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Sulfonated palladium(II) *N*-heterocyclic carbene complex immobilized on nano–micro size poly(4-vinylpyridinium chloride) for Suzuki-Miyaura cross-coupling reaction

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The sulfonated palladium(II) *N*-heterocyclic carbene complex Pd^{II}(NHC)SO$_3^-$, supported on poly(4-vinylpyridinium chloride), was used as a heterogeneous, recyclable and active catalyst for the Suzuki–Miyaura reaction. This catalyst was applied for coupling of various aryl halides with phenylboronic acid and the corresponding products were obtained in excellent yields and short reaction times. The catalyst was characterized using Fourier transform infrared and diffuse reflectance UV–visible spectroscopies, scanning electron microscopy and elemental analysis. After each reaction, the catalyst was recovered easily by simple filtration and reused several times without significant loss of its catalytic activity. Copyright © 2015 John Wiley & Sons, Ltd.

Keywords: palladium(II) complex; N-heterocyclic carbene; heterogeneous catalyst; polyvinylpyridine; C–C coupling reactions

Introduction

Recently, *N*-heterocyclic carbenes (NHCs) have attracted much attention because of their application as versatile ligands in homogeneous transition metal catalysis. This is due to the strong σ-donor properties, ease of preparation and effective binding ability to any transition metal.[1–7] NHC complexes as homogeneous catalysts have been used in a number of organic transformations such as olefin metathesis and C–C and C–N bond formation reactions.[8–19] Therefore, the discovery of catalytic properties of Pd–NHC complexes in the Mizoroki–Heck and Suzuki–Miyaura cross-coupling reactions is of great importance since these reactions have very many applications in the synthesis of natural products, numerous drugs and high-performance modern organic materials.[20–25] Despite the wide application of these homogeneous catalysts in organic transformations, their recycling is complicated and their separation is very difficult. Moreover, in consequence of the toxicity of palladium residuals, acceptable limits of palladium traces in pharmaceuticals are set usually at the parts per million level. Application of supported catalysts can solve this problem. In this regard, polymer-supported catalysts have received much attention due to the stability and uniformity of the obtained materials, and the possibility of easier separation of product and catalyst, combining the advantages of homogeneous and heterogeneous catalysis. For instance, the immobilization of Pd–NHCs on polystyrene[26] or silica[27,28] has been reported. Such systems have several advantages such as simple recycling of the catalysts by filtration, preventing the loss of both ligands and (heavy) metal, thus considerably decreasing the environmental problems of waste materials compared to homogeneous catalysis.[29,30]

Poly(4-vinylpyridine) (4-PVP) and poly(2-vinylpyridine) (2-PVP) have been known for many years and have found several industrial as well as laboratory applications.[31,32] Considering such novel chemical properties as acidity, basicity and hydrophilic–hydrophilic balance, 4-PVP is of great importance.[33] The polymers and copolymers of 4-vinylpyridine are intriguing materials with many applications such as in sensors and actuators, host–ligand of metal-containing chromophores, antimicrobial materials, and so on.[34–36] PVP is an attractive polymer for immobilization of nanoparticles because of the strong affinity of the pyridyl group to metals and because of its ability to undergo hydrogen bonding with polar species. Although cross-linked pyridinic resins are hydrophilic, their water affinity can be increased by the quaternization of pyridine.[34,37] In addition, PVP can interact electrostatically in quaternized or protonated forms with charged surfaces.[38–40]

Any ion-exchange resin should have two features: high ion-exchange capacity and fast ion-exchange kinetics. These characteristics could be obtained in network materials as a result of the functional groups present in the chemical structure and of their porosity.

In continuation of our previous work using supported catalysts in organic synthesis,[41–45] here we report the preparation and characterization of the sulfonated palladium(II) NHC complex Pd^{II}(NHC)SO$_3^-$, supported on poly(4-vinylpyridinium chloride), as a heterogeneous catalyst in Suzuki-Miyaura C–C coupling reactions (Scheme 1).

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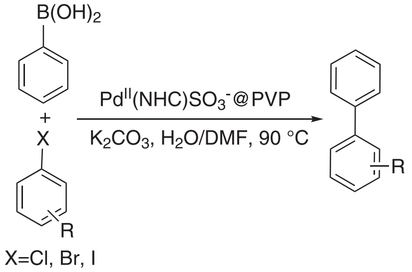


Scheme 1. Suzuki-Miyaura C–C coupling reaction catalysed by $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP.

