. The copper-free carbonylative Sonogashira coupling reaction of aryl iodides with terminal alkynes catalyzed by the MCM-41-2P-Pd(0) complex provides a better and practical procedure for the synthesis of α,β-alkynyl ketones compounds.

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