



Palladium(II)-exchanged hydroxyapatite-catalyzed Suzuki–Miyaura-type cross-coupling reactions with potassium aryltrifluoroborates

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ABSTRACT

Palladium(II)-exchanged hydroxyapatite (PdHAP; Pd/Ca ratio = ca. 1/200) was prepared by ion-exchanging with Ca^{2+} from calcined hydroxyapatite (2.0 mmol) and $\text{Pd}(\text{NO}_3)_2$ (0.10 mmol) in water (150 mL) at 70 °C for 24 h. The PdHAP (1 mol% Pd to aryl bromides) functioned as a catalyst for the Suzuki–Miyaura-type cross-coupling reaction of aryl bromides and potassium aryltrifluoroborates with triphenylphosphine (1 mol%) and excess potassium carbonate at 50 °C in methanol under air. This cross-coupling reaction was found to proceed in water, while the rate was slow compared with that in methanol. The heterogeneous PdHAP catalyst was used ten times repeatedly at 50 °C in methanol under air, though its catalytic activity gradually declined. XRD and XRF analyses and TEM images revealed that most of palladium species would still be supported as isolated ions on the repeatedly used PdHAP, in spite of the changes of the structure and composition of PdHAP surface at the late stages in the repetitive uses and the partial leaching of Pd species at the initial stages in the repetitive uses. The gradual decline of the catalytic activity might be dependent on either the changes of PdHAP surface at the late stages or the partial leaching of Pd species at the initial stages. Still, heterogeneous PdHAP served as a reusable catalyst for Suzuki–Miyaura-type cross-coupling reaction with potassium aryltrifluoroborates.

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1. Introduction

Suzuki–Miyaura cross-coupling reaction is one of the most versatile palladium-catalyzed reactions, in addition to being one of the most useful methods for the aromatic C–C bond formation with arylmetal compounds [1]. From a viewpoint of sustainable chemistry, the reusable heterogeneous palladium catalysts such as amphiphilic polymer-supported palladium [2–4] and palladium nanoparticles [5–8] have been developed for the Suzuki–Miyaura cross-coupling reaction in water. Hydrophobic supports were recently found to be effective for the reaction with supported palladium species in water, which took place near the hydrophobic faces of supports in solution phase after leaching of palladium species [9]. No palladium species on the hydrophobic supports have been applied to those repetitive uses in the reaction.

The availability of hydroxyapatites (HAP) for supporting transition metals has been demonstrated, which must be attributed to their cation-exchange ability and adsorption capability [10,11]. Some HAP-supported transition metals such as

ruthenium, palladium, copper, and silver, have been applied as heterogeneous catalysts to organic syntheses, and have been prepared by four kinds of methods; adsorption, incorporation, ion-exchange, and nanoparticle formation [12–18]. An incorporated non-ion-exchanged HAP-supported palladium, prepared from nonstoichiometric calcium-deficient hydroxyapatite [$\text{Ca}_9(\text{HPO}_4)(\text{PO}_4)_5(\text{OH})$] and $\text{PdCl}_2(\text{PhCN})_2$ in acetone, is available as a catalyst to carbon–carbon bond-forming reactions such as Mizoroki–Heck reaction and Suzuki–Miyaura coupling reaction via the formation of arylpalladium intermediates [19]. We have previously reported that an ion-exchanged HAP-supported palladium (PdHAP; Pd/Ca = 1/200), prepared from a calcium hydroxyapatite of Ca/P = 1.67 [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] and $\text{Pd}(\text{NO}_3)_2$ in water at 70 °C for 24 h, is superior as a reusable catalyst to the incorporated non-ion-exchanged HAP-supported palladium for the allylic alkylation of allyl methyl carbonate with carbon nucleophiles such as diethyl malonate in water [20]. The palladium(II), introduced by the ion-exchange with calcium(II) in HAP matrices, could be firmly supported by the chelation of phosphate moieties of HAP, which would suppress the leaching of the palladium(II) or in situ reduced palladium(0) into the reaction medium.

