Study on the hydrogenation of *p*-chloronitrobenzene over carbon nanotubes supported platinum catalysts modified by Mn, Fe, Co, Ni and Cu

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Abstract

Hydrogenation properties of *p*-chloronitrobenzene (CNB) have been studied over Pt/CNTs and PtM/CNTs catalysts (M = Mn, Fe, Co, Ni and Cu) in ethanol at 303 K and normal pressure. The results exhibit that the hydrogenation of *p*-CNB can be carried out over PtM/CNTs catalysts. Both catalytic activities and yields of *p*-chloroaniline (CAN) are improved. PtFe/CNTs catalyst exhibits the best catalytic activity (TOF is $0.47\,s^{-1}$), and PtMn/CNTs catalyst exhibits the highest yield of *p*-CAN (98.5 mol%). Different reductants have also influence on both catalytic activity and yield of *p*-CAN. Pt/CNTs(II) catalyst exhibits good catalytic activity and stability on the hydrogenation of *p*-CNB. PtM/CNTs(II) catalysts exhibit low catalytic activity and high yield of *p*-CAN.
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1. Introduction

Aromatic haloamines are important intermediates in the chemistry of dyes, drugs, herbicides and pesticides. The traditional synthesis routes are usually harmful for environment. Selective hydrogenation route has received much attention, owing to its lower impact on the environment since no acid effluents are produced. However, the hydrogentaion process is difficult because of extensive dehalogenation during the catalytic reduction of the compound [1]. Special catalyst has been proposed as a method of reducing hydrogenolysis of the halogen group [1–6]. Among these catalysts, platinum-based catalysts have received much attention for minimizing dehalogenation combined with a fast rate of nitro-group reduction. The hydrogenation selectivity could be improved by modified preparation of the catalyst or by use of special additives (promoters and inhibitors) [7–14].

Catalytic hydrogenation reactions are influenced by many factors, which include the presence of catalytic activators or poisons, temperature, pressure, solvent agitation and other conditions specific to the particular process. One of the more important considerations is the catalyst support. Carbon nanotubes (CNTs) have developed entirely new field in many natural science since their discovery at the beginning of the last decade [15]. The unique physical and chemical properties make them potential and promising applications in various areas like reinforcement materials and microelectronics devices. Recently, there has been great interest to use CNTs as catalyst supports. Such structures can display unusual behaviours compared to classical supports, especially for liquid-phase reactions and some meaningful results have been obtained [16–18]. Palladium [19–21], platinum [22,23] and ruthenium [16,24] catalysts supported carbon nanotubes exhibit better catalytic properties on the hydrogenation of α,β-unsaturated aldehydes. Good hydrogenation properties were also reported by Yao and co-workers [25,26] for nitrobenzene hydrogenation over Pt/CNTs catalysts.

The aim of the present work is to determine the influence of transition metals on the hydrogenation of *p*-chloronitrobenzene (abbreviated as *p*-CNB) over PtM/CNTs and PtM/CNTs(II) catalysts. Substrate concentration and reaction temperature are also studied on the hydrogenation of *p*-CNB over Pt/CNTs(II) catalyst in the liquid phase.

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Table 1
Results of the hydrogenation of *p*-CNB over PtM/CNTs catalysts

Catalyst	Reaction rate[a] $(molH_2\,g^{-1}\,min^{-1}) \times 10^{-2}$	TOF[b] (s^{-1})	TEM[c] (nm)	Conversion (%) (reaction time, min)	Yield of products (mol%)[d]		
					AN	NB	*p*-CAN
CNTs	Nil	–	–	–	–	–	–
Pt/CNTs	24.6	0.20	2.5	99.6 (115)	0.6	7.5	73.5
PtMn/CNTs	43.5	0.46	2.1	99.7 (67)	–	–	98.5
PtFe/CNTs	45.0	0.47	2.0	99.4 (65)	0.3	–	97.6
PtCo/CNTs	45.1	0.46	2.3	99.6 (64)	1.1	0.5	94.7
PtNi/CNTs	41.8	0.44	2.6	99.3 (70)	0.2	–	98.0
PtCu/CNTs	6.8	0.04	2.3	57.4 (240)	1.5	1.3	92.3

Reaction condition: 4×10^{-3} mol *p*-CNB; reaction temperature is 303 K; *P* is 0.1 MPa; the amount of catalyst is 0.15 g.

[a] The reaction rate is obtained by measuring the H_2 consumption within the reaction time.

