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Catalytic and kinetic study of the liquid-phase hydrogenation of various organic substrates over a polymer-anchored Pd(II) catalyst

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Abstract A polymer-anchored Pd(II) complex has been prepared and characterized using scanning electron microscopy, elemental analysis, atomic absorption spectroscopy and FTIR. The catalyst shows excellent catalytic activity in the liquid-phase hydrogenation of substituted nitrobenzenes at normal pressure of hydrogen gas at 25 °C in DMF medium. We have also studied the liquid-phase hydrogenation of other organic substrates such as alkenes, alkynes, aromatic aldehydes, etc. The influences of various parameters such as amount of catalyst, concentration of substrate, temperature and solvent have been studied. The catalyst can be used five times without much loss in activity.

Introduction

The catalytic hydrogenation of substituted nitrobenzenes (NB) is commonly used to manufacture substituted anilines (AN), which are important intermediates for polyurethanes, dyes, pharmaceuticals, explosives and agricultural products [1–4]. Aromatic chloroamines are important intermediates in the synthesis of dyes, drugs, herbicides and pesticides [5]. At present, these organic amines are generally produced through selective hydrogenation of the corresponding aromatic chloronitro compounds over transition metal catalysts such as noble metals and nickel. In this process, the hydrodechlorination of the aromatic chloroamines often occurs due to the electron-donating effect of the amino groups in the aromatic ring [6]. Various metals and their complexes have been used for the homogeneous catalysis of these reactions, but the inherent disadvantages associated with homogeneous catalysis can be minimized by using supported metal complexes. Polymer-anchored heterogeneous catalysts often offer advantages for carrying out organic transformations [7–10]. Ease of work-up, higher yields, product selectivity and re-usability of the catalysts make them more attractive than their homogeneous counterparts. Among supported noble metal catalysts, palladium is one of the most frequently used for hydrogenation reactions at laboratory as well as industrial scale. However, heterogeneous catalysts normally suffer from limited mass transfer, low specificity and selectivity and leaching of the catalytic species from the surface of the support [11]. Cross-linked poly(styrene–divinylbenzene) resin is one of the most widely used macromolecular supports for functionalization and attachment to various metals. Polymer supported catalysts for hydrogenation of organic substrates, notably involve complexes of Rh(I) [12] and Ru(II) [13]. These have been studied as mimics of their homogeneous counterparts which include, among others, Wilkinson's hydrogenation catalyst. Later examples of supported Pd(II) catalysts include polystyrene functionalized with phosphine [14–16], 2,2′-bipyridyl [17] and anthranilic acid [18]. The catalytic activity of these systems is greatly influenced by factors such as the nature of ligand bound to the polymeric backbone, type of olefinic substrate, percentage cross-linking and polarity of solvent, etc.

In the present work, we have developed a simple synthetic route for anchoring 1,10-Phenanthroline-5-amine on a moderately crosslinked chloromethylated poly(styrene–divinylbenzene) matrix followed by complexation with Pd(II). This material was evaluated for its performance as a heterogeneous catalyst in hydrogenation of substituted nitrobenzenes and other substrates. The effect of various

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reaction parameters on hydrogenation as well as the kinetics of the reaction have been studied at room temperature under atmospheric pressure of H_2 gas.

Experimental

Analytical grade reagents and freshly distilled solvents, along with pure and dry hydrogen gas, were used throughout the experiments. Liquid substrates were distilled and dried with appropriate molecular sieve, and solid substrates were recrystallized before use. Distillation and purification of the solvents and substrate were done by standard procedures [19]. Chloromethylated polystyrene and 1,10-phenanthroline-5-amine were supplied by Aldrich. Palladium acetate and other organic reagents were purchased from Merck and used without further purification.

The FTIR spectra of the samples were recorded from 400 to 4,000 cm^{-1} on a Perkin-Elmer FTIR 783 spectrophotometer using KBr pellets. Surface morphology of the samples was measured using a scanning electron microscope (SEM) (ZEISS EVO40, England) equipped with EDX facility. Palladium content in the catalyst was determined using a Varian AA240 atomic absorption spectrophotometer (AAS). The reaction products were quantified (GC data) using a Varian 3400 gas chromatograph equipped with a 30 m CP-SIL8CB capillary column and a flame ionization detector and identified by Trace DSQ II GC–MS equipped with a 60 m TR-50MS capillary column.

