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Silica Tethered Pd–DABCO Complex: An Efficient and Reusable Catalyst for Suzuki–Miyaura Reaction

Arjun Kumbhar · Santosh Kamble ·
Sanjay Jadhav · Gajanan Rashinkar ·
Rajashri Salunkhe

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Abstract A palladium-based catalyst supported on DABCO-functionalized silica was successfully prepared by a facile procedure. The prepared heterogeneous catalyst showed a high activity for the Suzuki–Miyaura reaction of aryl bromides, affording excellent yield in all the cases investigated. Interestingly, the catalyst could be quantitatively recovered from the reaction mixture and recycled for five times without any significant loss in activity. Furthermore, this protocol could be extended to the palladium catalyzed synthesis of annulated pyrazines.

Keywords DABCO · Suzuki reaction · Heterogeneous catalysis · Catalyst recycling

1 Introduction

Palladium catalyzed Suzuki–Miyaura reaction is one of the most effective synthetic method used for the construction of biaryl units [1–6] which are molecular components in pharmaceuticals. They are also present in many herbicides, alkaloids like ancistrocladine as well as in engineering materials such as conducting polymers, molecular wires and liquid crystals [7–11]. Tremendous amounts of results have been accumulated for this reaction and new findings are still increasing. One of the current interests in this reaction is the development of a catalyst of high performance involving sustainable and environmentally benign reaction conditions. The Suzuki–Miyaura reaction is often mediated by palladium species in the presence of phosphine ligands in homogeneous conditions which result in the difficulty in product separation and reuse of expensive catalysts continuously. One means to circumvent this problem is the design and synthesis of new improved heterogeneous catalysts with superior activity, easy separation and facile reusability. In the last few years, numerous methods have been developed to immobilize palladium on a large variety of solid supports such as mesoporous and amorphous silica [12–15], colloidal supported nanoparticles [16], polymers [17–20], sepiolite clay [21], carbon [22], hydrotalcite [23], biopolymers [24–26], zeolite [27], and perovskite [28]. More recently, new smart materials, such as magnetic nanoparticles have emerged with great potential for catalyst recovery [29–31].

As a consequence of the need for robust heterogeneous catalysts, the development of organic modified silica has gained tremendous attention in recent years and the advent of mesoporous silicas has spurred this further. The organic groups can be robustly anchored on the surface of silica to generate catalytic sites [32]. For useful activity, modified silicas generally possess a balance of high active site loading, together with a high surface area, large pore sizes and customized particle size. This ensures access to substrates and solvents if needed and, coupled with good chemical, mechanical and thermal stabilities, covers some of the broad spectrum of performance requirements.

1,4-Diazabicyclo[2.2.2]octane (DABCO), is a cage-like, small diazabicyclic molecule with medium-hindrance and received considerable attention as an inexpensive, eco-friendly, highly reactive and non-toxic catalyst for various organic transformations. The first use of DABCO as a ligand in Pd catalyzed phosphine-free cross-coupling reaction was reported in 2004 [33]. In continuation of our work related to green chemistry [34, 35], we report herein

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A. Kumbhar · S. Kamble · S. Jadhav · G. Rashinkar ·
R. Salunkhe (✉)
Department of Chemistry, Shivaji University, Kolhapur 416004, Maharashtra, India
e-mail: rss234@rediffmail.com

silica tethered DABCO–Pd complex that constitute a highly effective system for the Suzuki–Miyaura reaction of aryl halides with aryl boronic acids.

2 Experimental

2.1 General Remarks

^{1}H NMR and ^{13}C NMR spectra were recorded on a Brucker AC (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) spectrometer using $CDCl_3$ and DMSO-d_6 as solvent and tetramethylsilane as an internal standard. Infrared spectra were recorded on a Perkin-Elmer FTIR spectrometer. The samples were examined as KBr discs ~5 % w/w. A JEOL-6360 scanning electron microscope was used for scanning electron microscopy (SEM) observations and Energy Dispersive X-ray Spectroscopy (EDS) analysis. TGA–DTA analysis was recorded on SDS Q600 N20.9 in nitrogen. Melting points were determined with a DBK melting point apparatus and are uncorrected. All the chemicals were obtained from Aldrich and were used without further purification. The 3-*n*-propyl-1-azonia-4-azabicyclo[2.2.2] octane chloride (DABCO-SIL) (1) was synthesized by following literature procedure [36].

