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Article

Carbonylative Suzuki coupling reactions of aryl iodides with arylboronic acids over Pd/SiC

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ABSTRACT

High surface area SiC has been used to prepare a Pd/SiC catalyst using the liquid reduction method, and the resulting catalyst was used for the carbonylative Suzuki coupling reaction of aryl iodides with arylboronic acids. The catalyst was also characterized by X-ray diffraction, inductively coupled plasma-mass spectroscopy and high-resolution transmission electron microscopy. The results of these analyses showed that homogeneous Pd nanoparticles with a mean diameter of 2.8 nm were uniformly dispersed on the SiC surface. Optimization of the reaction conditions for the carbonylative Suzuki coupling reaction, including the solvent, base, pressure, temperature and reaction time, revealed that the model reaction of iodobenzene (1.0 mmol) with phenylboronic acid (1.5 mmol) could reach 90% conversion with a selectivity of 99% towards the diphenyl ketone using 3 wt% Pd/SiC under 1.0 MPa of CO pressure at 100 °C for 8 h with K₂CO₃ (3.0 mmol) as the base and anisole as the solvent. The Pd/SiC catalyst exhibited broad substrate scope towards the carbonylative Suzuki coupling reaction of aryl iodides with arylboronic acids bearing a variety of different substituents. Furthermore, the Pd/SiC catalyst exhibited good recyclability properties and could be recovered and reused up to five times with the conversion of iodobenzene decreasing only slightly from 90% to 76%. The decrease in the catalytic activity after five rounds was attributed to the loss of active Pd during the organic reaction.

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1. Introduction

Aromatic ketones are important building blocks in organic chemistry, and compounds belonging to this structural class can be found in a wide variety of biologically active natural products and pharmaceutical molecules [1,2]. In light of their importance and general utility, various methods have been reported for the preparation of aromatic ketones. Traditional synthetic routes for the construction of aromatic ketones include the Friedel-Crafts acylation of aromatic compounds with

acyl halides [3] and the transition-metal-catalyzed cross-coupling reactions of aryl metal reagents such as C₆H₅MgBr [4] and C₆H₅SiF_nMe_{3-n} [5] with carbon monoxide and a suitable electrophile [6]. However, the former of these two reactions requires the addition of a large excess of Lewis acid and is incompatible with many functional groups [3,7,8], whereas the latter usually results in the formation of biphenyl byproducts.

In 1993, Suzuki's group [9] reported a direct method (i.e., the carbonylative Suzuki coupling reaction) for the synthesis of aromatic ketones by the reaction of CO with an aryl halide and

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arylboronic acid in the presence of PdCl_2 and $\text{Pd}(\text{dba})_2$ (bis(dibenzylideneacetone)palladium). This reaction provides a versatile platform for the direct synthesis of aromatic ketones using boronic acids, which are generally non-toxic and stable to air and moisture. Ishiyama *et al.* [10] used a variety of different catalysts to affect this carbonylative Suzuki reaction, including $\text{PdCl}_2 (\text{PPh}_3)_2$ and $\text{PdCl}_2 (\text{dppf})$ ([1,1'-bis(diphenylphosphino)ferrocene]dichloropalladium(II)). However, the conversion and selectivity of these reactions are low and require the presence of a ligand to proceed effectively. Khedkar *et al.* [11] reported that immobilized palladium ion-containing ionic liquids exhibited excellent catalytic activity towards the carbonylative Suzuki coupling reaction of aryl halides with arylboronic acids, with the coupled products being isolated in high yields. However, the overall utility of this process is limited by its requirement for the use of toxic organic compounds, which are harmful to the environment. Niu *et al.* [12] reported the stabilization of Pd on hollow magnetic mesoporous spheres and the use of this material as a heterogeneous catalyst for the carbonylative Suzuki coupling reaction. However, the preparation of this catalyst is complex. Therefore, despite good progress during the last 20 years towards the development of robust methods for the preparation of aromatic ketones, the high costs associated with the preparation of complex catalysts and ligands, and the difficulties involved in the separation of catalysts from the reaction mixture have greatly limited the application and development of the carbonylative Suzuki coupling reaction. Based on these limitations, there is still considerable scope for exploring the development of new stable and efficient heterogeneous catalysts for the carbonylative Suzuki coupling reaction.