Experimental

General remarks

All the reagents were of analytical grade and used without further purification. The chemicals used in this work were purchased from Merck, Fluka and Sigma-Aldrich. Fourier transform infrared (FT-IR) spectra were recorded with a JASCO 6300 spectrophotometer. ^{1}H NMR spectra were recorded with a Bruker Avance 400 MHz spectrometer using DMSO-d_6 as solvent. The morphological features were investigated using a Hitachi S-4700 field emission scanning electron microscopy (SEM) instrument. The Pd content of the catalyst was determined using inductively coupled plasma (ICP) analysis (PerkinElmer). Substances were identified and quantified using GC with an Agilent GC 6890 equipped with a 19096C006 80/100 WHP packed column and a flame ionization detector. Diffuse reflectance UV–visible (DR UV–vis) spectra were recorded with a JASCO V-670 spectrophotometer. The $\mathrm{Pd^{II}(NHC)SO_3^-}$ complex was prepared according to a literature method.[46] The protonation of 4-PVP was carried out according to a literature method.[47]

Immobilization of $\mathrm{Pd^{II}}$(NHC) on poly(4-vinylpyridinum chloride): $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP

First, commercially obtained 4-PVP was protonated by reaction with excess HCl (1 N). The resulting protonated polymer was repeatedly washed with deionized water to ensure complete removal of the unreacted free acid from the gel. To a solution of $\mathrm{Pd^{II}(NHC)SO_3^-}$ (500 mg) in water (50 ml), poly(4-vinylpyridinum chloride) (5 g) was added and the mixture stirred for 24 h at room temperature. At the end of the reaction, the catalyst, $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP, was filtered, washed successively with dimethylformamide (DMF), water and acetone, and dried under vacuum. CHNS analysis (%): C, 73.02; H, 6.42; N, 11.46; S, 0.31.

General procedure for Suzuki-Miyaura cross-coupling reaction of aryl halides with phenylboronic acid in presence of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP

A mixture of aryl halide (1 mmol), arylboronic acid (1.2 mmol), K_2CO_3 (207 mg, 1.5 mmol) and catalyst (0.096 mol%) in 4 ml of DMF–H_2O (2:1) was stirred at 90°C under air atmosphere. The progress of the reaction was monitored using GC. After completion of the reaction, ethyl acetate (15 ml) was added and the catalyst was filtered. The organic phase was washed with H_2O (2 × 10 ml), dried over anhydrous $MgSO_4$ and evaporated. The residue was recrystallized from ethyl acetate and ether (1:3) to afford the pure product.

Results and discussion

Characterization of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP

The preparation route for the catalyst is shown in Scheme 2. As can be seen, the sulfonated $\mathrm{Pd^{II}}$(NHC), $\mathrm{Pd^{II}(NHC)SO_3^-}$, can be immobilized on poly(4-vinylpyridinium chloride) via electrostatic interactions, producing the heterogeneous $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP catalyst.

The prepared catalyst was characterized using elemental and ICP analyses, SEM, and DR UV–vis and FT-IR spectroscopies.

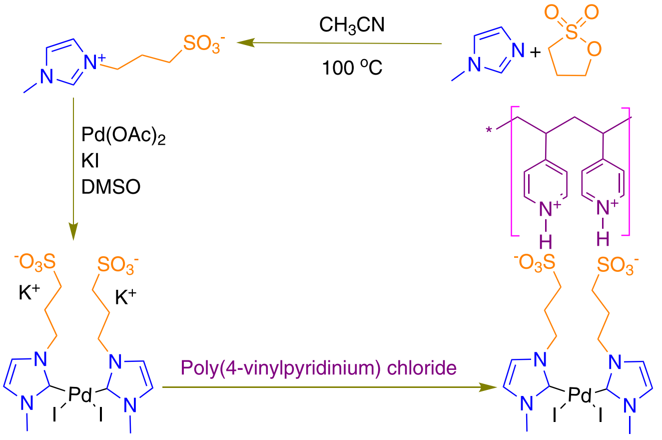
The DR UV–vis spectrum of the homogeneous catalyst shows an absorption peak at 322 nm (Fig. 1(A)), while for PVP two absorption peaks appear at 280 and 248 nm (Fig. 1(B)). The spectrum of the catalyst shows the characteristic peaks of the homogeneous catalyst and also the support. In the DR UV–vis spectrum of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP, the absorption peaks appear at 323, 282 and 249 nm (Fig. 1(C)). These observations clearly confirm that the $\mathrm{Pd^{II}(NHC)SO_3^-}$ complex is supported on poly(4-vinylpyridinum chloride).