2.2 Preparation of the Silica Tethered Pd–DABCO Catalyst (3)

2.2.1 *Synthesis of DABCO@SiO$_2$ (2)*

Mesoporous amorphous silica gel (average particle size 60–120 mesh) was activated in order to enhance the content of silanol groups on the silica surface. The silica gel (10 g) was mixed with 6 M hydrochloric acid (100 mL) and stirred at reflux temperature for 24 h. The solid product was recovered by filtration, washed with doubly distilled water to neutral pH and dried under vacuum at 80 °C for 12 h. The activated silica gel (10 g) was heated at 100 °C with DABCO-SIL (3.52 g, 10 mmol) in dry toluene for 24 h. The product was filtered off and washed with hot toluene for 12 h in continuous extraction apparatus (Soxhlet) and then dried in oven at 100 °C for overnight to give **2** (12.9 g).

2.2.2 *Synthesis of Pd–DABCO@SiO$_2$ (3)*

A mixture of **2** (2.0 g) and $Pd(OAc)_2$ (0.044 g, 0.19 mmol) in dry acetone (20 mL) was stirred at room temperature for 24 h. The solid product was filtered by suction, washed with acetone, distilled water and acetone successively and dried under vacuum at 60 °C for 4 h to give yellow palladium complex of Pd–DABCO@SiO$_2$ (**3**) (2.03 g).

2.3 General Procedure for the Suzuki–Miyaura Reaction

An oven-dried Schlenk flask, equipped with a magnetic stir bar, septum and a condenser was charged with aryl halide (1.0 mmol), arylboronic acid (1.2 mmol), K_2CO_3 (2 mmol), catalyst **3** (0.130 g, 1 mol%) and 5 mL of 95 % ethanol. The flask was immersed and stirred in an oil bath at 80 °C. Upon complete consumption of starting materials as determined by TLC analysis, catalyst was separated out by filtration and the water (20 mL) was added. The filtrate was extracted thrice with diethyl ether (10 mL). The combined organic layers were collected, dried over anhydrous Na_2SO_4 and concentrated in vacuum to afford product having sufficient purity for further characterization.

2.4 General Procedure for the Synthesis of Pyrido [2,3-*b*]pyrazine Derivatives (10 a–c)

An oven-dried Schlenk flask, equipped with a magnetic stir bar, septum and a condenser was charged with 7-bromo-2,3-diphenylpyrido[2,3-*b*]pyrazine **9** (1.0 mmol), arylboronic acid (1.2 mmol), K_2CO_3 (2 mmol), catalyst **3** (0.130 g, 1 mol%) and 5 mL of THF. The reaction mixture was refluxed for 14 h. Upon complete consumption of starting materials as determined by TLC analysis, catalyst was separated out by filtration and the water (20 mL) was added. The filtrate was extracted with diethyl ether (3 × 10 mL). The combined organic layers were collected, dried over anhydrous Na_2SO_4, concentrated in vacuum to afford product which was purified by silica gel column chromatography (eluent: *n*-hexane/ethyl acetate = 9:1).

2.5 Spectral Data for Selected Compounds

2.5.1 *4-Methyl-1,1′,4′.1″-terphenyl (Table 3, product entry 8)*

(White solid, mp 322–324 °C);^{1}H NMR ($CDCl_3$, 300 MHz): δ_H (ppm) 2.44 (s, 3H); 7.26–7.28 (m, 2H); 7.33–7.39 (m, 1H); 7.44–7.49 (m, 2H); 7.53–7.56 (m, 1H); 7.62–7.67 (m, 4H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ_C (ppm) 21.18, 126.87, 127.01, 127.21, 127.27, 127.43, 128.75, 129.50, 136.92, 137.86, 139.84, 140.06, 140.82.

2.5.2 *4-(4′-Fluoro-phenyl) benzophenone (Table 3, product entry 11)*

(White solid, mp 145–148 °C); ^{1}H NMR ($CDCl_3$, 300 MHz): δ_H (ppm) 7.13–7.21 (m, 2H); 7.48–7.53 (m, 2H); 7.58–7.66 (m, 5H); 7.82 (d, 2H, $J = 6.9$ Hz); 7.90 (d, 2H, $J = 8.1$ Hz); ^{13}C NMR ($CDCl_3$, 75 MHz): δ_C (ppm) 116.07, 126.75, 128.26, 128.84, 128.94, 129.98, 130.75, 132.29, 136.13, 136.28, 137.71, 144.12.