[b] TOF, specific activity per Pt surface atom (s^{-1}) for the hydrogenation of *p*-chloronitrobenzene over supported Pt catalysts.

[c] Pt particle size from TEM.

[d] Some products with high boiling point could not be determined by GC analysis; AN = aniline; NB = nitrobenzene.

Table 2
Results of the hydrogenation of *p*-CNB over PtM/CNTs(II) catalysts

Catalyst	Reaction rate (mol $H_2\,g^{-1}\,min^{-1}) \times 10^{-2}$	TOF (s^{-1})	TEM (nm)	Conversion (%) (reaction time, min)	Yield of products (mol%)		
					AN	NB	*p*-CAN
Pt/CNTs	37.9	0.36	3.0	99.5 (80)	–	5.2	88.9
PtMn/CNTs	28.5	0.29	4.0	99.5 (105)	2.3.	0.9	95.2
PtFe/CNTs	25.7	0.26	3.8	99.4 (120)	3.1	1.1	94.3
PtCo/CNTs	31.0	0.32	3.9	99.5 (95)	1.1	0.5	95.7
PtNi/CNTs	19.5	0.21	3.6	99.6 (160)	0.7	–	97.8
PtCu/CNTs	Nil	–	3.7	–	–	–	–

Reaction conditions are similar to Table 1.

2. Experimental

Carbon nanotubes (CNTs) are prepared according to method described in Ref. [27]. The surface area of CNTs is $110\,m^2/g$ and the average pore diameter value is 22 nm, the pore volume is 0.311 mL/g. The CNTs are pretreated in thick nitric acid for 4 h before used as catalyst carrier. The monometallic Pt/CNTs catalyst is prepared by conventional impregnation with an aqueous solution of hexachloroplatinic acid. The loading of platinum is 0.3 wt.%. The sample impregnated is filtered and washed with large amount of water, dried at 373 K overnight, and then reduced by H_2 at 673 K for 4 h. The bimetallic PtM/CNTs catalysts (M = Mn, Fe, Co, Ni and Cu) are also prepared by impregnation, i.e. platinum is impregnated after the transition metal is impregnated on CNTs. The preparation process is similar to monometallic Pt/CNTs catalyst, and the content of M and Pt is 0.1 and 0.3 wt.%, respectively. The sample impregnated is reduced by KBH_4, filtered with large amount of water, dried at 373 K overnight, and abbreviated as Pt/CNTs(II) catalyst. The preparation process of PtM/CNTs(II) catalyst is similar to monometallic Pt/CNTs(II) catalyst.

Transmission electron microscopy (TEM) is a conventional method to give detailed information about the shapes, mean size and size distribution of metallic dispersions. The size of the platinum metallic particles is checked by TEM using a JEM-200CX apparatus. The results are listed in Tables 1 and 2. X-ray photoelectron spectroscopy (XPS) measurements are performed on a VG ESCALAB 2201-XL spectrometer. Non-monochro Mg Ka

radiation are used as a primary excitation. The binding energies are calibrated with the C 1s level of adventitious carbon (284.6 eV) as the internal standard reference. The results are listed in Table 3.

Hydrogenation of *p*-CNB is carried out at 303 K and atmospheric pressure of hydrogen in a thermostated glass reactor closed with silicon rubber cap and connected with hydrogen gas burette. The atmosphere is replaced with hydrogen several times after 0.15 g platinum catalyst is added to the reactor, and then 10 mL of ethanol solution is added. The catalyst is activated under hydrogen with magnetic stirring at 303 K for 1 h before 10 mL ethanol solution of *p*-chloronitrobenzene (4×10^{-3} mol of substrate) is injected into the reactor. The hydrogenation consumption is monitored with a graduated gas burette. Chemical analysis of the products is performed by gas chromatography Perkin-XL, equipped with a FID detector and SE-30 capillary column. Reactants and products are identified by comparison with authentic samples. Biphenyl is used as internal standard.