The FT-IR spectrum of the parent $\mathrm{Pd^{II}(NHC)SO_3^-}$ shows characteristic SO vibrational bands at 1051 and 1187 cm^{-1}. These bands also appear in the FT-IR spectrum of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP, which confirms the supporting of $\mathrm{Pd^{II}(NHC)SO_3^-}$ on PVP (Fig. 2). Also, the presence of sulfur in the elemental analysis of the catalyst is a good indication for the immobilization of $\mathrm{Pd^{II}(NHC)SO_3^-}$ on PVP.

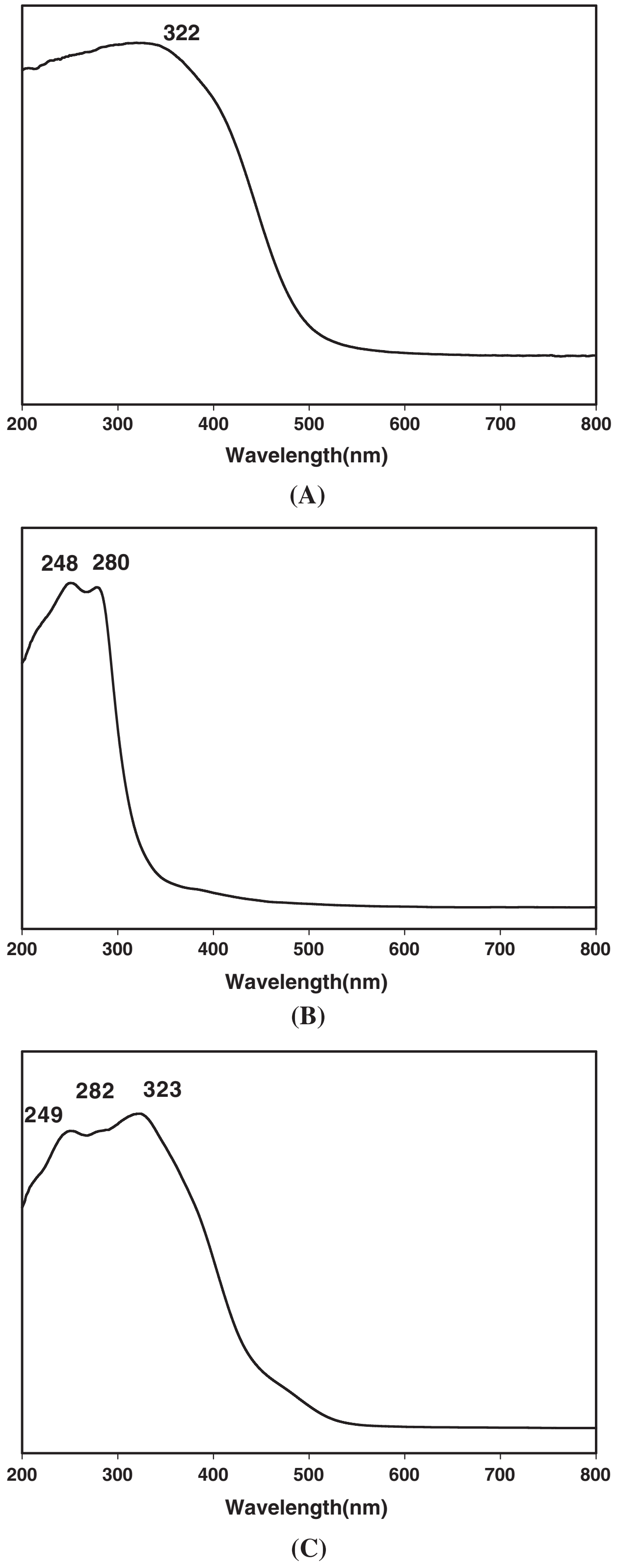
The SEM images of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP show that the PVP particles are aggregated as spherical particles (Fig. 3). The histogram of particle size distribution shows that about 78% of PVP particles have sizes less than 100 nm (Fig. 3(C)). The Pd content of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP, determined using ICP analysis, is 0.046 mmol g^{-1}.