Synthesis of the catalyst

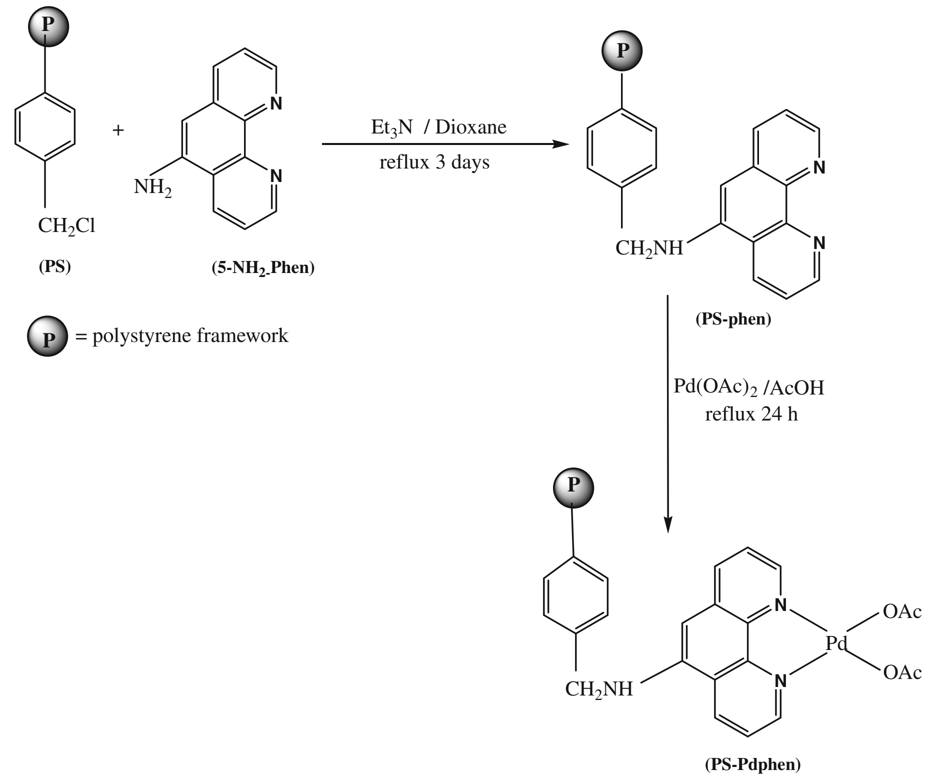
The preparation for the polymer-anchored Pd(II) catalyst is shown in Scheme 1.

Synthesis of polymer-bound 1,10-phenanthroline (PS-phen)

Chloromethylated polystyrene (1.2 g) was swelled for 36 h in dioxane and then added to a solution of 5-NH_2–phen (0.983 g) in dioxane (100 mL). After refluxing and stirring for 26 h, Et_3N (0.71 g) was added. With further refluxing for an additional 32 h, the solution became a yellow–brown color. The mixture was cooled to room temperature and then filtered. The polymer was washed with ethanol until the filtrate was colorless. The brown–yellow polymer was washed with hot dioxane three times and then immersed in dioxane overnight. The mixture was boiled for 5 min and then filtered. After drying under vacuum, the polymer so obtained was yellow–brown.

(PS) found (%); C (70.8), H (5.9), Cl (23.2)
(5-NH_2–phen) found (%); C (70.2), H (5.3), N (24.6)
(PS-phen) found (%); C (85.1), H (5.4), N (9.4)

Scheme 1 Synthesis of the polymer-anchored Pd(II) catalyst



Synthesis of the palladium complex (PS-Pdphen)

The typical method for synthesis of the Pd-complex (PS-Pdphen) is as follows: a mixture of PS-phen (3 g) and $Pd(OAc)_2$ (0.50 g) in acetic acid (40 mL) was refluxed for 24 h with stirring, whereupon the color of solution changed from yellow to deep brown. The deep brown catalyst was filtered off, washed thoroughly with methanol until the washings were colorless and dried in room temperature under vacuum.