Thus, for increasing the versatility of the palladium-exchanged hydroxyapatite (PdHAP) we planned an application of PdHAP,

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bearing a not so easily leaching monomeric palladium(II) component in HAP matrices, to the Suzuki–Miyaura-type cross-coupling reaction of aryl bromides with potassium aryltrifluoroborates in methanol [21]. We would also demonstrate the reusability of PdHAP in the cross-coupling reaction of either ethyl 4-bromobenzoate or 4-bromoanisole with potassium phenyltrifluoroborate.

2. Experimental

2.1. General methods

Hydroxyapatite [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] (HAP) was purchased from Taihei Chemical Industrial Co., Ltd. and was used as received or after calcining with a heating rate of $10^\circ\text{C min}^{-1}$ from room temperature to 800°C and then at 800°C for 3 h. Palladium nitrate was purchased from Furuya Metal Co., Ltd. and was used as received. Potassium bifluoride and magnesium were purchased from Kanto Chemical Co. and were used as received. 4-Chlorophenylmagnesium bromide and trimethyl borate were purchased from Sigma–Aldrich, Inc. and were used as received. Ethyl 2-bromobenzoate, ethyl 3-bromobenzoate, ethyl 4-bromobenzoate, 4-bromoanisole, 4-bromobenzonitrile, 1-bromo-4-trifluoromethylbenzene, bromobenzene, 1-bromo-4-*t*-butylbenzene and 2-bromothiophene were purchased from Sigma–Aldrich, Inc. Ethyl 4-chlorobenzoate, ethyl 4-iodobenzoate and triphenylphosphine were purchased from Wako Pure Chemical Industries, Ltd. 1-Bromo-4-chlorobenzene and 1,4-bis(diphenylphosphino)butane were purchased from Kanto Chemical Co. 1-Bromonaphthalene and 1,2-bis(diphenylphosphino)ethane were purchased from Tokyo Chemical Industry Co., Ltd. The commercial organic compounds were purified by distillation, by column chromatography or by recrystallization. Potassium aryltrifluoroborates were prepared by the reaction of the corresponding arylmagnesium bromide with trimethyl borate in THF at -70°C , followed by potassium bifluoride in H_2O at 0°C [17].

Inductively coupled plasma (ICP) analysis was carried out on a Seiko Instruments Inc. SPS7700. The textural properties of fresh PdHAP and used PdHAP were determined by nitrogen adsorption at 77 K (BEL JAPAN, BELSORP-mini). XRD measurements were performed on a RIGAKU Electronic RINT 2100/PC X-ray diffraction spectrometer equipped with a carbon monochromator and Cu $K\alpha$ (30 kV, 15 mA) irradiation, covering 2θ values between 10° and 90° . Samples were run as fine powders mounted on glass slides. X-ray fluorescence (XRF) analyses were performed on a Shimadzu energy dispersive X-ray fluorescence spectrometer EDX-720. High-resolution transmission electron microscopy (TEM) images were recorded on imaging plates (FDL-UR-V) with JEM-4010I (JEOL Ltd.) operated at 400 kV. Sample powder was suspended in chloroform, and the suspension was dropped on a carbon-coated micro-grid for TEM and then was dried.

Cross-coupling reactions were carried out on an AS ONE Co. shaking water bath SWB-17 (stroke: 30 mm; speed: 130 rpm) or on a NISSIN block shaker NX-70B (stroke: 1.5 mm; rotation rate: 220–230 rpm). TLC analyses were carried out with silica gel plates (Merck Art. 5735), and column chromatography was carried out with silica gel (Kanto Chemical Co., Inc. Cat. No. 37564). HPLC purification was carried out on a Japan Analysis Industry Co. Ltd. LC-908 (JAIGEL-2H; CHCl_3). NMR spectra were recorded on a JEOL JMS-LA300 spectrometer. IR spectra were recorded on a Shimadzu FTIR-8300 spectrometer.

2.2. Preparation of palladium(II)-exchanged hydroxyapatite (PdHAP)

A calcium hydroxyapatite (2.0 mmol) sample of $\text{Ca/P}=1.67$ [$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$] (HAP) was stirred with 150 mL of a 6.7×10^{-4} M

aqueous $\text{Pd}(\text{NO}_3)_2$ solution at 70°C for 24 h. The obtained slurry was filtered, washed with deionized water, and dried overnight at 110°C to afford a Pd(II)-exchanged hydroxyapatite (PdHAP, Pd content: 0.05 mmol g^{-1}) as a brown powder. Since no Pd(II) was detected in the filtrate or in the washing water by ICP analysis, the Pd content was estimated to be the same molar amount as that of the $\text{Pd}(\text{NO}_3)_2$ used. The specific surface area of fresh PdHAP calculated according to the B.E.T. model was $20.3 \text{ m}^2 \text{ g}^{-1}$, and the pore volume of fresh PdHAP calculated according to the Dollimore and Heal model was $0.0501 \text{ cm}^3 \text{ g}^{-1}$.