2.5.3 *2-Phenyl-9H-fluorene (Table 3, product entry 12)*

(White solid, mp 295–297 °C); ^{1}H NMR (CDCl$_3$, 300 MHz): δ_H (ppm) 3.97 (s, 2H); 7.29–7.38 (m, 2H); 7.40–7.47(m, 2H); 7.53–7.65 (m, 4H); 7.79 (d, 2H, J = 7.0 Hz); 7.84 (s, 1H); ^{13}C NMR (CDCl$_3$, 75 MHz): δ_C (ppm) 36.94, 119.90, 120.04, 123.76, 124.94, 125.99, 126.65, 126.79, 127.00, 127.15, 128.66, 139.95, 140.93, 141.43, 141.56, 143.23, 143.67.

2.5.4 ***10b***

(Yellow solid, mp 182–184 °C); ^{1}H NMR (CDCl$_3$, 300 MHz): δ_H (ppm) 2.41 (s, 6H); 3.80 (s, 3H); 7.31–7.40 (m, 6H); 7.45 (s, 2H); 7.55 (d, 2H, J = 7.8 Hz); 7.64 (d, 2H, J = 6.6 Hz); 8.59 (d, 1H, J = 1.8 Hz); 9.41 (d, 2H, J = 1.8 Hz); ^{13}C NMR (CDCl$_3$, 75 MHz): δ_C (ppm) 16.17, 59.46, 128.02, 128.27, 129.22, 129.43, 129.89, 130.40, 131.63, 132.10, 134.18, 134.39, 136.09, 137.91, 138.12, 138.59, 148.26, 152.89, 155.05, 156.73, 158.15; ESI–MS (M^+= 433); Elemental analyses; (Found: C = 80.4, H = 6.2, N = 9.7, O = 3.7 %; Calc. for $C_{28}H_{23}N_3O$: C = 80.3, H = 6.3, N = 9.7, O = 3.7 %).

2.5.5 ***10c***

(Yellow solid, mp 162–165 °C); ^{1}H NMR (CDCl$_3$, 300 MHz): δ_H (ppm) 7.34–7.41 (m, 6H); 7.49–7.63 (m, 6H); 7.65–7.69 (m, 2H); 7.92–7.98 (m, 3H); 8.63 (d, 1H, J = 2.7 Hz); 9.33 (d, 1H, J = 2.1 Hz); ^{13}C NMR (CDCl$_3$, 75 MHz): δ_C (ppm) 125.06, 125.41, 126.29, 127.01, 127.96, 128.14, 128.30, 128.64, 129.24, 129.38, 129.94, 130.37, 131.39, 133.96, 135.09, 135.75, 137.78, 138.10, 138.22, 138.62, 143.70, 149.06, 154.96, 156.70, 158.10; ESI–MS (M^+= 409); Elemental analyses; (Found: C = 85.1, H = 4.7, N = 10.2 %; Calc. for $C_{29}H_{19}N_3$: C = 85.0, H = 4.8, N = 10.2 %).

3 Results and Discussion

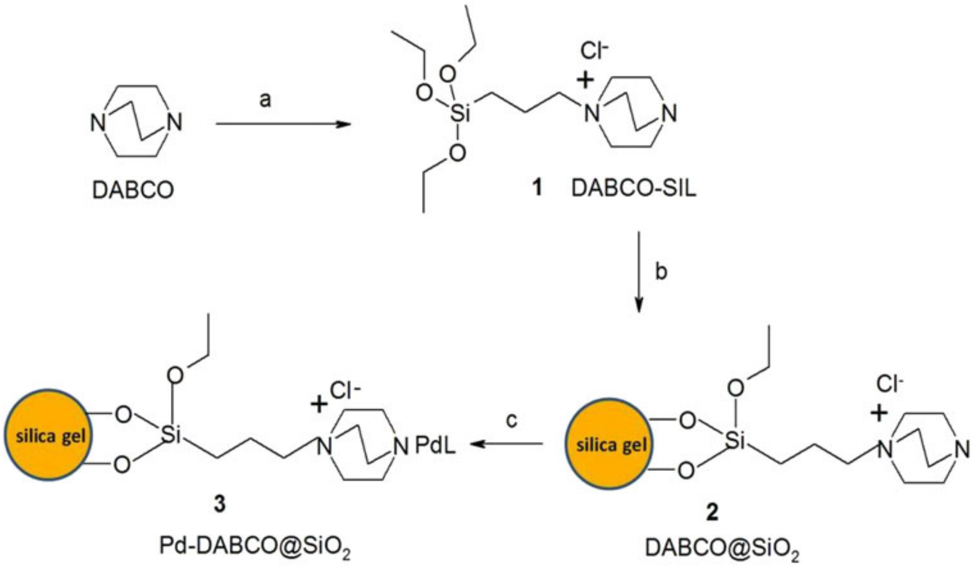
The process for preparation of the silica tethered Pd–DABCO catalyst (acronymed as Pd–DABCO@SiO$_2$) **3** is schematically described in Scheme 1.