(PS-Pdphen) found (%); C (49.1), H (3.7), N (9.05)

Hydrogenation procedure

In a typical procedure, a solution of the desired substrate (5 mmol) in DMF (10 mL) containing the catalyst (5 mg, 9.02×10^{-4} mmol) was subjected to hydrogenation under hydrogen (1.0 atm) in a magnetically stirred glass reactor. The detailed experimental setup and hydrogenation procedure have been described earlier [20]. The reaction mixture was sampled at regular time intervals and analyzed with a Varian 3400 gas chromatograph equipped with a 30 m CP-SIL8CB capillary column and Flame Ionization Detector.

Results and discussion

Characterization of the polymer-anchored Pd(II) complex

Due to the insolubility of the polymer-anchored Pd(II) complex in all common organic solvents, its characterization was limited to its physicochemical properties, chemical analysis, SEM, EDAX, IR and UV–Vis spectra. The metal contents of the catalyst determined by atomic absorption spectroscopy suggested 1.92% (w/w) Pd in the immobilized metal complex.

The morphology of the PS-Pdphen sample was studied using SEM. The micrographs of the PS-phen ligand and PS-Pdphen catalyst are presented in Fig. 1a and b, respectively. The morphological change is quite evident from these images and suggests the loading of palladium on the surface of the polymer matrix. Energy dispersive spectroscopy analysis of X-rays (EDAX) data for the free ligand and the complex polymers are given in Fig. 2a and b, respectively. The EDX data are also consistent with the attachment of palladium on the surface of the polymer matrix.

The mode of attachment of the palladium onto the support was confirmed by comparison of the FTIR spectra of the metal catalyst in various steps of its synthesis. The IR spectra showed a stretching vibration for N–H at 3,463 cm^{-1} for 5-NH_2 phen, and near 3,371 cm^{-1} for the polymer-bound ligand and its complex. The stretching vibrations of the C=N and C=C double bonds were observed at: 1,613 and 1,596 cm^{-1} for PS-phen, and 1,622 and 1,601 cm^{-1} for PS-Pdphen. N-coordination of the phenanthroline moiety to the palladium is suggested from the bond at 567 cm^{-1} [21] assigned to the Pd–N stretching vibration. Two new peaks at 1,629 cm^{-1} (νC=O, free) and 1,313 cm^{-1} (νC–O coordinated) [22] were observed in the spectrum of the complex. From the microanalytical and spectroscopic data, a probable structure of the polymer-anchored palladium(II) complex may be proposed according to Scheme 1.

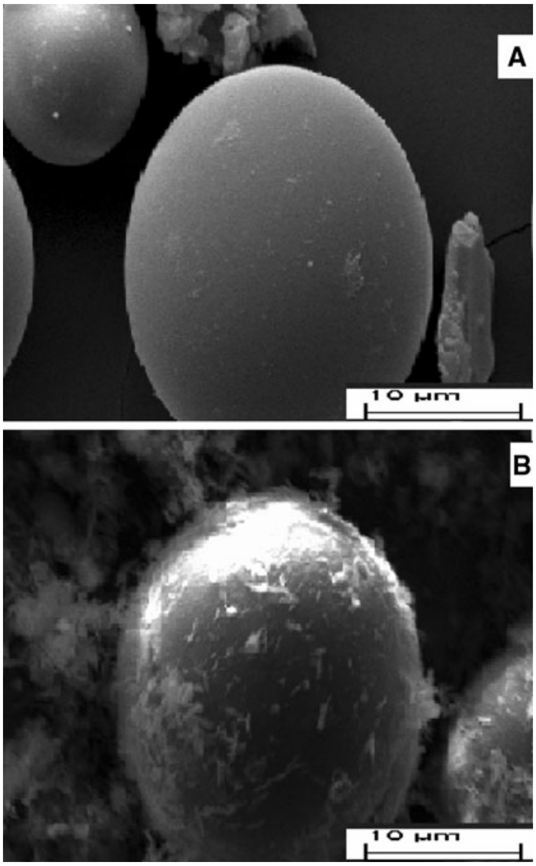


Fig. 1 FE SEM image of PS-phen (**a**) and PS-Pdphen (**b**)