SiC has excellent chemical stability and thermal conductivity properties, and has been successfully used as catalyst support in various catalytic reactions, including the oxidation of CO [13], reforming of CH_4 and CO_2 [14,15], photocatalytic water splitting [16], methanation [17] and the photocatalytic reduction of CO_2 [18]. We recently reported that SiC-supported Pd nanoparticles significantly enhanced the hydrogenation of furan derivatives under low temperature (25 °C) and low pressure (1.0 MPa of H_2) conditions by visible light irradiation [19]. Notably, the yield of tetrahydrofuran from the hydrogenation of furan under these conditions was 99% with a turnover frequency of 70 h^{-1} . In this study, a Pd/SiC catalyst has been prepared by a liquid reduction method using high surface area SiC as a support, and investigated its catalytic performance towards the carbonylative Suzuki coupling reaction under heterogeneous conditions.

2. Experimental

2.1. Preparation and characterization of Pd/SiC

The Pd/SiC catalyst (3 wt%) was prepared using a liquid phase reduction method. Palladium nitrate (132.6 mg, 0.564 mmol) and SiC powder (1.94 g, $S_{\text{BET}} = 50 \text{ m}^2/\text{g}$) were dispersed in absolute ethanol (50 mL) under sonication conditions, and the resulting suspension was magnetically stirred to get a homogeneous mixture of palladium nitrate and SiC. The mixture was dispersed in 80 mL of diethylene glycol (DEG), and the

resulting suspension was heated to 140 °C for 30 min. Water (2.5 mL) was then added to the reaction, and the resulting mixture was reduced for 2 h at 180 °C to give the 3 wt% Pd/SiC catalyst.

The Pd content of the catalyst was determined by inductively coupled plasma (ICP) atomic emission spectrometry using an Atomscan 16 system (Thermo Fisher Scientific, Shanghai, China). The phase characterization of the catalyst was determined using a Rigaku D-Max/RB X-ray diffraction (XRD) system (Tokyo, Japan) using $\text{Cu-K}\alpha$ radiation. The structure and morphology of the catalyst were characterized by transmission electron microscopy (TEM) using a JEM-2100F system (JEOL, Tokyo, Japan). X-ray photoelectron spectroscopy (XPS) measurements were conducted on an ESCALAB 3 MKII de VG spectrometer (VG Scientific, East Sussex, United Kingdom) using $\text{Mg K}\alpha$ (15 kV, 20 mA) as an X-ray source.

2.2. Carbonylative Suzuki coupling reactions

All of the carbonylative Suzuki coupling reactions were conducted in a sealed stainless steel reaction kettle. Arylboronic acid (1.5 mmol), aryl iodide (1.0 mmol), base (3.0 mmol), 3 wt% Pd/SiC and anisole (10 mL) were added to the reaction kettle, and the resulting mixture was placed under a certain pressure of CO. All of the reactions were conducted over several hours at certain temperatures. The products of the reactions were analyzed by GC-MS on a Bruker SCION SQ 456 GC-MS system (Karlsruhe, Germany).

3. Results and discussion

3.1. Morphology and structure of the Pd/SiC catalyst

The Pd loading of the Pd/SiC catalyst was determined to be 2.91 wt% by ICP-MS analysis, which is similar to the calculated value. BET analysis showed that the surface area of the Pd/SiC catalyst was $56 \text{ m}^2/\text{g}$. The TEM images of the Pd/SiC catalyst (Fig. 1) showed that homogeneous metallic Pd nanoparticles were uniformly dispersed on the SiC surface with a narrow size distribution of 1–5 nm and a mean diameter of 2.8 nm. The lattice fringe of the nanoparticles had an interplanar spacing of 0.19 nm, which was in agreement with the (200) plane of Pd. The XRD pattern of the Pd/SiC catalyst (Fig. 2) showed that all of the strong diffraction peaks could be indexed to β -SiC. The

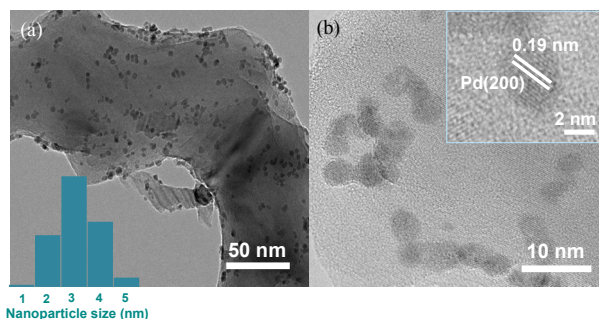


Fig. 1. TEM and HRTEM images (inset of (b)) of Pd/SiC catalyst, and the size distribution of Pd nanoparticles (inset of (a)).