Suzuki–Miyaura cross-coupling of aryl halides with phenylboronic acid in presence of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP

After preparation and characterization of the catalyst, its catalytic activity was investigated in the Suzuki–Miaura C–C coupling reaction. First, the reaction parameters such as kind of base and solvent, temperature and catalyst amount were optimized in the reaction of 4-iodoanisole (1 mmol) with phenylboronic acid (1.2 mmol). The progress of the reaction was monitored using GC. The results are summarized in Table 1. The results show that the optimized amount of catalyst is 0.00096 mmol (0.01 mol%) of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP (Table 1, entries 1–4). This coupling reaction is found to be highly sensitive to the reaction temperature. The highest yield is obtained at 90°C (Table 1, entries 3 and 5–7). When the model reaction is carried out in various aqueous–organic media, the reaction is more



Scheme 2. Preparation of $\mathrm{Pd^{II}(NHC)SO_3^-}$@PVP catalyst.



(A)

(B)

(C)

Figure 1. DR UV–vis spectra: (A) $Pd^{II}(NHC)SO_3^-$; (B) PVP; (C) $Pd^{II}(NHC)SO_3^-$@PVP.

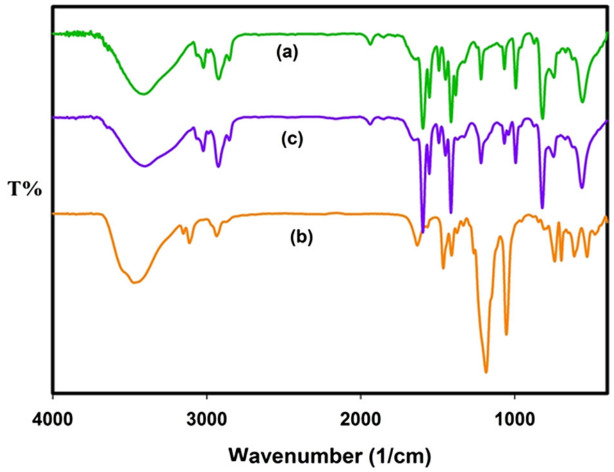


Figure 2. FT-IR spectra: (a) PVP; (b) $Pd^{II}(NHC)SO_3^-$; (c) $Pd^{II}(NHC)SO_3^-$@PVP.

effective in DMF–H_2O mixture (2:1). When EtOH–H_2O mixture is used as solvent, lower yield of the desired cross-coupling product is obtained, while in toluene–H_2O the product yield is very low. Therefore, all reactions are carried out in DMF–H_2O mixture (Table 1, entries 3, 11 and 12). When the model reaction is carried out in the absence of base, no appreciable amount of product is produced. Therefore, the presence of a base is crucial in these reactions. Various organic and inorganic bases such as NEt_3, Na_3PO_4 and K_2CO_3 were applied in the model reaction. Among them, K_2CO_3 is found to be the most efficient base. Comparison of inorganic bases utilized shows that carbonate base is more stable than the other inorganic base, and an organic base like Et_3N is not as efficient as K_2CO_3 (Table 1, entries 3, 8–10). Therefore, it is concluded that the optimum reaction conditions involve 4-iodoanisole (1 mmol), phenylboronic acid (1.2 mmol), K_2CO_3 (1.5 mmol) and $Pd^{II}(NHC)SO_3^-$@PVP (0.096 mol% Pd) in DMF–H_2O (2:1, 4 ml) at 90°C under air (Table 1, entry 3).

The scope and generality of this catalytic system was examined in the Suzuki–Miyaura cross-coupling of aryl halides with phenylboronic acid. Under the optimized reaction conditions, vatious aryl halides were reacted with phenylboronic acid to produce substituted biphenyls (Table 2). Various aryl iodides, bromides and chlorides bearing electron-donating and electron-withdrawing groups react efficiently with phenylboronic acid and the desired cross-coupling products are produced in high yields. The results show that the electronic properties of the substituents on the aromatic rings of aryl halide have no significant influence on the reaction. As expected, aryl iodides are found to be more reactive than aryl bromides and chlorides.

The catalytic activity of homogeneous $Pd^{II}(NHC)SO_3^-$ was also investigated in the model reaction. The results show that in the reaction involving 4-iodoanisole (1 mmol), phenylboronic acid (1.2 mmol), K_2CO_3 (1.5 mmol) and $Pd^{II}(NHC)SO_3^-$ (0.096 mol% Pd) in DMF–H_2O (2:1, 4 ml) at 90°C under air, the amount of desired product is 100% after 12 min. The main disadvantage of homogeneous $Pd^{II}(NHC)SO_3^-$ is its solubility in the reaction mixture, and it cannot be recovered and reused.