Catalytic reduction experiments

The performance of the supported palladium catalyst was evaluated in the hydrogenation of various substrates at constant temperature (25 °C) and pressure of hydrogen (1 atm). PS-PdPhen catalyzed the hydrogenation of various alkenes, alkynes, aldehydes and substituted nitrobenzenes with high yield. For a proper understanding of the efficacy of the present polymer supported catalyst compared to its homogeneous counterpart, direct comparison of catalytic

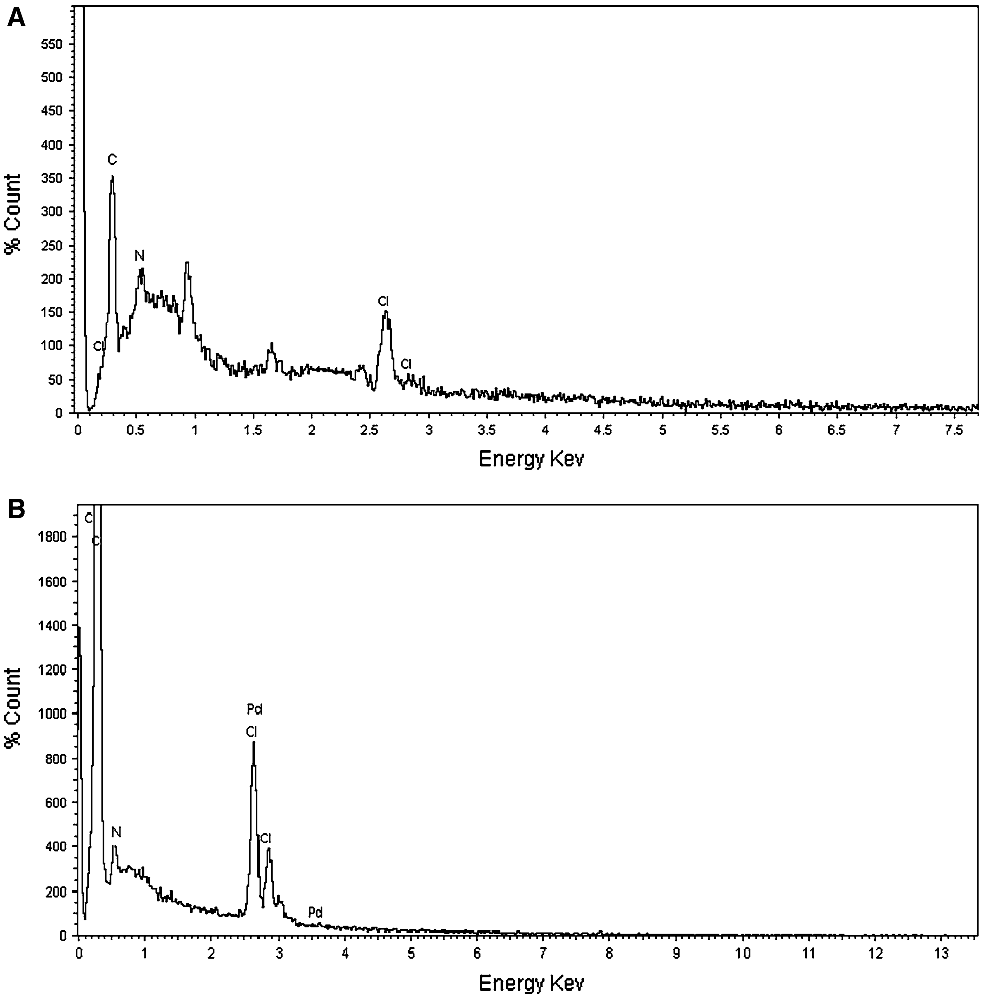


Fig. 2 EDX of PS-phen (**a**) and PS-Pdphen (**b**)

activities under optimized reaction conditions was essential. In the absence of relevant data on hydrogenation of nitrobenzene using homogeneous Pdphen catalysts, an attempt was made to generate in situ a simple 1,10-phenanthroline complex of Pd(II) by mixing Phen with $Pd(OAc)_2$ in 1:1 molar ratio in acetic acid. After a reaction period of 6 h at 25 °C, an aliquot of the solution containing an equivalent amount of Pd (5 mg) as in PS-PdPhen was taken up for subsequent hydrogenation of *p*-hydroxynitrobenzene. The overall yield for this species was only slightly lower than that obtained with the supported catalyst (Fig. 3). The effects of temperature, substrate concentration, catalyst amount, reaction time, solvent and electronic effects on the catalytic hydrogenation of various substituted nitrobenzenes and other substrates with the PS-PdPhen catalyst were investigated (Tables 1, 2). Additionally, comparative evaluation was made with a commercially available hydrogenation catalyst, namely Pd on activated carbon. These results are shown in Table 3. The polymer-anchored palladium complex appears to be superior to Pd/C in the rate of reduction in substrates and yields of products (Table 3). We also compared the activity of this catalyst in the hydrogenation reaction with other reported catalysts (Table 4).