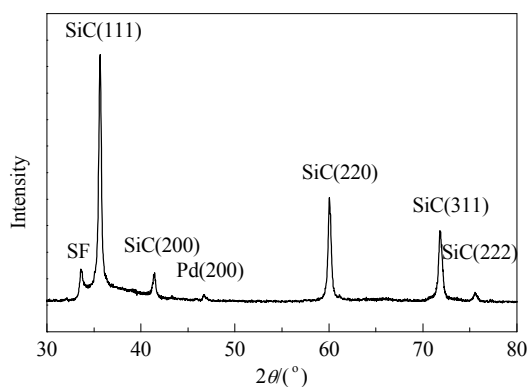


Fig. 2. XRD pattern of the Pd/SiC catalyst.

diffraction peaks at $2\theta = 33.6^\circ$ and 46.7° were attributed to stacking faults and cubic Pd, respectively.

3.2. Effect of reaction conditions

The effects of various reaction parameters, including the solvent, base, temperature, time and CO pressure, were investigated for the reaction of iodobenzene with phenylboronic acid, and the results are summarized in Table 1. Several different bases, including K_2CO_3 , Cs_2CO_3 and Na_2CO_3 , were investigated in toluene at 100°C under 0.5 MPa of CO pressure over 5 h (Table 1, entries 1–3). The iodobenzene conversion in these three cases were found to be 51%, 10% and 42%, respectively, with selectivity towards the diphenyl ketone of 93%, 91% and 90%, respectively, which suggested that the nature of the base was having a significant impact of the outcome of the reaction. Because K_2CO_3 gave the highest conversion, it was selected as the optimal base for the remaining optimization reactions. When anisole was used instead of toluene as the reaction solvent (Table 1, entry 4), the conversion of iodobenzene and selectivity towards diphenyl ketone reached 62% and 98%, re-

Table 1

The catalytic performance of Pd/SiC for carbonylative Suzuki coupling reaction of iodobenzene with phenylboronic acid under different reaction conditions ^a.

Entry	Solvent	Base	p/MPa	$T/^\circ\text{C}$	t/h	$C/\%$	$S/\%$
1	Toluene	K_2CO_3	0.5	100	5	51	93
2	Toluene	Cs_2CO_3	0.5	100	5	10	91
3	Toluene	Na_2CO_3	0.5	100	5	42	90
4	Anisole	K_2CO_3	0.5	100	5	62	98
5	Anisole	K_2CO_3	0.5	80	5	21	95
6	Anisole	K_2CO_3	0.5	120	5	78	93
7	Anisole	K_2CO_3	0.5	100	8	74	97
8	Anisole	K_2CO_3	1.0	100	8	90	99
9 ^b	Anisole	K_2CO_3	1.0	100	8	17	96
10 ^c	Anisole	K_2CO_3	1.0	100	8	29	94

Reaction conditions: (a) A mixture of iodobenzene (1.0 mmol), phenylboronic acid (1.5 mmol), base (3.0 mmol) and 3 wt% Pd/SiC (50 mg, containing 0.014 mmol Pd) in solvent (10 mL) was heated at a specific temperature under a certain pressure of CO for a specific time; (b) pure SiC (50 mg) was used as the catalyst; (c) $\text{Pd}(\text{NO}_3)_2$ (3.2 mg, containing 0.014 mmol Pd) was used as the catalyst.

Note: p = pressure, T = temperature, t = time, C = conversion and S = selectivity. Calculations for conversion and selectivity are based on iodobenzene.

spectively, which are higher than the values achieved using toluene as solvent. Taken together with the fact that toluene is more toxic than anisole, the latter of these two solvents was chosen as the optimal solvent for the reaction system.

An increase in the reaction temperature led to an increase in the conversion of iodobenzene (Table 1, entries 4–6). When the temperature was increased from 100 to 120°C , the conversion increased from 62% to 78%, although the selectivity towards the diphenyl ketone decreased slightly from 98% to 93%. The use of higher temperatures can lead to an increase in the quantity of adsorbed reactant molecules in the excited states, as dictated by the Bose-Einstein distribution, which means that the reactant molecules then require less energy to overcome the reaction barrier. However, side reactions leading to the formation of biphenyl also occur much more readily at higher temperatures, leading to a decrease in selectivity towards the diphenyl ketone. Increasing the CO pressure can lead to an increase in the number of CO molecules being absorbed and activated by the active sites of the Pd catalyst, and this effect led to an improvement in the catalytic activity of the Pd/SiC catalyst (Table 1, entry 8). In the same way, extending the reaction time led to an increase in the conversion of iodobenzene (Table 1, entries 7 and 8). Taken together, the results of these optimization reactions revealed that iodobenzene conversion and diphenyl selectivity values of 90% and 99%, respectively, could be achieved using anisole as a solvent and K_2CO_3 as a base under 1.0 MPa of CO pressure at 100°C following a reaction time of 8 h (Table 1, entry 8).