2.3. Typical procedure for Suzuki–Miyaura-type cross-coupling reaction

To a suspension of PdHAP (0.2 g, 1 mol% Pd), PPh_3 (3 mg, 0.01 mmol) and K_2CO_3 (0.41 g, 3 mmol) in methanol (1 mL) were added ethyl 4-bromobenzoate (**1a**, 0.23 g, 1.0 mmol) and potassium phenyltrifluoroborate (**2**, 0.24 g, 1.3 mmol). After the suspension was shaken at 50°C for 24 h under air in shaking water bath (AS ONE Co., SWB-17, stroke: 30 mm, speed: 130–150 rpm), the solution was separated from PdHAP by centrifugation followed by decantation. The separated PdHAP was washed with methanol ($3 \text{ mL} \times 2$), ether ($3 \text{ mL} \times 5$) and water ($3 \text{ mL} \times 1$) successively by the same operations, and was reused after being evacuated to dryness. The residue, after removing all of the organic solvents, was combined with ether (200 mL). The mixed solution was washed with water (30 mL) and brine (30 mL), and then was dried over anhydrous MgSO_4 . After evaporation of ether, purification by column chromatography (silica gel, hexane/EtOAc = 20/1) and HPLC (Japan Analytical Industry Co. Ltd., LC-908, JAIGEL-2H; CHCl_3) afforded 0.19 g (85%) of ethyl 4-phenylbenzoate (**3a**) as a colorless solid.

Ethyl 4-phenylbenzoate (**3a**) [8]: $R_f=0.43$ (n-hexane: EtOAc = 3: 1); $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 1.42 (t, $J=7.0$, 3H), 4.41 (q, $J=7.0$, 2H), 7.40–7.42 (m, 1H), 7.46–7.49 (m, 2H), 7.62–7.65 (m, 2H), 7.67 (d, 2H), 8.12 (d, 2H) ppm; $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 14.4, 61.0, 127.0, 127.3, 128.1, 128.7, 128.9, 130.1, 140.1, 145.5, 166.5 ppm; IR (KBr) 1705, 1605, 1481, 1408, 1369, 1281, 1196, 1177, 1107, 1026, 864, 752, 698 cm^{-1} .

The structures of all products were confirmed by the comparison of spectroscopic values (IR and NMR) with those of authentic samples in the literature; 4-methoxybiphenyl [8,22], 4-chlorobiphenyl [23], 4-trifluoromethylbiphenyl [5], 4-cyanobiphenyl [8,22], 4-*t*-butylbiphenyl [24], ethyl 3-phenylbenzoate [25], ethyl 2-phenylbenzoate [26], 4-chloro-4'-methoxybiphenyl [27], ethyl 4-(4-methoxyphenyl)benzoate [8,27], 4-*t*-butyl-4'-methoxybiphenyl [24], 1-phenyl-naphthalene [8,22], 2-phenylthiophene [28].

3. Results and discussion

Palladium ion-exchange to HAP (2 mmol) can be completely performed with $\text{Pd}(\text{NO}_3)_2$ (0.1 mmol) at 70°C in water (150 mL) to prepare palladium-exchanged hydroxyapatite (PdHAP, Pd content: 0.05 mmol g^{-1}) that has been convenient for the allylic alkylation of allyl methyl carbonate with carbon nucleophiles in water; the fresh PdHAP has been found to retain the structural properties of the calcined HAP [20]. According to the TEM images of the PdHAP, nanoparticles of palladium(0) were not observed either on the fresh PdHAP or on the used PdHAP. Since palladium species seemed to be pretty smaller than usual nanoparticles, those would be supported as isolated ions on the HAP surface. The catalytic activity and reusability of PdHAP were investigated for the Suzuki–Miyaura-type cross-coupling reaction of ethyl 4-bromobenzoate (**1a**) with potassium phenyltrifluoroborate (**2**). The results are summarized in Table 1. Using PPh_3 (0.01 mmol) and K_2CO_3 (3.0 mmol), the Suzuki–Miyaura-type cross-coupling

Table 1
PdHAP-catalyzed Suzuki–Miyaura-type cross-coupling reaction of **1** and **2**.