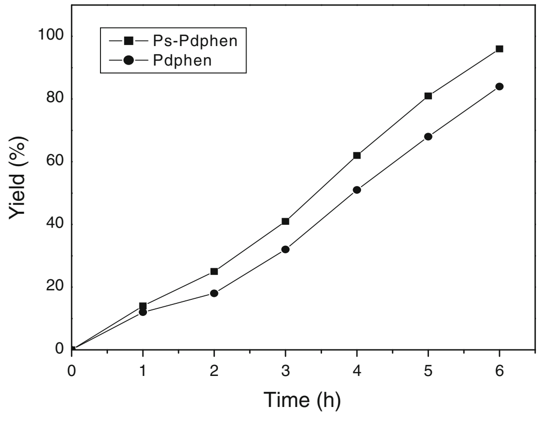


Fig. 3 Hydrogenation of *p*-hydroxynitrobenzene using PS-Pdphen and PS-phen catalyst. Substrate = 5 mmol, catalyst = 9.02×10^{-4} mmol, $P_{H_2} = 1$ atm, $T = 25$ °C, DMF = 10 mL and time = 6 h

The hydrogenation reactions of nitro compounds, alkenes, alkynes, aromatic aldehydes and Schiff bases were

Table 1 Hydrogenation of various substrates and yields of products at 1 atm of H_2 and 25 °C using polymer-anchored palladium catalyst

Expt. no	Substrates	Reaction time (h)	% of yield	TOF (min^{-1})	Products
1.	Nitrobenzene	6	98	15.08	Aniline
2.	*o*-Chloronitrobenzene	8	86.5	10.1	*o*-Chloroaniline
3.	*o*-Nitrotoluene	8	89	10.28	*o*-Toluidine
4.	*m*-Chloronitrobenzene	10	88	8.13	*m*-Chloroaniline
5.	*p*-nitroanisole	6	96	14.78	*p*-Aminoanisole
6.	*p*-Bromonitrobenzene	6	92	14.16	*p*-Bromoaniline
7.	*p*-Nitrotoluene	6	94	14.47	*p*-Toluidine
8.	*m*-Dinitrobenzene	7	96	12.67	*m*-Phenylenediamine
9.	*p*-Hydroxynitrobenzene	6	96	14.32	*p*-Hydroxyanilene
10.	*p*-Chloronitrobenzene	10	93	8.58	*p*-Chloroaniline
11.	*p*-Nitroacetophenone	12	87	6.7	*p*-Aminoacetophenone
12.	*p*-Nitrobenzoicacid	12	85	6.55	*p*-Aminobenzoicacid

Reaction condition: substrate = 5 mmol; catalyst = 5 mg (9.02×10^{-4} mmol); solvent (DMF) = 10 mL

Yield refers to GC & GC–MS analysis

Turn over frequency (TOF) = moles of substrate converted per mole of Pd per min

Table 2 Hydrogenation of various substrates and yields of products at 1 atm of H_2 and 25 °C using polymer-anchored palladium catalyst

Expt. no	Substrates	Reaction time (h)	% of yield	TOF min^{-1}	Products
1.	Styrene	6	92	14.17	Ethylbenzene
2	Pent-1-ene	10	87	8.03	Pentane
3.	Hex-1-ene	12	85	6.55	Hexane
4.	Phenyl acetylene	10	90	8.35	Ethylbenzene
5.	Cyclohexene	6	83	12.78	Cyclohexane
6.	Maleic acid	8	78	9.00	Succinic acid
7.	Benzaldehyde	10	90	8.31	Benzylalcohol

Reaction condition: substrate = 5 mmol; catalyst = 5 mg (9.02×10^{-4} mmol); solvent (DMF) = 10 mL