Table 3
Binding energies of the Pt $4f_{72}$ XPS level for Pt/CNTs, Pt/CNTs(II), Pt–Co/CNTs and Pt–Co/CNTs(II) catalysts

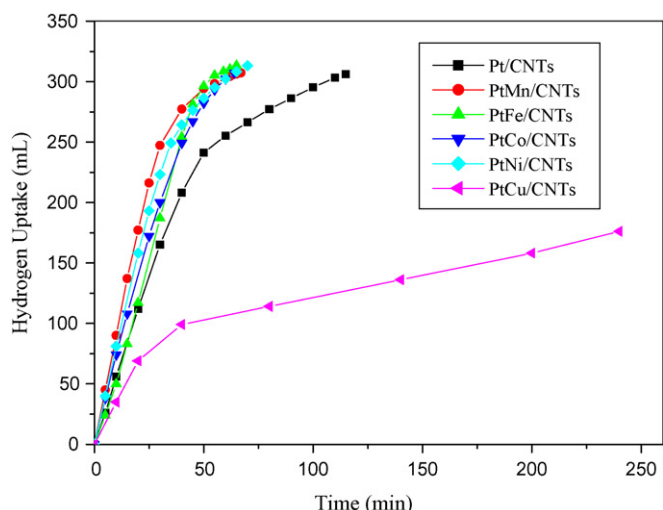| Sample	Pt $4f_{7/2}$ (eV)	
Pt/CNTs	70.8 (15.5)	75.0 (84.5)
Pt/CNTs(II)	71.0 (47.7)	75.2 (52.3)
PtCo/CNTs	70.9 (36.9)	75.1 (63.1)
PtCo/CNTs(II)	71.1 (10.9)	74.9 (89.1)

Fig. 1. Cumulative hydrogen uptake profiles of catalytic hydrogenation of *p*-chloronitrobenzene over PtM/CNTs catalysts.

Several experiments using different amounts of catalyst and different speed of agitation are carried out in order to determine the diffusion control region and eliminate mass-transfer limitations during our studies.

3. Results and discussion

Hydrogenation of CNB is a complicated process as described elsewhere [28,29], and it often yields many by-products. The effect of small amount of transition metals (Mn, Fe, Co, Ni and Cu) on the hydrogenation of *p*-CNB over Pt/CNTs catalyst has been investigated, and the results are listed in Table 1. No products are observed when carbon nanotubes are tested in *p*-CNB hydrogenation. From Table 1, we can see that Pt/CNTs can catalyze the hydrogenation of *p*-CNB at 303 K and normal pressure. Additions of transition metals in Pt/CNTs catalysts have obvious influence on the hydrogenation properties of *p*-CNB. Both catalytic activity and yield of *p*-CAN are improved for PtMn/CNTs, PtFe/CNTs, PtCo/CNTs and PtNi/CNTs catalysts. PtFe/CNTs catalyst exhibits the best catalytic activity (the value of TOF is $0.47 \, s^{-1}$), and PtMn/CNTs catalyst exhibits the highest yield of *p*-CAN (98.5 mol%). PtCu/CNTs catalyst exhibits the worst catalytic activity on the hydrogenation of *p*-CNB, but yield of *p*-CAN is improved.

Fig. 1 gives the kinetic curves of hydrogenation of *p*-CNB catalyzed by PtM/CNTs catalysts. From Fig. 1, we find that platinum catalysts modified by transition metals exhibit similar catalytic behaviour on the hydrogenation of *p*-CNB. The catalytic activities are all higher except for PtCu/CNTs catalyst than Pt/CNTs catalyst is used alone. The hydrogen consumption is lined with reaction time at the prophase of reaction.

It is known that the hydrogenation rates are influenced by many factors in catalytic hydrogenation reactions. Platinum catalysts reduced by different reductants exhibit different catalytic properties for the hydrogenation of CNB. In our previous research [14], we find that platinum catalysts reduced by KBH_4 often exhibit good catalytic activity and high yield of CAN. So we investigate the hydrogenation properties of PtM/CNTs catalysts reduced by KBH_4 on the hydrogenation of *p*-CNB, and the results are listed in Table 2. From Table 2, we also find that Pt/CNTs catalyst reduced by KBH_4 exhibits good catalytic activity and high yield of *p*-CAN. Higher yields of *p*-CAN and lower catalytic activities are obtained for PtM/CNTs(II) catalysts than Pt/CNTs(II) catalyst is used alone. PtNi/CNTs(II) catalyst exhibits the highest yield of *p*-CAN (97.8 mol%). Low catalytic activity of PtM/CNTs(II) catalysts is probably related to the chemical state of Pt of the catalysts. Table 3 lists the binding energies of the Pt $4f_{72}$ level for the Pt/CNTs, Pt/CNTs(II), PtCo/CNTs and PtCo/CNTs(II) catalysts. From Table 3, it can be seen that two doublets are obtained for catalysts: one at a low binding energy, which can be assigned to metallic platinum, the BE values for Pt $4f_{7/2}$ levels of bulk metallic Pt, reported in the literature [30], ranged between 71.0 and 71.3 eV, and a second one at a higher binding energy, corresponding to Pt(IV) oxidized species [31]. The XPS results indicate that no significant difference is present in the Pt 4f spectra between Pt/CNTs and PtCo/CNTs subjected to H_2 or KBH_4 treatment, but the atomic percents vary a lot, as shown in Table 3. The percentage of Pt(0) and Pt(IV) are included in brackets. Atomic percents of Pt(0) increase for Pt/CNTs(II) catalyst. Low percentage of Pt(0) of PtCo/CNTs(II) catalyst is probably responsible for low catalytic activity on the hydrogenation of *p*-CNB.