Control experiments using only pure SiC or $\text{Pd}(\text{NO}_3)_2$ instead of the Pd/SiC catalyst were conducted in the absence of a ligand under the optimized conditions and gave iodobenzene conversion values of only 27% and 29%, respectively (Table 1, entries 9 and 10). These results indicated that metallic Pd was involved as the active phase for this reaction and therefore critical to a high level of conversion. The rate of carbonylative Suzuki coupling reaction between an aryl iodide and an arylboronic acid is usually slow, whereas the self-coupling of arylboronic acids generally occurs at a much faster rate. With this in mind, the stoichiometry of the reaction was fixed with a significant excess of the phenylboronic acid (1.5 mmol) relative to the iodobenzene (1.0 mmol) to achieve the highest possible level of iodobenzene conversion. It was envisaged that the excess arylboronic acid would form the corresponding biphenyl.

A hot filtration experiment was conducted to further confirm the active phase of the catalyst. Briefly, the Pd/SiC catalyst was separated from the reaction system by hot filtration after the reaction had proceeded for 4 h with a 51% conversion of iodobenzene, and the resulting filtrate was allowed to react under the same reaction conditions in the absence of the Pd/SiC catalyst for 4 h. The results of this experiment showed that there was no change in iodobenzene conversion, which provided further confirmation that metallic Pd nanoparticles were performing as the active phase in the current carbonylative Suzuki reaction.

Based on these results, the optimized reaction conditions were determined to be as follows: iodobenzene (1.0 mmol), phenylboronic acid (1.5 mmol) and K_2CO_3 (3.0 mmol) in anisole

(10 mL). The reaction was catalyzed by 3 wt% Pd/SiC at 100 °C and 1.0 MPa of CO pressure for 8 h. The optimized conditions (Table 2, entry 1) gave an iodobenzene conversion of 90% with a selectivity towards diphenyl ketone of 99%.

3.3. Catalytic performance of Pd/SiC for different aryl iodides with arylboronic acids

To test the general applicability of our newly developed Pd/SiC catalyst, we evaluated its performance as a catalyst towards the reaction of a wide range of aryl iodides and arylboronic acids bearing various different substituents. The results of these experiments are summarized in Table 2, and show that the Pd/SiC catalyst exhibited excellent activity towards a wide range of different substrates.

The presence of an electron-withdrawing group on the aryl iodide, such as a NO₂, CN, Ac or Cl group, greatly improved the reaction conversion (Table 2, entries 2–5). In contrast, the presence of an electron-donating group on the aryl iodide, such as an NH₂, Me or OMe group, led to a decrease in the conversion compared with iodobenzene (Table 2, entries 6–8). This difference in the reactivity patterns of the aryl iodide substrates was attributed to the electron-withdrawing group effectively enhancing the rate of the nucleophilic substitution reaction. The carbonylative Suzuki coupling reactions of iodobenzene with 4-methoxybenzeneboronic acid and 4-*t*-butylphenylboronic acid proceeded smoothly to give the corresponding products in high yields of 82% and 88%, respectively (Table 2, entries 9 and 10).

Pleasingly, the reaction was found to be amenable to

scale-up and the Pd/SiC catalyst exhibited good catalytic activity when it was scaled-up 10-fold (i.e., 10.0 mmol aryl iodide, 15.0 mmol arylboronic acid, 30.0 mmol K₂CO₃ and 500 mg of 3 wt% Pd/SiC in 100 mL of anisole under 1.0 MPa of CO and 100 °C for 8 h). For example, the conversion of iodobenzene, *p*-iodonitrobenzene and *p*-iodotoluene were found to be 87%, 95% and 79%, respectively, at the larger scale and the selectivity towards the corresponding aromatic ketones was 99% in all three cases.