Catalyst recovery and reuse

Since the homogeneous catalyst cannot be recovered and reused even once, the reusability of the heterogeneous catalyst was investigated in the reaction of 4-iodoanisole with phenylboronic acid in the presence of K_2CO_3. At the end of each reaction, the catalyst was filtered and washed thoroughly successively with acetone, diethyl ether and water, and reused with fresh 4-iodoanisole and phenylboronic acid. The results are summarized in Table 3. As can be seen, the catalyst can be recovered and recycled five consecutive

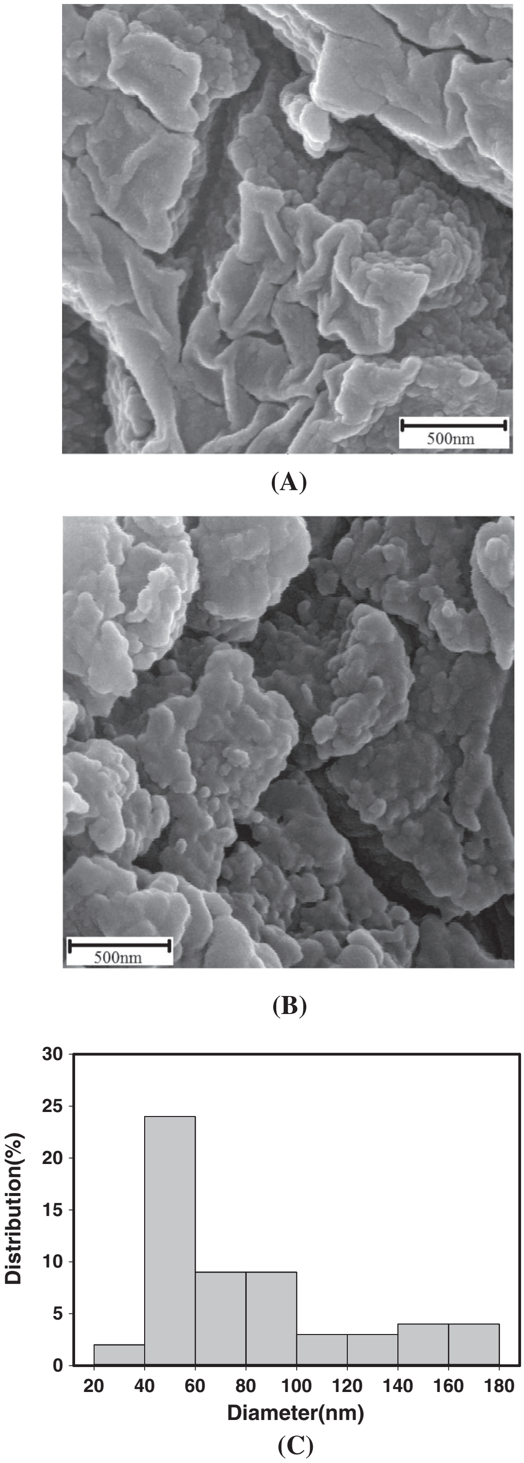


Figure 3. (A, B) SEM images of $Pd^{II}(NHC)SO_3^-@PVP$. (C) Histogram of particle size distribution of catalyst.

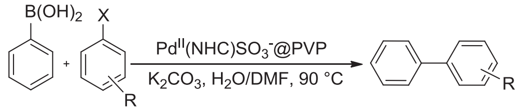
Table 1. Optimization of reaction conditions in the Suzuki-Miyaura reaction of 4-iodoanisole with phenylboronic acid catalysed by $Pd^{II}(NHC)SO_3^-@PVP$[a]

Entry	Solvent	Base	Pd (mol%)	Temperature (°C)	Yield (%)[b]
1	DMF–H_2O (2:1)	K_2CO_3	0	90	0
2	DMF–H_2O (2:1)	K_2CO_3	0.048	90	70
3	DMF–H_2O (2:1)	K_2CO_3	0.096	90	97
4	DMF–H_2O (2:1)	K_2CO_3	0.144	90	98
5	DMF–H_2O (2:1)	K_2CO_3	0.096	Room temp.	16
6	DMF–H_2O (2:1)	K_2CO_3	0.096	50	43
7	DMF–H_2O (2:1)	K_2CO_3	0.096	110	98
8	DMF–H_2O (2:1)	Na_3PO_4	0.096	90	73
9	DMF–H_2O (2:1)	NEt_3	0.096	90	25
10	DMF–H_2O (2:1)	No base	0.096	90	6
11	EtOH–H_2O (2:1)	K_2CO_3	0.096	90	81
12	Toluene–H_2O (2:1)	K_2CO_3	0.096	90	45

[a] Reaction conditions: 4-iodoanisole (1 mmol), phenylboronic acid (1.2 mmol), base (1.5 mmol), solvent (4 ml) under air atmosphere.