Yield refers to GC & GC–MS analysis

Turn over frequency (TOF) = moles of substrate converted per mole of Pd per min

employed to investigate the influence of temperature on the rate of hydrogenation using PS-Pdphen catalyst. The hydrogenations were conducted by varying the temperature from 25 to 50 °C at a fixed substrate concentration (5 mmol) and catalyst amount (5 mg) at 1 atm of H_2 in DMF. The hydrogenations of the above substrates were not influenced by temperature, giving near-constant yields. The optimum temperature was therefore taken as 25 °C. We also studied the effect of solvent on the hydrogenation of nitrobenzene at a fixed substrate concentration (5 mmol), catalyst amount (5 mg), reaction temperature (25 °C) and 1 atm of H_2. The nature of the solvent proved to be an important variable. DMF was found to be the best solvent; the rate of hydrogenation was slow in DMSO and very slow in co-ordinating solvents like trimethylamine. No catalytic hydrogenation was observed in non-coordinating solvents like CH_2Cl_2 and petroleum ether.

The yield of the product for the reduction of nitrobenzene was found to increase with increase in catalyst amount within the range 2.5–7.5 mg. Above 5 mg of catalyst, the yield remained almost constant. The substrate concentration (5 mmol), temperature (25 °C) and H_2 pressure of 1 atm in DMF solvent were kept constant during these experiments. The results are shown in Fig. 4.

Table 3 Comparison of catalytic activities of PS-Pdphen and Pd/C

Entry	Substrates	PS-Pdphen		Pd/C		Products
		Yield (%)	TOF (min^{-1})	Yield (%)	TOF (min^{-1})	
1.	Nitrobenzene	98	15.08	96	12.3	Aniline
2.	*m*-dinitrobenzene	96	12.67	91	7.18	*m*-Phelenediamine
3.	*o*-nitrotoluene	89	10.28	87	8.3	*o*-Toluidine
4.	Phenylacetylene	90	8.35	92	8.38	Ethylbenzene
5.	Styrene	92	14.17	94	10.48	Ethylbenzene
6.	Benzaldehyde	90	8.31	87	5.63	Benzylalcohol

Reaction condition: substrate = 5 mmol; catalyst = 5 mg; solvent (DMF) = 10 mL

Yield refers to GC & GC–MS analysis

Turn over frequency (TOF) = moles of substrate converted per mole of Pd per min

Table 4 Comparison of different catalysts in the hydrogenation reaction

Substrates	Catalyst	TOF (min^{-1})	Products	Reference
Nitrobenzene	PS-Pdphen	15.08	Aniline	This study
	Fe^{II}/$EDTANa_2$	8.82		[23]
	Poly(S-DVB) supported amino acid Pd(II) complex	0.23		[24]
o-Nitrotoluene	PS-Pdphen	10.28	*o*-Toluidine	This study
	Fe^{II}/$EDTANa_2$	7.62		[23]
p-Chloronitrobenzene	PS-Pdphen	8.58	Chloroaniline	This study
	Fe^{II}/$EDTANa_2$	6.67		[23]
	Al-MCM-41supported Pd catalyst	0.88		[25]
Styrene	PS-Pdphen	14.17	Ethylbenzene	This study
	NH_2-MCM-41-[RuHCl(CO)$(PPh_3)_2$]	1.33		[26]
	NH_2-SBA-15-[RuHCl(CO)$(PPh_3)_2$]	2.05		[26]

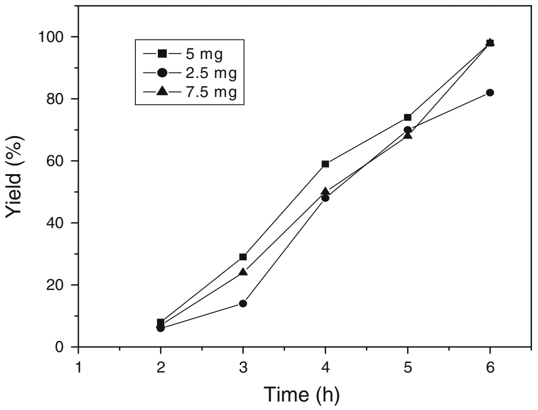


Fig. 4 Effect of catalyst amount on the hydrogenation of nitrobenzene using PS-Pdphen catalyst. Substrate = 5 mmol, $P_{H_2} = 1$ atm, $T = 25$ °C, DMF = 10 mL and time = 6 h