Initially, monoquaternization of DABCO with 3-chloropropyltriethoxysilane in ethanol:acetone (1:1 v/v) mixture under reflux gave silylated DABCO-SIL **1** in 99 % yield. The hydroxyl groups on the surface of the silica gel were enriched by refluxing silica in aqueous solution of acid facilitating the surface modification. The resulting activated silica gel was then functionalized with DABCO by heating with **1** in toluene at 100 °C to afford DABCO@SiO$_2$ **2**. The reaction of **2** with Pd(OAc)$_2$ in dry acetone lead to the formation of **3**.

FT–IR spectroscopy was performed to confirm the grafting of DABCO in the matrix of silica as well as formation of **3** (Fig. 1).

FT–IR spectra are rather inconclusive, mainly due to relatively minor changes that reflect the low amounts of the anchored organic groups and palladium (II) acetate and the fact that the strongly dominant bands of the support (mainly due to O–H and Si–O bonds) remain invariant. Nevertheless, in comparison with pure silica, **2** and **3** displayed new peaks at 2,949 cm^{-1} [C–H methylene antisym. str.],

Scheme 1 Synthesis of Pd–DABCO@SiO$_2$ **3**



a) $(EtO)_3Si(CH_2)_3Cl$, EtOH:acetone (1:1), reflux, 24 h b) Silica gel, toluene, 100 °C, 24 h
c) Pd(OAc)$_2$, acetone , rt, 24 h

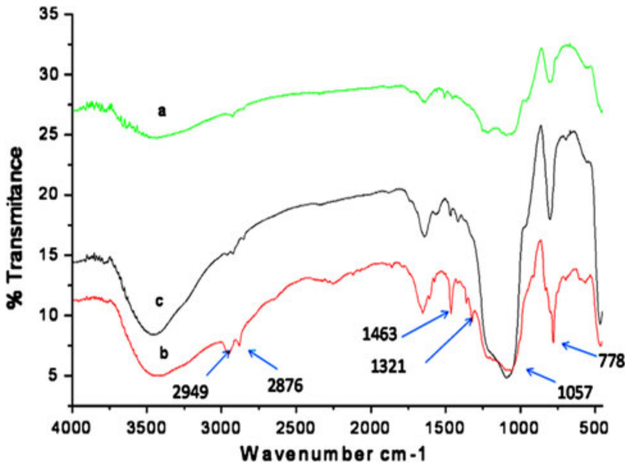


Fig. 1 FTIR spectra of (*a*) silica gel, (*b*) DABCO@SiO$_2$ and (*c*) Pd–DABCO@SiO$_2$

2,876 cm^{-1} [C−H methylene sym. str.], 1,463 cm^{-1}[$\upsilon_{\text{C–N}}$ DABCO], 1,057 and 778 cm^{-1} [$\upsilon_{\text{(asym)}}$NC$_3$ and $\upsilon_{\text{(sym)}}$NC$_3$ of DABCO]. The peaks assigned to the silica network were observed at 1,090, 809 and 472 cm^{-1} (υ Si–O–Si). These facts evidenced that the DABCO was successfully anchored on the silica surface. The formation of **3** was confirmed by detecting Pd species using energy dispersive X-ray spectroscopy (EDS) (Fig. 2).

TGA analysis was carried out to investigate the thermal stability of **3**. TGA profile of **3** is shown in Fig. 3. Weight loss is mainly divided into three temperature regions: below 160 °C, 160–420 °C and above 420 °C. Weight loss during heating from 25 to 160 °C was assigned to the loss of loosely bound water (3.4 %). The large weight loss of around 12–13 % at temperatures between 160 °C and 620 °C was due to the decomposition of covalently bonded organics. The TGA analysis of catalyst indicated that 0.85 mmol g^{-1} of the DABCO was immobilized on the silica gel. This loading was also supported by the elemental analysis of the N content of **3**.