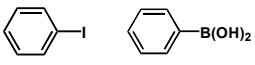
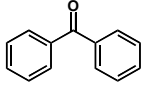
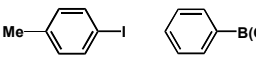
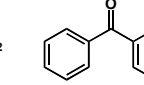
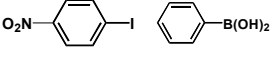
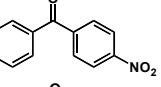
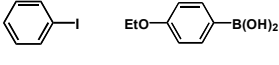
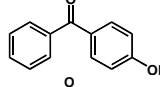
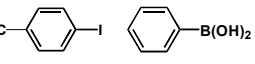
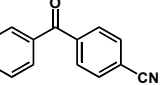
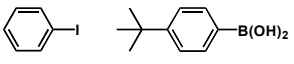
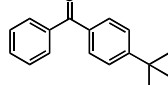
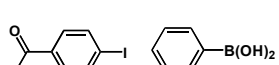
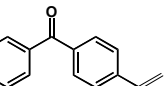
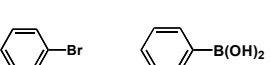
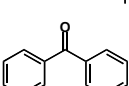
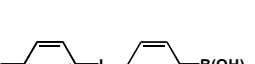
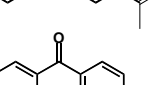
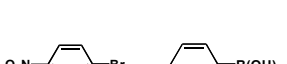
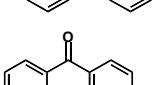
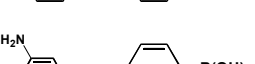
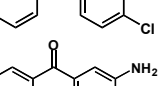
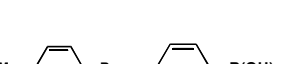
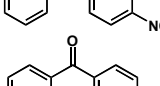
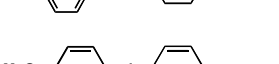
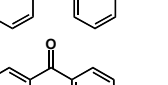
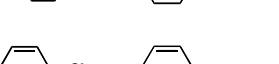
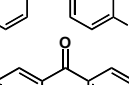
Besides aryl iodides, the Pd/SiC catalyst also catalyzed the carbonylative Suzuki coupling reactions of several aryl bromides or chlorobenzene with phenylboronic acid to produce the corresponding aromatic ketones. Unsurprisingly, however, the Pd/SiC catalyst showed lower activity towards the aryl bromides or chlorobenzene than it did towards iodobenzene because of their low reactivity (Table 2, entries 11–14).

3.4. The stability of Pd/SiC catalyst

Good recyclability is a highly desirable property for an effective heterogeneous catalyst. To test the recyclability of the Pd/SiC catalyst towards the carbonylative Suzuki coupling reaction of iodobenzene and phenylboronic acid, the catalyst was reused five times after being filtered from a completed reaction and dried. A decrease in the activity of the catalyst was found after five reaction cycles, with the conversion decreasing from 90% for the first cycle to 76% at the fifth cycle (Fig. 3(a)). Analysis of the used catalyst by TEM did not reveal any obvious changes in the morphology or aggregation of the Pd nanoparticles (Fig. 3(b)). The binding energy (BE) values of metallic Pd

Table 2

Performance of the Pd/SiC catalyst for the carbonylative Suzuki coupling reaction of various aryl halides with arylboronic acids bearing different substituents.

Entry	Reactants	Product	C/%	S/%	Entry	Reactants	Product	C/%	S/%
1			90	99	8			82	99
2			96	98	9			82	98
3			89	97	10			88	99
4			97	99	11			42	97
5			93	98	12			46	98
6			88	99	13			38	98
7			84	99	14			21	99

Reaction conditions: A mixture of aryl halide (1.0 mmol), arylboronic acid (1.0 mmol), K₂CO₃ (3.0 mmol) and 3 wt% Pd/SiC (50 mg) in anisole (100 mL) was heated at 80 °C under 1.0 MPa of CO pressure for 8 h. The conversion calculation and selectivity calculation are based on the amount of iodobenzene.

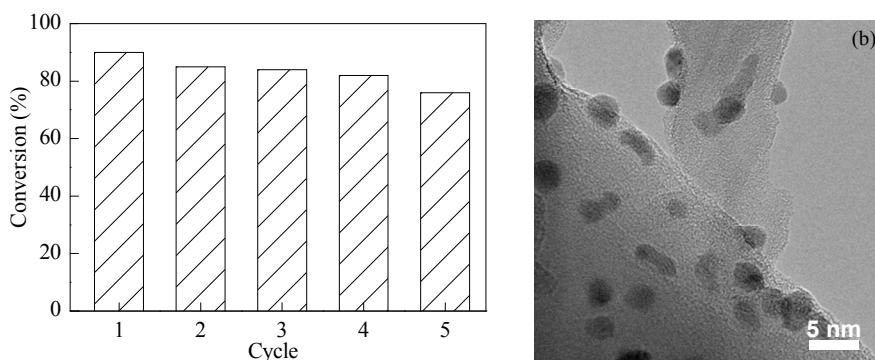


Fig. 3. Catalytic stability of the Pd/SiC catalyst for the carbonylative Suzuki coupling reaction of iodobenzene with phenylboronic acid (a), and TEM image of the catalyst after the fifth catalytic cycle (b).

