

Palladium supported on triphenylphosphine functionalized porous organic polymer: A highly active and recyclable catalyst for alkoxy carbonylation of aryl iodides



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ABSTRACT

An efficient method for the alkoxy carbonylation of aryl iodides using palladium supported on triphenylphosphine functionalized porous organic polymer (Pd@KAPs(Ph-PPh₃)) as the catalyst is reported. Under balloon pressure of CO, various aryl iodides on carbonylation with alcohols and phenols give the corresponding products in moderate to excellent yields (74–96%). The catalyst can be easily separated by simple filtration process and recycled up to ten times without significant decrease in activity. The salient features of this protocol are the simplicity in handling of the catalyst, low CO pressure, negligible palladium leaching and good catalyst recyclability.

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1. Introduction

Palladium catalyzed carbonylation reactions of aromatic halides in the presence of various nucleophiles represent a powerful method for the synthesis of a diverse array of benzoic acids and aryl ketone derivatives [1–3]. This field of research has attracted great interest because of its high compatibility to a wide variety of functional groups [4–9]. Nevertheless, there is still a constant strive dedicated to the developments of new carbonylative transformations, new catalysts, as well as to the improvement of existing Pd-catalyzed carbonylation reactions, including the heterogenization of homogeneous catalysts, the identification of milder reaction conditions, the use of lower carbon monoxide pressures and the development of carbon monoxide surrogates [4–9]. A number of research groups has been highly active in this field and has achieved great success including those of Beller and co-workers [10–18], Buchwald and co-workers [19–21], and Alper and co-workers [22–24]. Palladium catalyzed alkoxy carbonylation is an alternative route for ester synthesis, which has a number of advantages over the traditional methods that use carboxylic acid derivatives and alcohols or phenols. This new route is able to produce esters from inexpensive and easily available feedstock such as carbon

monoxide and organic halides in the presence of suitable alcohols or phenols [25,26]. Although homogeneous catalysts have been extensively investigated, their practical applications on a large scale remain a big challenge because the catalysts are expensive and it is difficult to recover the metal and remove residual metal from the product, which is very important for pharmaceutical uses. In order to address these problems, heterogeneous Pd catalysis is a promising option [27,28]. Researchers have immobilized palladium complexes on various supports such as activated carbon [29–32], silica [33–36], MCM-41 [37], organic polymer [38,39], SBA-15 [40,41], ZIF-8 [42], Fe₃O₄ [43], and MOF-5 [44] to create heterogeneous catalysts for carbonylation of aryl halides. The latest and impressive heterogeneous catalyst example reported is the use of immobilized palladium metal-containing ionic liquid as a catalyst for the alkoxy carbonylation and aminocarbonylation of aryl iodides. Using this catalyst, high activities were achieved under 0.5–1.0 MPa CO pressure, and the catalyst could be recycled 4 times [45]. However, the main challenges remained in developing active and stable heterogeneous catalysts to carry out the reaction under mild reaction conditions and addressing the contamination of the leached metal impurities in the product.

The combination of the advantageous properties of molecular and solid catalysts was considered as the “Holy Grail” in catalysis research. Great potential was provided recently by porous polymers [46]. Porous organic polymers (POPs), emerged just in a few years ago, have attracted great attention due to their unique prop-

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erties such as large surface area, low skeletal density, and high chemical stability [47]. One particular advantage of POPs is its ability to introduce a broad range of useful chemical functionalities into the porous framework. Porous organic polymers containing functional units such as phthalocyanine, bipyridine, porphyrins, and triazine rings have been reported, and these materials exhibited excellent catalytic activity in catalytic reactions [46]. Recently, palladium supported on triphenylphosphine-functionalized microporous knitting aryl network polymers (Pd@KAPs(Ph-PPh₃)) has been used in Suzuki reactions [47]. And it has displayed many advantages such as good reusability of the expensive metal and phosphine ligand, the abundant open micropore and macroporous structure that is favorable for catalysis process and the possibility to prevent the contamination of palladium and ligand residue in products. However, to the best of our knowledge, such porous polymer-supported palladium catalyst has not been used in carbonylation reactions.

In continuation of our interest on carbonylation reactions [48–50], herein we report a facile protocol for the atmospheric pressure carbonylation of aryl iodides with alcohols and phenols using Pd@KAPs(Ph-PPh₃) as an efficient, heterogeneous and recyclable catalyst.