Entry	Solvent	Phosphine	Base (mmol)	3a , yield (%) ^a
1	EtOH	PPh ₃	K ₂ CO ₃ /3.0	(53) ^b
2	H ₂ O	PPh ₃	K ₂ CO ₃ /3.0	16 (68) ^b
3	MeOH–H ₂ O (1:1)	PPh ₃	K ₂ CO ₃ /3.0	47
4	MeOH	PPh ₃	K ₂ CO ₃ /3.0	85
5	MeOH	–	K ₂ CO ₃ /3.0	64
6	MeOH	dppe	K ₂ CO ₃ /3.0	10
7	MeOH	dppb	K ₂ CO ₃ /3.0	8
8	MeOH	PPh ₃	K ₂ CO ₃ /2.0	75
9	MeOH	PPh ₃	K ₂ CO ₃ /1.0	60
10	MeOH	PPh ₃	Cs ₂ CO ₃ /3.0	26
11	MeOH	PPh ₃	<i>t</i> -BuOK/3.0	Trace
12	MeOH	PPh ₃	Et ₃ N/3.0	Trace
13	MeOH	PPh ₃	–	9

^a Isolated yields.

^b The reaction was carried out for 72 h.

reaction of **1a** (1 mmol) and **2** (1.3 mmol) with PdHAP (Pd content: 0.01 mmol) proceeded in methanol (1 mL) at 50 °C for 24 h under air to produce ethyl 4-phenylbenzoate (**3a**) in a 85% yield (entry 4). The reaction proceeded without PPh₃ to produce **3a** in a 64% yield, in contrast to the allylic alkylation of allyl methyl carbonate (entry 5) [16]. Suzuki–Miyaura cross-coupling reaction can be catalyzed by palladium(0) species that are easily prepared in situ by the reduction of palladium(II) species with phosphines or starting boron compounds [1]. PPh₃ would reduce palladium(II) species to palladium(0) species in PdHAP matrices for this PdHAP-catalyzed cross-coupling reaction [29], while in the absence of PPh₃, potassium phenyltrifluoroborate (**2**) would function as a reducing agent instead of PPh₃ to afford a small amount of biphenyl. Bidentate phosphines such as 1,2-bis(diphenylphosphino)ethane (dppe)

and 1,4-bis(diphenylphosphino)butane (dppb) suppressed the reaction (entries 6 and 7). The reaction in water would become an environmentally-benign process, though the rate is slow compared with that in methanol (entry 2). K₂CO₃ was superior to other bases such as Cs₂CO₃, *t*-BuOK and Et₃N, and the reaction without K₂CO₃ slightly occurred (entries 10–13). The reaction seems to require an excess amount of K₂CO₃ to potassium phenyltrifluoroborate for improving the yield (entries 4, 8 and 9). Under the same conditions as those of entry 4 in Table 1, ethyl 4-iodobenzoate caused the cross-coupling reaction with **2** to produce **3a** in a 16% yield with some undetermined byproducts, while ethyl 4-chlorobenzoate caused no cross-coupling reaction. Thus, aryl bromides seem to be suitable as starting halides for the cross-coupling reaction.