are generally in the range of 334.7–335.5 eV for Pd 3d_{5/2} and 340.3–340.8 eV for Pd 3d_{3/2}. Analysis of the used Pd/SiC catalyst by XPS revealed that the Pd nanoparticles were still metallic after the fifth reaction cycle (Fig. 4). However, the results of ICP-MS analysis revealed that the Pd loading of the Pd/SiC catalyst had decreased from 2.91% to 2.37% after the fifth reaction cycle, which indicated that the loss of the active Pd phase from the Pd/SiC catalyst during the reaction process was the main reason for the observed decrease in the conversion.

4. Conclusions

High surface area SiC has been used as a support to prepare a Pd/SiC catalyst using a liquid reduction method, and the performance of the resulting catalyst was evaluated for the carbonylative Suzuki coupling reaction of aryl iodides with arylboronic acids. Analysis of the Pd/SiC catalyst by TEM showed that homogenous Pd nanoparticles were uniformly dispersed on the SiC surface with an average particle size of 2.8 nm. The influence of various reaction parameters, including the solvent, base, temperature, reaction time and CO pressure, were examined for the model reaction between iodobenzene and phenylboronic acid and the reaction condition was optimized as follows: 1.0 mmol iodobenzene, 1.5 mmol phenylboronic acid, 3.0 mmol K₂CO₃ and 3 wt% Pd/SiC in 10 mL anisole at 100 °C under 1.0

MPa of CO pressure for 8 h. Under the optimized conditions the conversion of iodobenzene reached up to 90% with 99% selectivity towards the diphenyl ketone product. The general scope of the Pd/SiC catalyst was also evaluated for the carbonylative Suzuki coupling reaction of a variety of different aryl iodides and arylboronic acids and found to be good. Stability experiments indicated that the decrease in the catalytic activity of the Pd/SiC catalyst after five reaction cycles was mainly caused by the loss of the active Pd phase from the surface of the catalyst. Overall, this new heterogeneous Pd/SiC catalyst exhibited high efficiency, high selectivity and a stable catalytic performance towards the carbonylative Suzuki coupling reaction of aryl iodides and arylboronic acids, and therefore represents an efficient and environmentally friendly approach for the synthesis of aromatic ketone.

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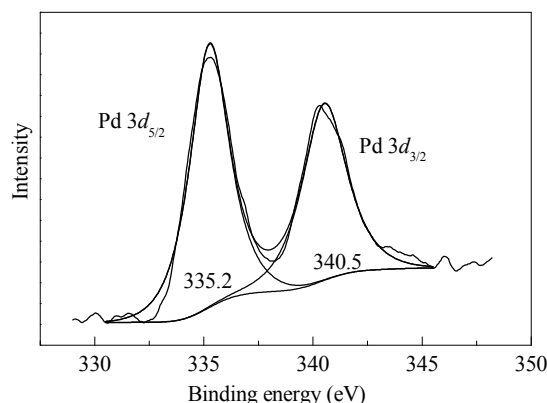


Fig. 4. XPS results for the used Pd/SiC catalyst after five rounds.

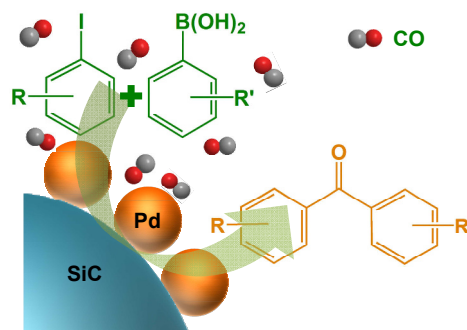
Graphical Abstract

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Carbonylative Suzuki coupling reactions of aryl iodides with arylboronic acids over Pd/SiC

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Pd/SiC as a heterogeneous catalyst exhibits both excellent activity and stability for the carbonylative Suzuki coupling reaction of various aryl iodides with arylboronic acids bearing different substituents.



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