Base on above experiment results, we study the influence of substrate concentration and reaction temperature on the hydrogenation of *p*-CNB over Pt/CNTs(II) catalyst in particular. The results are listed in Tables 4 and 5. In our experiment, we also find that the yield of *p*-CAN increases with increasing the concentration of substrate on the hydrogenation of *p*-CNB. High concentration of substrate probably prevents products from con-

Table 4
Effect of substrate concentration on the hydrogenation of *p*-CNB over Pt/CNTs(II) catalyst

Substrate (mol/L)	Reaction rate (mol $H_2 \, g^{-1} \, min^{-1}) \times 10^{-2}$	Conversion (%) (reaction time, min)	Yield of products (mol%)		
			AN	NB	*p*-CAN
0.144	33.3	99.7 (58)	1.3	6.3	87.8
0.183	35.1	99.5 (70)	1.8	3.5	89.0
0.227	37.9	99.7 (80)	–	5.2	88.9
0.273	39.3	99.8 (95)	2.2	3.6	89.5
0.333	41.1	99.6 (107)	1.7	4.1	90.3

Reaction conditions are similar to Table 1.

Table 5
Effect of reaction temperature on the hydrogenation of *p*-CNB over Pt/CNTs(II) catalyst

Temperature (°C)	Reaction rate (mol $H_2\,g^{-1}\,min^{-1}) \times 10^{-2}$	TOF (s^{-1})	Conversion (%) (reaction time, min)	Yield of products (mol%)		
				AN	NB	*p*-CAN
20	30.5	0.28	99.6 (105)	0.9	3.5	91.3
30	37.9	0.36	99.5 (80)	–	5.2	88.9
35	37.4	0.37	99.7 (80)	1.6	3.3	92.6
40	45.2	0.42	99.5 (68)	1.7	2.9	90.7

Reaction conditions are similar to Table 1.

tacting with active sites, which partly inhibits hydrogenolysis of C–Cl bond [32]. From Table 4, we can see that the reaction rate strongly depends on substrate concentration. Under constant hydrogen pressure, reaction temperature and catalyst concentration, the reaction rate equation may be expressed as follows:

$$R = kC^n \qquad (1)$$

where *R*, *k*, *C* and *n* are the reaction rate, rate constant, concentration of *p*-CNB and reaction order, respectively. A plot of log *R* versus log *C* results in a straight line, and the reaction order "*n*" is 0.26.

Hydrogenation rate often increases with increasing reaction temperature. In our experiment, we also obtain similar results, but the yield of *p*-CAN decreases (see Table 5). The main reason is that hydrogenolysis rate of the C–Cl bond increases with increasing reaction temperature. The partial hydrogen pressure in the reaction flask at 35 °C will decrease for the evaporation of solvent and a lower hydrogenation rate is observed. At a higher than 40 °C temperature, high catalytic activity resumed but yield of *p*-CAN decreases sharply. The activation energy was determined for Pt/CNTs(II) catalyst. The value found, i.e. $12 \pm 2\,kJ\,mol^{-1}$, is lower than those reported by Coq et al. [32] for the hydrogenation of *p*-CNB over Pt/Al$_2$O$_3$ ($25 \pm 2\,kJ\,mol^{-1}$), or by Moreau et al. [33], who found a value of $27\,kJ\,mol^{-1}$ for CNB hydrogenation over sulfide catalysts.