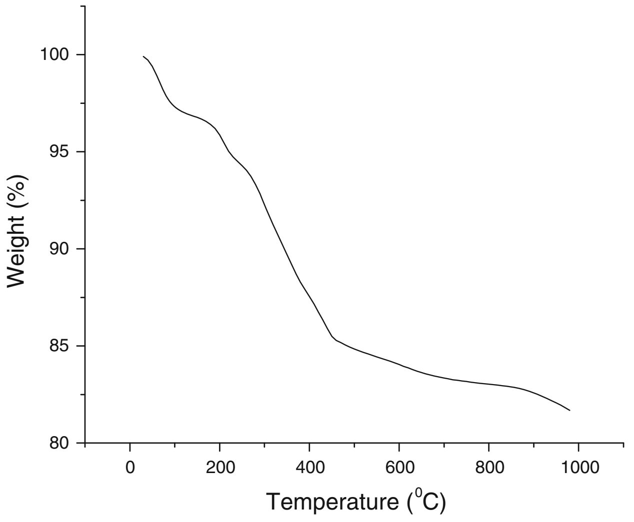


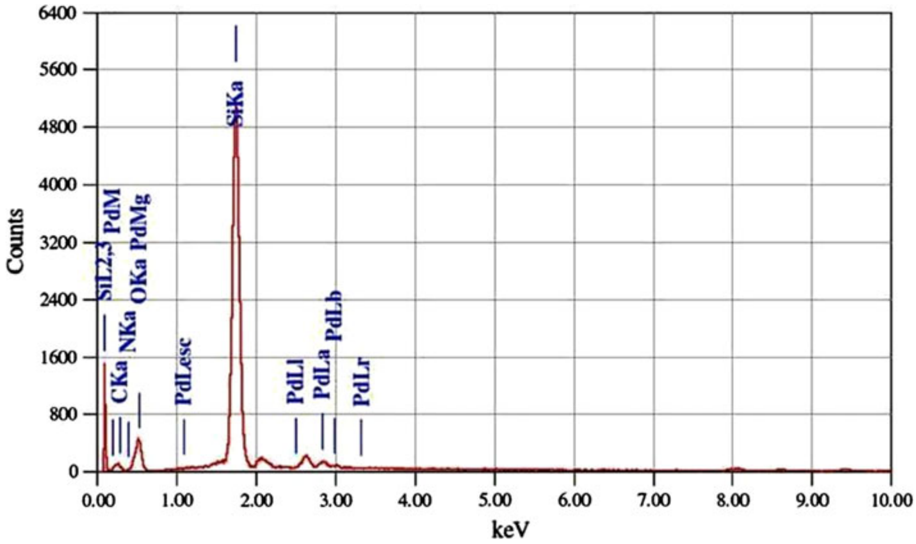
Fig. 3 TGA spectrum of Pd–DABCO@SiO$_2$

The elemental analysis revealed the presence of C, H and N content in **2** and **3**. It was found that 0.84 mmol g^{-1} of DABCO was grafted on the silica surface in **2**. The content of anchored Pd complex in **3** as determined by ICP–AES was suggested to be 0.079 mmol g^{-1} and for 0.130 g of catalyst, the loading of Pd was calculated to be 1 mol% with respect to 1 mmol of reactants.

Scanning electron microscopy studies were undertaken to observe the morphology of Pd supported on silica gel. The micrograph obtained by SEM showed the particles with irregular shape with diameters in the range of several hundreds of nanometer up to a few micrometers (Fig. 4).

To evaluate the catalytic activity of **3** in mediating the Suzuki–Miyaura reaction, coupling of bromobenzene **4** and phenyl boronic acid **5** was examined to optimize various