2. Experimental

2.1. Reagents and characterization

Benzene, triphenylphosphine, PdCl₂, FeCl₃ (anhydrous) and 1,2-dichloroethane (DCE), acetonitrile were obtained from National Medicines Corporation Ltd., of China, all of which were of analytical grade and were used as received. Palladium on carbon (Pd/C, 5 wt%), aryl iodides, alcohols, phenols, and bases were of analytical grade and commercially available. Formaldehyde dimethyl acetal (Alfa Aesar, 98%) was also used as received. All solvents were analytical grade and distilled prior to use. FT-IR spectra were recorded under ambient conditions in the wave number range of 4000–400 cm⁻¹ using a Bruker Equinox 55 FTIR spectrophotometer. Pd content data were obtained on AAS using a PerkinElmer AA-300 spectrophotometer. Nuclear magnetic resonance (NMR)

spectra were recorded in CDCl₃ on a Bruker AVANCE III 400 MHz spectrometer at room temperature using tetramethylsilane (TMS) as an internal reference.

2.2. Preparation of the catalysts

The Pd@KAPs(Ph-PPh₃) [47], Pd/Fe₃O₄ [43] were prepared according to the literature. PdCl₂(phen)@Y was prepared as we reported previously [48].

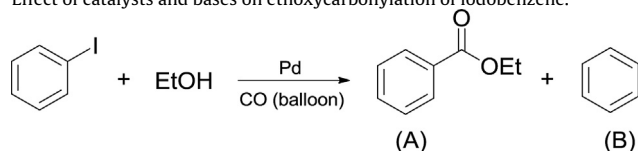
2.3. Typical procedure for carbonylation reactions

The catalytic reactions were carried out in a 10 mL reaction flask and fitted with condenser and carbon monoxide balloon. In a typical run, a catalyst containing 1.0 mol% Pd, aryl iodide (0.5 mmol) and DBU (1.5 mmol) were added to solvent and allowed to react under CO atmosphere at 80 °C temperature for 6–10 h. After the reaction, the flask was cooled to room temperature and carbon monoxide balloon was removed. The reaction mixture was then centrifuged and the clear supernatant was analyzed with GC by using *n*-butanol as an internal standard. For the study of substrate scope, after completion of the reaction, the catalyst was centrifuged and extracted with copious ethanol. The obtained liquid was concentrated. For phenoxycarbonylation, the obtained liquid was diluted with saturated NH₄Cl and extracted with diethyl ether. The organic layer was dried over anhydrous Na₂SO₄ and then concentrated. The product was obtained by preparative thin-layer chromatography (PTLC) using petroleum ether and ethyl acetate (30:1, v/v) as eluting solvent. The purity of products was checked by NMR and yields were based on aryl iodides.

3. Results and discussion

The Pd@KAPs(Ph-PPh₃) was characterized by Fourier transform infrared (FTIR) spectroscopy and atomic absorption spectrum (AAS). FTIR spectroscopy of KAPs(Ph-PPh₃) and Pd@KAPs(Ph-PPh₃) (Fig. S1 of Supplementary content) displays a series of bands around 1600–1450, 1250–950, and 900–650 cm⁻¹ which can be attributed to benzene skeleton stretching, C–H out-of-plane bending and in-

Table 1
Effect of catalysts and bases on ethoxycarbonylation of iodobenzene.^a



Entry	Catalyst	Pd [wt%]	Base	Yield A (%) ^b	Yield B (%) ^b
1	Pd@KAPs(Ph-PPh ₃)	0.8	Et ₃ N	76	0
2	Pd/C	5	Et ₃ N	34	0
3	Pd/Fe ₃ O ₄	10	Et ₃ N	19	0
4	PdCl ₂ (phen)@Y	2.1	Et ₃ N	17	0
5	Pd@KAPs(Ph-PPh ₃)	0.8	K ₂ CO ₃	45	55
6	Pd@KAPs(Ph-PPh ₃)	0.8	CsCO ₃	9	90
7	Pd@KAPs(Ph-PPh ₃)	0.8	Na ₂ CO ₃	49	49
8	Pd@KAPs(Ph-PPh ₃)	0.8	K ₃ PO ₄ ·3H ₂ O	6	91
9	Pd@KAPs(Ph-PPh ₃)	0.8	NaHCO ₃	86	0
10	Pd@KAPs(Ph-PPh ₃)	0.8	KHCO ₃	91	0
11	Pd@KAPs(Ph-PPh ₃)	0.8	Na ₂ HPO ₄	5	0
12	Pd@KAPs(Ph-PPh ₃)	0.8	K ₂ HPO ₄	30	0
13	Pd@KAPs(Ph-PPh ₃)	0.8	DBU	99	0
14 ^c	Pd@KAPs(Ph-PPh ₃)	0.8	DMAP	8	0
15	Pd@KAPs(Ph-PPh ₃)	0.8	Pyridine	5	0
16 ^d	Pd@KAPs(Ph-PPh ₃)	0.8	DBU	85	0