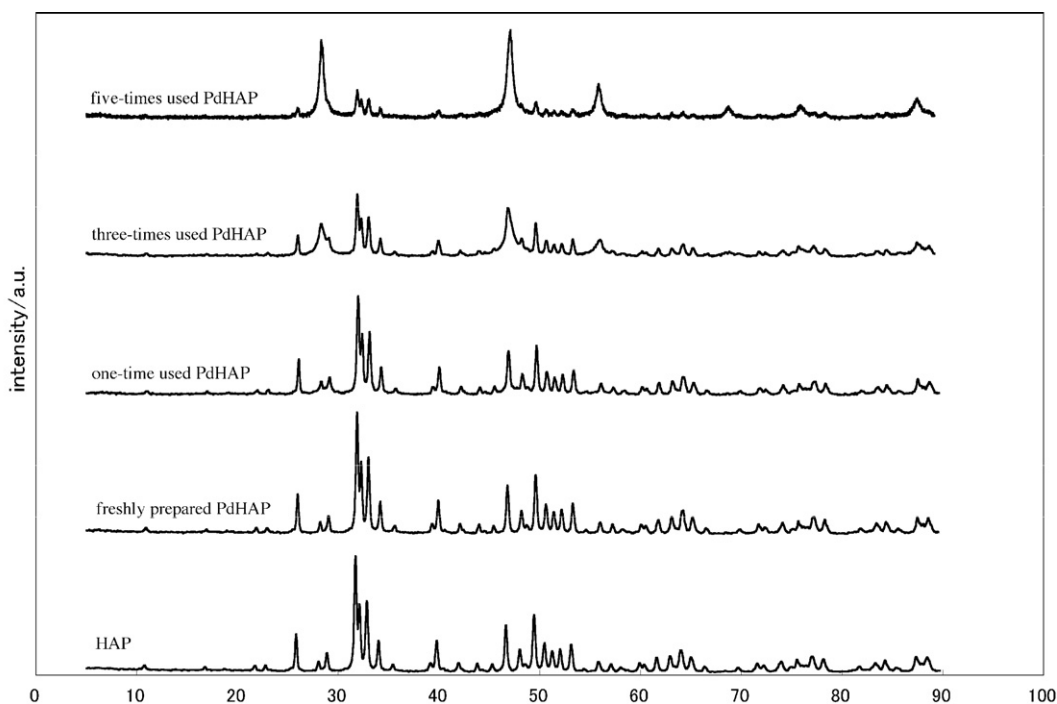


Fig. 1. XRD spectra of calcined HAP, fresh PdHAP and used PdHAPs.