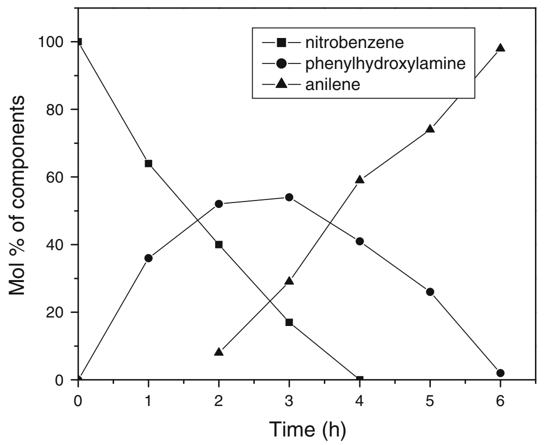


Fig. 6 Hydrogenation of nitrobenzene using PS-Pdphen catalyst. Substrate = 5 mmol, catalyst = 9.02×10^{-4} mmol, $P_{H_2} = 1$ atm, $T = 25$ °C, DMF = 10 mL and time = 6 h

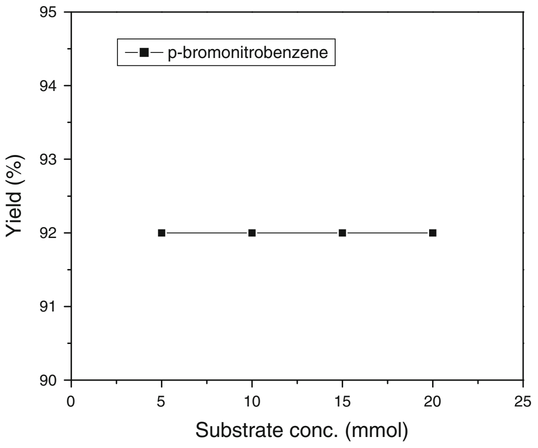


Fig. 5 Effect of substrate concentration on the hydrogenation of *p*-bromonitrobenzene using PS-Pdphen catalyst. Catalyst = 9.02×10^{-4} mmol, $P_{H_2} = 1$ atm, $T = 25$ °C, DMF = 10 mL and time = 6 h

The yield of the product for the reduction in *p*-bromonitrobenzene was independent of substrate concentration in the range 5–20 mmol at a fixed catalyst amount (5 mg) and other conditions as before (Fig. 5).

Electronic effects on the hydrogenation of substituted nitrobenzenes

The yields of products for the hydrogenation of substituted aromatic nitro compounds were lower than those for hydrogenation of nitrobenzene (Table 1). The reduction of nitrobenzene proceeds via the intermediate formation of phenyl hydroxylamine, but coupling products such as azo- or azoxy-compounds were not formed at any stage during their reduction (Fig. 6). High yield was obtained for *p*-nitroanisole, while relatively low yields were obtained for *p*-, *o*-nitrotoluenes and *p*-bromonitrobenzene. Figure 7 shows, in the case of dinitrobenzene reduction, the presence

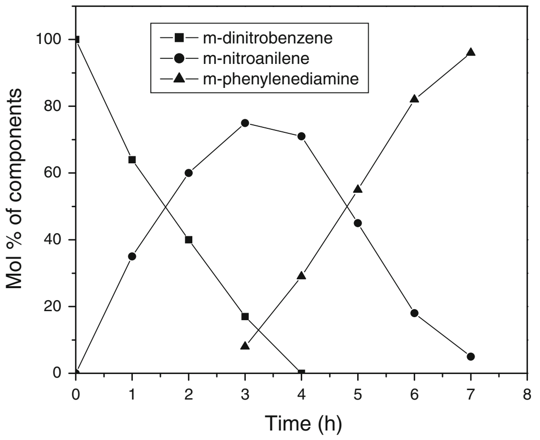


Fig. 7 Hydrogenation of m-dinitrobenzene using PS-Pdphen catalyst. Substrate = 5 mmol, catalyst = 9.02×10^{-4} mmol, $P_{H_2} = 1$ atm, $T = 25$ °C, DMF = 10 mL and time = 7 h