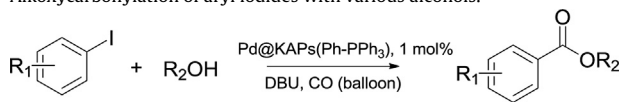
^a Reaction conditions: Pd catalyst (1.0 mol%), iodobenzene (0.5 mmol), base (1.5 mmol), EtOH (3 mL), 80 °C, 6 h, CO (1 atm).

^b GC yield.

^c DMAP = 4-dimethylaminopyridine.

^d Pd catalyst was used in 0.5 mol%.

Table 2
Alkoxy carbonylation of aryl iodides with various alcohols.^a



Entry	Ar-I	R ₂ OH	Product	Yield (%) ^b
1		MeOH		95
2		<i>n</i> -PrOH		93
3		<i>n</i> -BuOH		95
4				91
5		<i>i</i> -PrOH		86
6				74
7		<i>t</i> -BuOH		0
8		EtOH		94
9		EtOH		93
10		EtOH		92
11		EtOH		96
12		EtOH		93

Table 2 (Continued)

Entry	Ar-I	R ₂ OH	Product	Yield (%) ^b
13		EtOH		91
14		EtOH		93
15		EtOH		82
16		EtOH		94
17		EtOH		82

^a Reaction conditions: Pd catalyst (1.0 mol%), aryl iodide (0.5 mmol), DBU (1.5 mmol), alcohol (3 mL), 80 °C, 6 h, CO (1 atm).

^b Isolated yield.

plane bending vibrations of the benzene ring, respectively [47,51]. The peaks at 1437 cm⁻¹ correspond to the vibrations of the P–CH₂ bond [47,52]. The Pd content of Pd@KAPs(Ph-PPh₃) was 0.8 wt%, as measured by AAS.

To evaluate the catalytic activity of the thereby obtained Pd@KAPs(Ph-PPh₃) catalyst, ethoxycarbonylation of iodobenzene was chosen as a model reaction. The reaction was performed in ethanol under atmospheric CO pressure. Firstly, the influences of catalysts and bases were examined on the model reaction, and the obtained results are summarized in Table 1. Pd@KAPs(Ph-PPh₃) gave 76% yield of ethyl benzoate within 6 h at 80 °C using 1 mol% Pd in the presence of Et₃N (Table 1, entry 1). Some other heterogeneous Pd catalysts, such as Pd/C, Pd/Fe₃O₄, and PdCl₂(phen)@Y, were also tested under the identical reaction conditions, and the yields of ethyl benzoate reached only to 34%, 19%, and 17%, respectively (Table 1, entries 2–4).

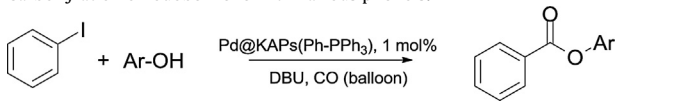
It is well known that, in the carbonylation of aryl iodide, bases can remarkably influence the catalytic activity and selectivity [29–45]. To clarify the effect of base in Pd@KAPs(Ph-PPh₃)-catalyzed reaction, various organic and inorganic bases were then tested (Table 1, entries 5–15). It was observed that the base indeed strongly affected the reaction selectivity and activity. When strong inorganic bases, such as K₂CO₃, Cs₂CO₃, Na₂CO₃, and K₃PO₄·3H₂O, were used, significant amount of benzene that was generated through a reductive deiodination of iodobenzene was detected (Table 1, entries 5–8). Weak inorganic bases, such as NaHCO₃ and KHCO₃, worked well, and the yields of ethyl benzoate reached to 86% and 91%, respectively (Table 1, entries 9 and 10). Among all the bases screened, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene) displayed the best performance, with which 99% yield could be obtained (Table 1, entry 13). Effect of the catalyst loading was also studied by using DBU as a base. When 0.5 mol% of Pd@KAPs(Ph-PPh₃) was used, the reaction proceeded also well, but the yield

obtained was slightly inferior compared with that of 1.0 mol% catalyst (Table 1, entry 16). Thus, the optimal conditions are: DBU as the base, and 1.0 mol% catalyst.