[b] GC yield after 100 min.

Table 2. Suzuki-Miyaura cross-coupling reaction of aryl halides and phenylboronic acid catalysed by $Pd^{II}(NHC)SO_3^-@PVP$[a]



Entry	R	X	$Pd^{II}(NHC)SO_3^-@PVP$[b]	
			Time (h)	Yield (%)[c]
1	H	I	1.55	98
2	4-Me	I	1.6	97
3	4-MeO	I	1.67	97
4	4-NO_2	I	1.55	99
5	H	Br	5	93
6	4-MeO	Br	4	87
7	4-NO_2	Br	3.4	93
8	4-Ac	Br	4	99
9	H	Cl	12	68
10	4-Ac	Cl	4	99
11	4-MeO	Cl	12	31
12	4-NO_2	Cl	11	97

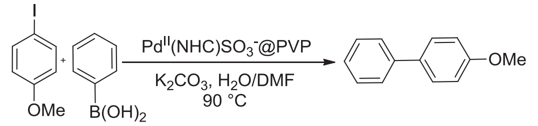
[a] Reaction conditions: aryl halide (1 mmol), phenylboronic acid (1.2 mmol), K_2CO_3 (1.5 mmol), DMF–H_2O (2:1, 4 ml) under air.

[b] 90°C, catalyst (0.096 mol% Pd).

[c] Determined using GC.

times. The yield for the fifth run is 88% for the heterogeneous catalyst. The amount of Pd leached was determined using ICP analysis. As evident from Table 3, no Pd is detected in the filtrates after the first two runs. The nature of the recovered catalyst was studied using FT-IR spectroscopy. The FT-IR spectrum of the recovered catalyst is the same as that of the fresh catalyst (Fig. 4). A probable reaction is that the K_2CO_3 deprotonates the pyridinium units in the PVP polymer, and then the palladium catalyst attaches to the pyridines. The absence of a band corresponding to Pd–N_{Py} vibration (418 cm^{-1}) in the FT-IR spectrum of the recovered catalyst confirms that the catalyst retains its initial structure during the catalytic reactions.[48]

Table 3. Investigation of catalyst recycling and reuse in the Suzuki-Miyaura reaction[a]



Entry	Pd^{II}(NHC)SO$_3^-$@PVP[b]	
	Yield (%)[c]	Pd leached (%)[d]
1	97	4.23
2	92	3.31
3	92	—
4	90	—
5	88	—

[a] Reaction conditions: 4-iodoanisole (1 mmol), phenylboronic acid (1.2 mmol), K$_2$CO$_3$ (1.5 mmol), DMF–H$_2$O (2:1, 4 ml) under air.

[b] 90°C, catalyst (0.096 mol% Pd), 100 min.

[c] Determined using GC.

[d] Determined using ICP analysis.

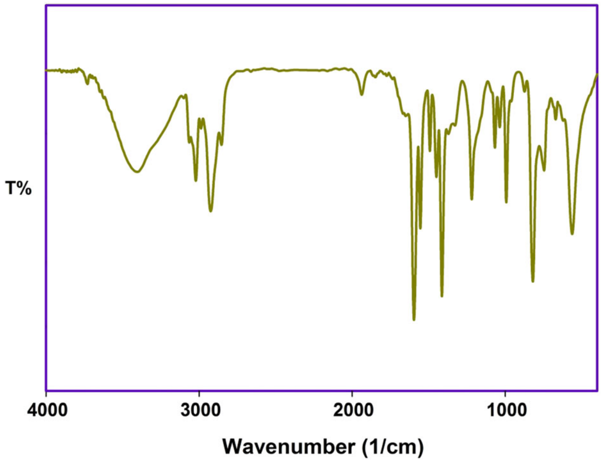


Figure 4. FT-IR spectrum of recovered catalyst.

Conclusions

The NHC complex Pd^{II}(NHC)SO$_3^-$, supported on nano–micro size poly(4-vinylpyridinium chloride), was used as a heterogeneous, recyclable and active catalyst for the Suzuki–Miyaura reaction. Simple preparation of catalyst, good catalytic activity and high reusability are noteworthy advantages of this catalytic system in the C–C coupling reactions.

Acknowledgements

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