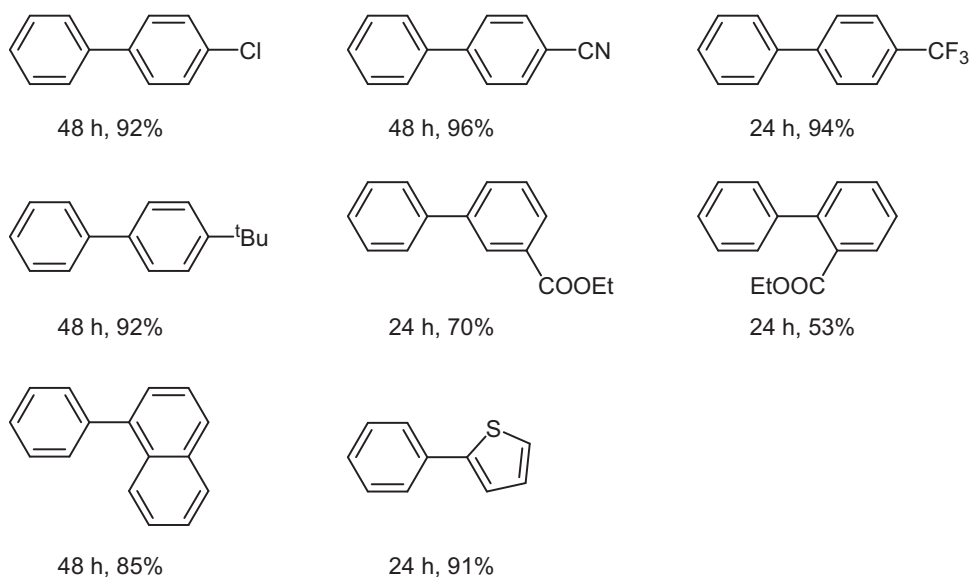
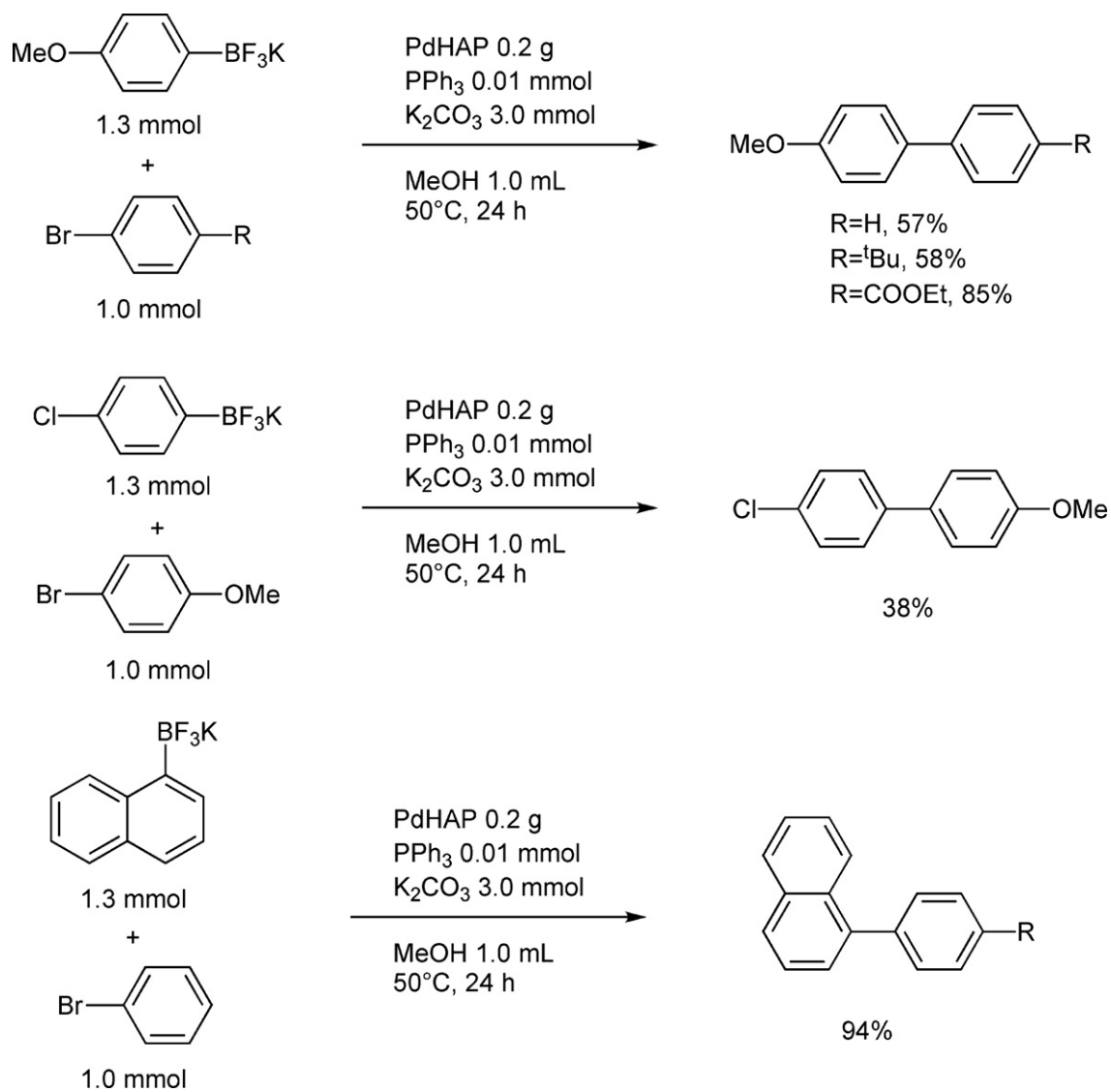


Fig. 2. PdHAP-catalyzed Suzuki–Miyaura-type cross-coupling reactions of aryl bromides and potassium phenyltrifluoroborates.



Scheme 1. Cross-coupling reaction of potassium aryltrifluoroborates.

Table 2
Reusability of PdHAP observed in 10 repetitive uses.

Repetitive use	1st	2nd	3rd	4th	5th	6th	7th	8th	9th	10th
3a , yield (%) ^a	83	82	80	77	74	77	79	79	76	74
3b , yield (%) ^a	79	77	76	73	75	64	65	61	53	52

^a Isolated yields after allowing to react at 50 °C for 24 h.