Fig. 2 EDS spectrum of Pd–DABCO@SiO$_2$



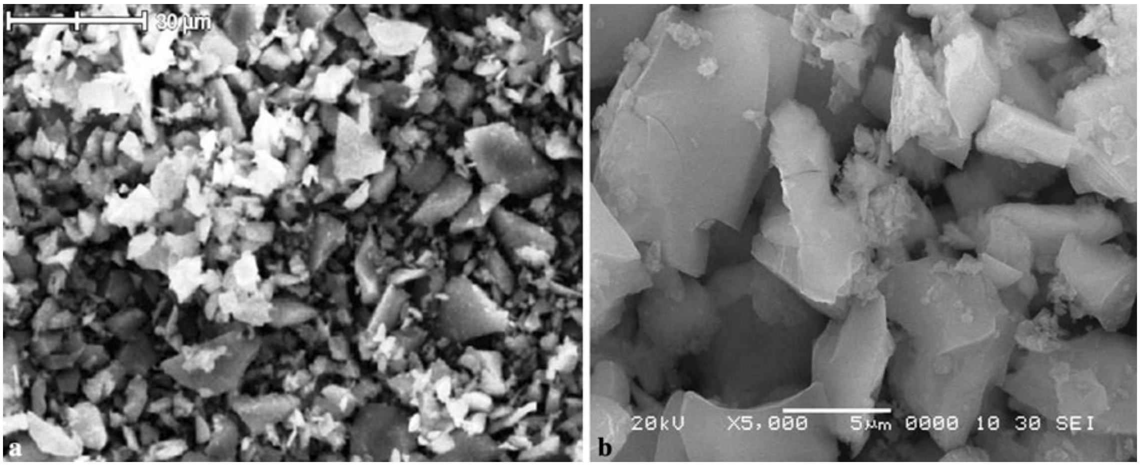
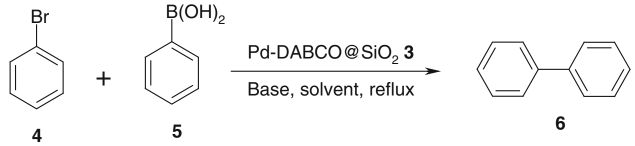


Fig. 4 Scanning electron microscopy image (**a**) normal view and (**b**) magnified view

Table 1 Optimization studies of the Suzuki–Miyaura cross-coupling reaction



Entry	Base	Solvent	Time (h)	Yield (%)[a]
1	K_2CO_3	95 % EtOH	1	96
2	NaOH	95 % EtOH	4	75
3	$K_3PO_4 \cdot 3H_2O$	95 % EtOH	2	90
4	NEt_3	95 % EtOH	4	79
5	KOH	95 % EtOH	5	72
6	K_2CO_3	EtOH	1	92
7	K_2CO_3	CH_3CN	6	70
8	K_2CO_3	DMF	3	84
9	K_2CO_3	CH_2Cl_2	6	69
10	K_2CO_3	THF	3	75

Reaction conditions: bromobenzene (1.0 mmol), phenylboronic acid (1.2 mmol), 3 (1 mol%), base (2.0 mmol), solvent (5 mL) at 80 °C under aerobic conditions

[a] Isolated yields based on the bromobenzene used

Table 2 Effect of the amount of catalyst on the Suzuki–Miyaura cross-coupling reaction

Entry	Amount of catalyst (mol%)	Time (h)	Yield (%)[a]
1	0.1	4	80
2	0.5	2	86
3	1.0	1	95
4	2.0	1	96

Reaction conditions: bromobenzene (1.0 mmol), phenylboronic acid (1.2 mmol), 3 (0.1–2.0 mol%), K_2CO_3 (2.0 mmol), 95 % ethanol (5 mL) at 80 °C under aerobic conditions

[a] Isolated yields based on the bromobenzene used

conditions like selection of solvent, base and catalyst loading. The choice of base was found to be crucial for the reduction of catalyst degradation with enhancement of the reaction rate [37]. Several organic as well inorganic bases and various solvents were screened and the superior reaction efficiency was observed with K_2CO_3 as base in 95 % ethanol at 80 °C (Table 1). The other bases and solvent were not as effective as K_2CO_3. There are various reports [38, 39] emphasizing the important role played by the solvent in the reaction system prompted us to screen various solvents. Screening of solvents indicated that 95 % ethanol was the best solvent for this system. Use of 95 % ethanol renders this protocol quite practical and amenable to large scale synthesis.

In order to evaluate the potential of the catalyst, the standard reaction between **4** and **5** was run with varying catalyst amount and the results are shown in Table 2. The results showed that among the different loading 1 mol% proved to be the best and was chosen as the optimum loading in the Suzuki–Miyaura cross-coupling reaction.