Then, we turned our focus to the scope and limitations of the system. Firstly, the reaction of iodobenzene with different alcohols was investigated, and the results are summarized in Table 2. Iodobenzene reacted efficiently with primary alcohols, such as methanol, *n*-propanol, *n*-butanol, and benzyl alcohol, and the corresponding benzoates were obtained in excellent yields (Table 2, entries 1–4). When secondary alcohols, such as isopropanol and cyclohexanol, were used, moderate yields were obtained (Table 2, entries 5 and 6). Unfortunately, no desired product could be detected when *t*-butanol was used as a nucleophile. This might result from the steric hindrance effect of *tert*-butyl group that impedes the coordination of alcohol with the metal center (Table 2, entry 7) [53]. Next, the alkoxy carbonylation of various aryl iodides with ethanol was also investigated. Aryl iodides bearing both electron-withdrawing and electron-donating groups participated in the reactions readily, affording the corresponding benzoates in moderate to excellent yields (Table 2, entries 8–15). Interestingly, when methyl 4-iodobenzoate was used as substrate, an unsymmetrical dialkyl terephthalate was obtained in 82% yield. Considering the possible effect of transesterification reaction, this result is quite remarkable (Table 2, entry 15). 3-Iodopyridine, as a representative example of aromatic heterocyclic iodide, resulted in 94% yield of ethyl nicotinate (Table 2, entry 16). Benzyl chloride gave the desired product in 82% yield (Table 2, entry 17). Indeed, organic bromides, such as bromobenzene, 3-bromopyridine, β-bromostyrene were also tested, unfortunately, no target products were obtained.

This catalyst was also used in the alkoxy carbonylation of aryl iodides with phenols, so called phenoxy carbonylation. Considering the fact that the nucleophilicity of phenol is lower than that of ethanol, reaction time of the phenoxy carbonylation was increased

Table 3
Carbonylation of iodobenzene with various phenols.^a



Entry	Ar-OH	Product	Yield (%) ^b
1			96
2			81
3			94
4			96
5			94
6 ^c			92

^a Reaction conditions: Pd catalyst (1.0 mol%), iodobenzene (0.5 mmol), phenol (0.7 mmol), DBU (1.5 mmol), DMAc (3 mL), 80 °C, 10 h, CO (1 atm).

^b Isolated yield.

^c NaHCO₃ (1.5 mmol) was used as the base.

to 10 h. As we expected, iodobenzene reacted smoothly with phenol, providing phenyl benzoate in 96% of yield (Table 3, entry 1). Substituted phenols, such as *o*-methyl phenol, *m*-methyl phenol, *p*-methyl phenol, and *p*-chlorophenol, are also viable substrates in this reaction, with which the phenyl benzoates could be obtained in good to excellent yields (Table 3, entries 2–5). 2-Acetylphenyl benzoate [54,55], an important intermediate for the synthesis of bio-active flavone, could be obtained in 92% yield via phenoxycarbonylation of iodobenzene and 2-hydroxy acetophenone (Table 3, entry 6).

When a supported metal catalyst was used in a liquid phase reaction, two points need to be clarified. The first one is the metal leaching that sometimes is partially responsible for the observed good catalytic activity in a reaction in liquid phase. To prove the heterogeneous nature of the catalyst, a hot filtration test was carried out. A Pd@KAPs(Ph-PPh₃)-catalyzed ethoxycarbonylation of iodobenzene was stopped after 1 h, when the yield reached to 41%. The solid was then filtered off at the reaction temperature. The liquid was injected to another reaction tube with CO balloon. No significant increase in terms of the product concentration was observed after 5 h of reaction under the identical conditions (Fig. 1). We also tested Pd content in the filtrate by means of AAS, the result showed that palladium leaching is very low, to be ca. 1.1 ppm, indicating 99% Pd species are still on the solid catalyst during the reaction. These results suggested that all the palladium