The stability of Pt/CNTs(II) catalyst was also investigated, and the results are summarized in Table 6. In the recycling experiment, five recycles were carried out for the hydrogenation of *p*-CNB. It is found that the catalytic activity decrease very slightly, but the yield of *p*-CAN increases. Ninety-three mole percentage of yield of *p*-CAN is obtained when catalyst is

used for four recycles. Lower catalytic activity is influenced by many factors on the hydrogenation of *p*-CNB. For example, a part of Pt could be lost when the catalyst is reused (the level of Pt measured by ICP still is 0.26 wt.%), and results in decreasing in the number of active sites for hydrogen of the Pt surface. Some intermediates could be also adsorbed on the surface of catalyst and partly cover active center, and which decrease catalytic activity. After Pt/CNTs(II) catalyst is used for five recycles, the catalyst is reduced by KBH$_4$ again, filtered and washed with large amount of water, and then dried at 373 K. We find that catalytic activity is partly resumed. The result exhibits that loss part of B is also a reason of lower catalytic activity.

As indicated above, Pt/CNTs catalyst modified by small amount of transition metals (Mn, Fe, Co, Ni and Cu) exhibits different hydrogenation properties for the hydrogenation of *p*-CNB. Among the PtM/CNTs catalysts, PtFe/CNTs catalyst exhibits the best catalytic activity on the hydrogenation of *p*-CNB. PtMn/CNTs catalyst exhibits the highest yield of *p*-CAN. In PtM/CNTs(II) catalysts, PtNi/CNTs(II) catalyst exhibits the highest yield of *p*-CAN (97.8 mol%). The effect of transition metal may be usually interpreted by electronic effect and/or geometric effect. As reported by literature, good hydrogenation properties are found on large Pt particles. Such behaviour was also reported by Galvagno et al. [34] for nitrobenzene hydrogenation over PtSn/nylon catalysts and Coq [32] for *p*-CNB hydrogenation over Pt/Al$_2$O$_3$ catalysts. It was explained in part by a redispersion of the Pt particles. This interpretation cannot be completely applied in the present work for these Pt particles did not change distinctly in size when adding the second metal (see Tables 1 and 2). We prefer the interpretation proposed by Galvagno et al. [34], suggesting that the tin species on the N–O bond, which becomes more reactive towards the attack by chemisorbed hydrogen. PtFe/CNTs and PtCo/CNTs(II) cata-

Table 6
Stability of Pt/CNTs(II) catalyst

Cycle	Reaction rate (mol $H_2\,g^{-1}\,min^{-1}) \times 10^{-2}$	TOF (s^{-1})	Conversion (%) (reaction time, min)	Yield of products (mol%)		
				AN	NB	*p*-CAN
1	37.9	0.36	99.5 (80)	–	5.2	88.9
2	36.2	0.36	99.6 (80)	3.6	1.2	91.4
3	35.5	0.35	99.7 (82)	1.0	4.0	92.2
4	32.3	0.32	99.7 (90)	1.2	3.3	92.6
5	29.1	0.29	99.6 (100)	1.6	–	93.0
5[a]	30.6	0.30	99.6 (105)	2.7	0.5	92.2

Reaction conditions are similar to Table 1.
  [a] Catalyst is recycled four times, and then reduced by KBH$_4$ again.

lysts exhibit better catalytic activity than that of catalysts. Ionic and electron-deficient Fe or Co species would be the active centers for N=O activation in these bimetallic formations. The good effects are also related to the chemical state of Pt of the catalysts. It is well known that copper metal catalysts usually exhibit better catalytic properties on the selective hydrogenation of various organic compounds. However, copper metal catalysts are easily poisoned by sulfide and chloride. In our experiment, PtCu/CNTs catalyst exhibits low catalytic activity, which may be related to the chlorine from the hydrogenolysis of the C–Cl bond and/or from hexachloroplatinic acid.

4. Conclusions

Pt/CNTs catalyst can catalyze the hydrogenation of *p*-CNB at 303 K and normal pressure. Both catalytic activities and yields of *p*-CAN are all improved with the additions of small amount of transition metal to Pt/CNTs catalyst except Cu. The effect of transition metal may be interpreted by electronic effect. Electron-deficient species of the second element promote the turnover frequency of Pt atoms by activating the nitrogen–oxygen bond. PtFe/CNTs catalyst exhibits the best catalytic activity (TOF is $0.47\,s^{-1}$), and PtMn/CNTs catalyst exhibits the highest yield of *p*-CAN (98.5 mol%). Pt/CNTs catalyst reduced by KBH_4 exhibits good catalytic activity and stability on the hydrogenation of *p*-CNB. Higher yields of *p*-CAN and lower catalytic activities are obtained for PtM/CNTs(II) catalysts. PtNi/CNTs(II) catalyst exhibits the highest yield of *p*-CAN (97.8 mol%).

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