To explore the scope and generality of the catalyst, the optimized protocol [aryl bromide (1 equiv.), aryl boronic acid (1.2 equiv.), K_2CO_3 (2 equiv.), catalyst (1 mol%), temperature 80 °C under air] was applied for reactions of variety of differently substituted and non-substituted aryl halides and aryl boronic acids (Table 3). Both activated and deactivated aryl bromides were completely converted to the corresponding products in 70–96 % yields within 1–4 h. The reaction showed good diversity in the presence of both electron withdrawing and donating groups. It has been found that 1–4 h are required for full conversion of aryl bromides, but a much longer reaction time as 12–15 h was required for less reactive aryl chlorides giving low yield of the coupling product (Table 3, entries 18–21). For

Table 3 The Suzuki–Miyaura reaction of various aryl halides and arylboronic acids over Pd–DABCO@SiO_2

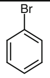
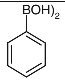
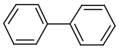
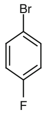
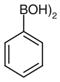
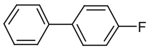
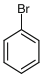
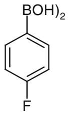
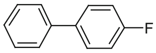
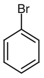
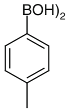
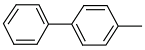
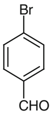
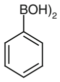
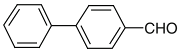
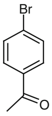
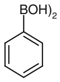
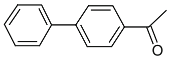
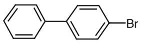
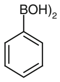
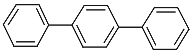
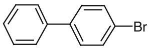
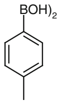
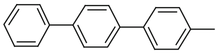
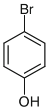
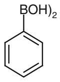
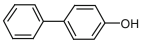
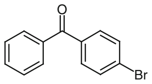
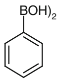
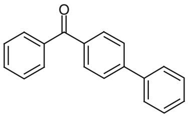
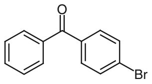
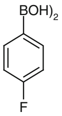
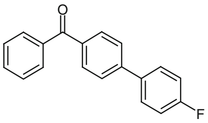
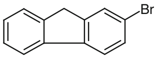
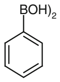
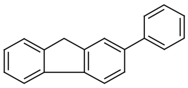
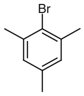
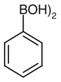
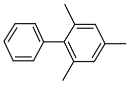
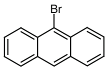
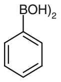
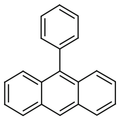
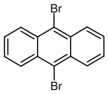
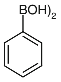
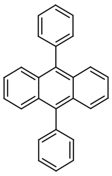
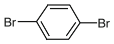
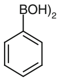
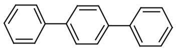
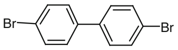
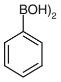
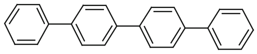
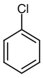
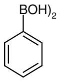
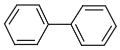
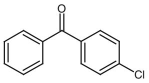
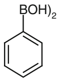
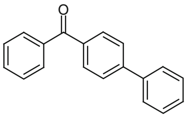
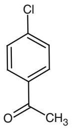
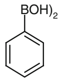
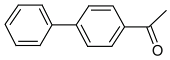
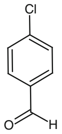
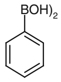
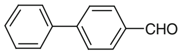
Entry	Aryl halide	Arylboronic acid	Product	Time (h)	Yield[a] (%)
1				1	96
2				1	94
3				1	90
4				1.5	92
5				2	90
6				2	89
7				1.5	93
8				1.5	92
9				2	70
10				2	94
11				2	91
12				2	93

Table 3 continued

Entry	Aryl halide	Arylboronic acid	Product	Time (h)	Yield[a] (%)
13				16	70
14				4	88
15				4	81
16				2	92
17				3	80
18				12	76
19				14	20
20				15	58
21				15	60

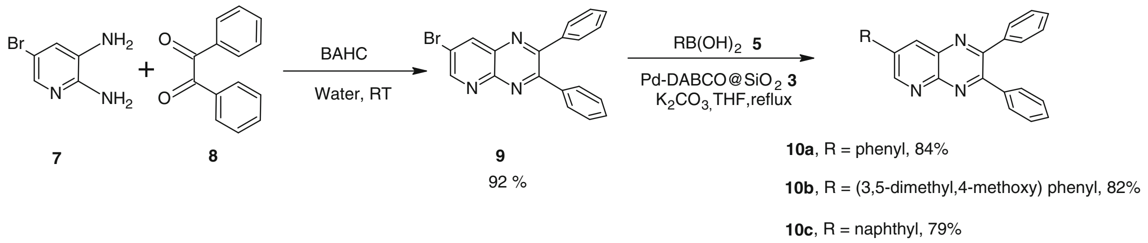
Reaction conditions: aryl halide (1.0 mmol), arylboronic acid (1.2 mmol), 3 (1 mol%), K_2CO_3 (2.0 mmol), 95 % EtOH (5 mL) at 80 °C under aerobic conditions

[a] Isolated yields based on aryl halide

the highly sterically hindered, deactivated 2,4,6-trimethylbromobenzene, a satisfactory yield could be obtained within 16 h (70 %, Table 3, entry 13).