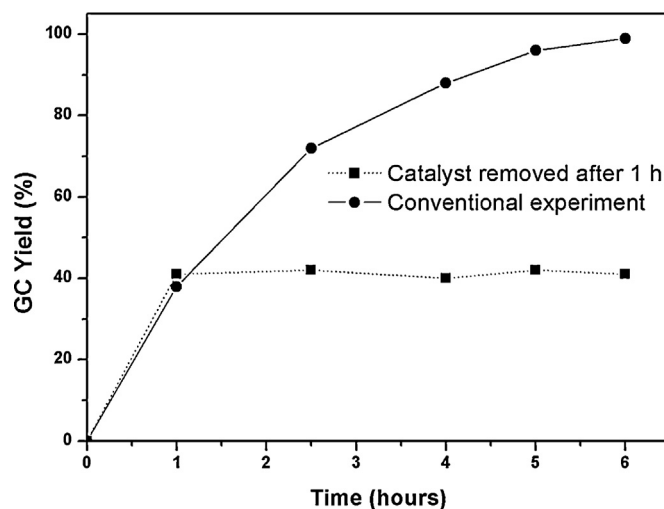


Fig. 1. Hot filtration reaction profile for Pd@KAPs(Ph-PPh₃) for the ethoxycarbonylation of iodobenzene. Reaction conditions: iodobenzene (0.5 mmol), DBU (1.5 mmol), EtOH (3 mL), 80 °C, CO (1 atm), GC yield.

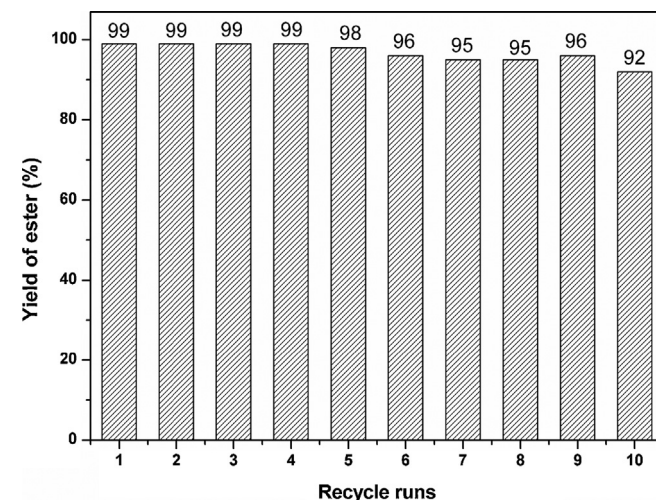


Fig. 2. Reuse of Pd@KAPs(Ph-PPh₃) (1 mol%) catalyst for the ethoxycarbonylation of iodobenzene under ambient pressure of CO. Reaction conditions: iodobenzene (0.5 mmol), DBU (1.5 mmol), EtOH (3 mL), 80 °C, 6 h, GC yield.

species were strongly absorbed on to the support and no significant quantities of metal were lost to the reaction liquid during the process.

The second one is the recyclability of the catalyst. To this end, at the end of the model reaction, Pd@KAPs(Ph-PPh₃) was separated by simple centrifugation and extracted with copious ethanol, and then subjected to the next run. As shown in Fig. 2, the catalyst exhibited good reusability and could be reused ten times. Only a slight decrease of its activity was observed, which can be attributed partially to a physical loss of the catalyst during the operation.

4. Conclusion

In conclusion, the present study reports an efficient and a reusable protocol for the alkoxycarbonylation of aryl iodides with different alcohols and phenols by using a Pd@KAPs(Ph-PPh₃) catalyst. With this catalyst, the corresponding products could be obtained in moderate to excellent yields. The catalyst can be recycled at least ten times without significant loss of its catalytic activity. Thus, this heterogeneous catalyst has significant advantages over the corresponding homogeneous catalysts as it is not

only applicable for the synthesis of a wide range of benzoates under mild conditions, but also benefits the catalyst-product separation and reduces the amount of residual palladium in the isolated products. Hence, the present catalyst system offers an environmentally attractive carbonylation method for the synthesis of various benzoates from readily available feedstock.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.molcata.2014.12.008>.

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