of phenylenediamine was detected only after 73–77% formation of mono-nitroaniline. As seen from Table 1, hydrogenation of *p*-nitroacetophenone and *p*-nitrobenzoic-acid gave the corresponding amino derivatives without affecting the other reducible groups. Even the hydrogenation of halonitrobenzenes showed high selectivity for haloanilines without any dehalogenation of halonitrobenzenes. *o*-Chloronitrobenzene and *o*-nitrotoluene were reduced at slower rates than those of non-substituted and *p*-substituted compounds (Table 1).

Reduction in alkenes, alkynes and aromatic aldehydes

The catalyst proved to be highly effective for the hydrogenation of linear and cyclic alkenes, alkynes and aromatic aldehydes under atmospheric pressure of H_2. The yields of various products are presented in Table 2. From the rates of reduction in various substrates, it appears that double bonds which are part of a delocalized system are reduced more quickly than localized double bonds, since styrene was reduced more quickly than hex-1-ene. Phenylacetylene was successively reduced first to styrene and than to ethylbenzene. In general, increase in chain length of alk-1-enes caused decrease in reduction rate, probably due to steric factors which would prevent approach of the substrate to the metal centre in the polymer matrix.

Catalyst recycling and heterogeneity tests

One of the main advantages of a heterogeneous catalyst is enhanced lifespan. To investigate the reusability of this polymer-anchored complex, the catalyst was separated by filtration after the first run, washed, dried under vacuum and then subjected to a second run under the same conditions. This was repeated with further addition of substrate, and the nature and yield of the final products were comparable to the original results. As shown in Table 5, the catalyst can be reused up to five times, without any appreciable loss of its catalytic activity.

To determine whether the catalyst was actually functioning in a heterogeneous manner, a hot-filtration test was performed in the hydrogenation of nitrobenzene. The solid catalyst was filtered out after the reaction had proceeded for 4 h, at which point the determined conversion was 59%, and the liquid phase of the reaction mixture was collected by filtration at the reaction temperature. Atomic absorption spectrometric analysis of the liquid phase confirmed the absence of Pd. The obtained filtrate was then stirred under the reaction conditions. After 2 h, the conversion was still 59%. These results indicate that the catalytic reaction was caused by the solid catalyst. Pd was also not detected in the liquid phase of the reaction mixture after the completion of the reaction. It was noteworthy that the DMF remains completely colorless on addition of Pd catalyst. These

Table 5 Recycling of the catalyst for hydrogenation of various organic substrates

Expt. no	Substrates	Products	1st cycle		3rd cycle		5th cycle	
			Yield (%)	TOF (min^{-1})	Yield (%)	TOF (min^{-1})	Yield (%)	TOF (min^{-1})
1.	Nitrobenzene	Aniline	98	15.08	96	14.78	95	14.63
2.	*p*-Bromonitrobenzene	*p*-Bromoaniline	92	14.16	91	14.01	90	13.86
3[a].	*p*-Nitrobenzoicacid	*p*-Aminobenzoicacid	85	6.55	82	6.31	81	6.23
4.	Styrene	Ethylbenzene	92	14.17	90	13.85	89	13.70
5[b].	Benzaldehyde	Benzylalcohol	90	8.31	90	8.31	89	8.22

Reaction condition: substrate = 5 mmol; catalyst = 5 mg (9.02×10^{-4} mmol); solvent (DMF) = 10 mL; $P_{H_2} = 1$ atm, time = 6 h, $T = 25$ °C

[a] Time = 12 h

[b] Time = 10 h

Yield refers to GC & GC–MS analysis

results suggest that the Pd was not being leached out from the catalyst during hydrogenation reactions.

Conclusion

By immobilizing palladium on the surface of a polymer-anchored phenanthroline ligand, an efficient and highly recyclable heterogeneous catalyst was prepared. This catalyst shows high activity in liquid-phase hydrogenation reactions of substituted nitrobenzenes at normal pressure. The catalyst exhibits excellent performance in the reduction in alkenes, alkynes, aromatic aldehydes and acids. It can be easily recovered by filtration and good yields are obtained even when the catalyst is reused at least five times.

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