The reusability of PdHAP for the Suzuki–Miyaura-type cross-coupling reaction of either **1a** or 4-bromoanisole (**1b**) with **2** was investigated under the same conditions as those of entry 4 in Table 1 (Table 2). The catalytic activity of PdHAP for the reaction of **1a** was maintained in ten repetitive uses, while that for the reaction of **1b** declined gradually.

The participation of Pd species leaching from PdHAP was investigated during the repetitive uses as described below. To the suspension of fresh PdHAP or used PdHAP (after being used once or three times) in MeOH (1 mL) was added PPh₃ (0.01 mmol). After the suspension was shaken at 50 °C for 2 h under nitrogen atmosphere, the methanol solution was separated from the PdHAPs by filtration. The cross-coupling reaction of **1a** (1.3 mmol) and **2** (1.0 mmol) in each of the methanol filtrates including another PPh₃ (0.04 mmol) was carried out with K₂CO₃ (3.0 mmol) at 50 °C for 24 h to produce **3a** in a 20% yield (fresh PdHAP), a 5% yield (PdHAP after being used once), or a trace (PdHAP after being used either three times or five times). The results suggested that palladium species would be leached from the PdHAPs at the initial stages in the repetitive uses, whereas little leaching would occur at the late stages in the repetitive uses. Thus, apart from the reaction with fresh PdHAP, palladium species on the HAP matrix must catalyze the Suzuki–Miyaura-type cross-coupling reaction. This PdHAP-catalyzed Suzuki–Miyaura-type cross-coupling reaction would proceed similarly to usual Suzuki–Miyaura cross-coupling reactions, after the reduction of Pd(II) species on the HAP matrix to Pd(0) species with PPh₃ or **2** [1].

The variation of palladium in repeatedly used PdHAP was analyzed by X-ray fluorescence (XRF) spectrometer. The P/Ca molar ratio of repeatedly used PdHAP (after being used three times or five times) was 0.19 or 0.14 respectively, and was quite different from that of fresh PdHAP (0.45). In contrast to the variation of P/Ca molar ratios, a Pd/Ca molar ratio (0.0048 after being used three times or 0.0043 after being used five times) of repeatedly used PdHAPs did not differ much from that of fresh PdHAP (0.0049). The powder X-ray diffraction (XRD) spectra of calcined HAP, fresh PdHAP and used PdHAPs are shown in Fig. 1. The XRD peaks for fresh PdHAP were the same as those of calcined HAP. After the material had been repeatedly used, new peaks of $2\theta = 28^\circ$, 47° , 56° , 69° , 77° and 88° appeared along with the loss of the structural properties of HAP surfaces. Since the new peaks were the same as those of CaF₂, most of the surface of PdHAP at the late stages in the repetitive uses would be covered with CaF₂. Thus, the repeatedly used PdHAPs were found to keep most of the palladium species on HAP matrices, though the structure and composition of HAP moiety in the original PdHAP had changed with repeated uses.

The PdHAP-catalyzed Suzuki–Miyaura-type cross-coupling reactions of various aryl bromides (1.0 mmol) and potassium phenyltrifluoroborate (1.5 mmol) can be carried out with PPh₃ (0.01 mmol) and K₂CO₃ (3.0 mmol) in MeOH (1 mL) at 50 °C under air to produce the corresponding cross-coupling biaryl compounds in good yields. The results are summarized in Fig. 2. And furthermore, some potassium aryltrifluoroborates can be applied to the cross-coupling reactions with aryl bromides, as shown in Scheme 1.

4. Conclusions

We developed a C–C bond-forming reaction via the formation of organometallic intermediates with a reusable heterogeneous

catalyst. Palladium(II) introduced by the ion-exchange of calcium(II) into an insoluble inorganic matrix HAP functioned as a catalyst for the Suzuki–Miyaura-type cross-coupling reaction of aryl bromides with potassium aryltrifluoroborates at 50 °C in methanol under air. The heterogeneous palladium catalyst (PdHAP) was used ten times repeatedly, though its catalytic activity gradually declined. Water can also be used as a solvent, though the reaction rate in water is slower than that in methanol. XRD and XRF analyses and TEM images revealed that most of palladium species would still be supported as isolated ions on the repeatedly used PdHAP, in spite of the changes of the structure and composition of PdHAP surface at the late stages in the repetitive uses and the partial leaching of Pd species at the initial stages in the repetitive uses.

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