To further extend the scope of this methodology, we carried out reactions of dibromoarenes with phenyl boronic acid. The results of these reactions were of particular interest since their outcome involves formation of products which are extensively used in the synthesis of molecular wires. The reactions of dibromoarenes with aryl boronic acids afforded desired products in quantitative yields (Table 3, entries 16 and 17). Diphenylanthracene which is used as a fluorescer in a peroxyoxalate chemiluminescence system [40] was also obtained by coupling 9,10-dibromoanthracene with phenylboronic acid (Table 3, entry 15) in 81 % yield.

The protocol was extended for the synthesis of annulated pyrazines such as pyrido[2,3-*b*]pyrazine derivatives



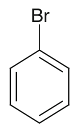
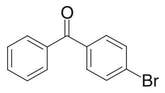
Scheme 2 Suzuki–Miyaura reaction between 7-bromo-2,3-diphenylpyrido[2,3-*b*]pyrazine and arylboronic acids using Pd–DABCO@SiO_2

10a–c (Scheme 2) as these are part of important anticancer drugs like CQS (chloroquinoxalinesulphonamide) and XK469 as well as present in a number of synthetic antibiotics such as echinomycin. They are also recognized in a great number of naturally occurring compounds and many fluorescent materials [41–43]. The key precursor 7-bromo-2,3-diphenylpyrido[2,3-*b*]pyrazine **9** required for the synthesis of **10a–c** was obtained in 92 % yield from commercially available 5-bromo-2,3-diaminopyridine **7** and benzil **8** in water in the presence of Brönsted acid hydrotrope combined catalyst [44]. In order to substitute functionalized aromatic ring at position **7** of **9**, Suzuki–Miyaura reactions (Scheme 2) were carried out in presence of **3** with various aryl boronic acids **5**. Though we were able to effect these reactions, the yields of products **10a–c** were very scarce in ethanol. This could be rationalized on the basis of less solubility of **9** in ethanol. However, switching of solvent from ethanol to THF resulted in excellent yields of **10a–c** in 14 h.

In order to evaluate the heterogeneity of catalyst, hot filtration test was performed by coupling bromobenzene with phenylboronic acid in presence of the catalyst [45]. After the 30 min of reaction time, the catalyst was filtered off and the filtrate was allowed to react further. It was found that, after this hot filtration, no further reaction was observed. This result suggests that the palladium catalyst remains on the support during the reaction.

For practical applications of heterogeneous systems, the lifetime of the catalyst and its level of reusability are very important features. The recyclability of **3** was investigated with consecutive Suzuki–Miyaura reactions of bromobenzene and 4-bromobenzophenone with phenyl boronic acid. After first cycle, the catalyst was recovered by filtration and extensively washed with water, dichloromethane, and acetone. The catalyst was then dried under vacuum overnight before performing the reusability test. The recycling results of **3** are summarized in Table 4. Investigations of the catalyst recycling mirrored a slight decrease of the total yield in every run. After reaction the amount of Pd in catalyst was measured by ICP–AES and it was found to be 0.075 mol g^{-1}. There was 5–6 % weight loss of catalyst observed after the five repeated runs. Additionally, the catalyst exhibited high stability in air. It was found that there was no decrease in activity when the catalyst was exposed to air for several days.

Table 4 Recycling of catalyst **3** in Suzuki–Miyaura coupling of aryl halide with phenyl boronic acid

Entry	Aryl halide	Cycle	Time (h)	Yield (%)
1		1	1	96
		2	1	94
		3	1.5	95
		4	2.0	93
		5	2.5	90
2		1	2	94
		2	2.5	92
		3	2.5	91
		4	3	89
		5	4	87

4 Conclusions

In conclusion, silica tethered DABCO–Pd complex was successfully synthesized by immobilization of DABCO–Pd on 3-chloropropyltriethoxysilane modified silica gel by covalent bonding and efficiently used as heterogeneous catalyst for Suzuki–Miyaura reactions involving various mono and dibromoarenes. The catalyst showed interesting features such as low Pd leaching, high efficiency and economy gain by simple reaction processing and easy recovery and re-use of the catalyst. Moreover, the catalyst showed excellent catalytic activity for the synthesis of pyrido[2,3-*b*]pyrazines.

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