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Highly efficient epoxidation of alkenes with hydrogen peroxide catalyzed by tungsten hexacarbonyl supported on multi-wall carbon nanotubes

**Mehdi Nooraeipour · Majid Moghadam ·
Shahram Tangestaninejad · Valiollah Mirkhani ·
Iraj Mohammadpoor-Baltork · S. Masoud Nabavizadeh**

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Abstract The preparation, characterization, and catalytic activity of $W(CO)_6$ supported on multi-wall carbon nanotubes modified with 4-aminopyridine is reported. The catalyst, $[W(CO)_5@Apy\text{-}MWCNT]$, was characterized by physico-chemical and spectroscopic methods and found to be an efficient heterogeneous catalyst for green epoxidation of alkenes with hydrogen peroxide in MeCN solvent. The catalyst showed good stability and reusability properties in the epoxidation reactions.

Introduction

Catalytic epoxidation of alkenes is an important industrial reaction and also is a useful synthetic method for the production of a wide variety of fine chemicals [1, 2]. Different single oxygen donors such as $NaIO_4$, NaOCl, PhIO, ROOH, O_2, and H_2O_2 have been used for alkene epoxidation in the presence of a catalyst. Among these, hydrogen peroxide is an efficient and attractive oxidant, particularly due to its low cost and green nature in which water is produced as the by-product [3].

Transition metal complexes are efficient catalysts for a wide variety of organic reactions such as the epoxidation of alkenes [4]. However, the major problem associated with these homogeneous catalysts is the recovery of catalyst from the reaction medium. In recent years, there have been intense efforts to develop the recovery and reuse of homogeneous catalysts [5]. An efficient and practical way to achieve commercial applications of these expensive catalysts is their immobilization on solid supports. These immobilized catalysts can offer numerous advantages over their homogeneous counterparts, including easy separation of the catalyst from the reaction media; reduced toxicity; simple recycling of expensive catalysts; and minimization of catalyst deactivation through site isolation. The use of polymer-supported catalysts in organic transformations has been receiving a great deal of attention [6, 7], and the design of functionalized polymers carrying catalytically active metal species has attracted considerable interest [8–16].

Supported catalysts based on tungsten species show many applications in heterogeneous catalysis. Thus, they are widely used in several industrially important reactions such as hydrotreatments, hydrocracking of heavy fractions from oil, dehydration of alcohols, metathesis, oxidations, and isomerization of olefins [17]. The supports used with these catalysts are $A1_2O_3$ and TiO_2 [18, 19], SiO_2 [20], MgO [21], activated carbon [22], polymers [8, 23], and MgF_2 [24].

Carbon nanotubes (CNTs) have attracted much attention because of their unique structural, mechanical, thermal, optical, and electronic properties [25]. Since CNTs are insoluble in most solvents and have high surface areas, they can be used as catalyst supports. For example, Pt nanoparticles supported on CNTs have been used for methanol oxidation [26], palladium nanoparticles supported on CNTs for hydrogenation of phenylacetylene [27], and a chiral vanadyl salen complex supported on

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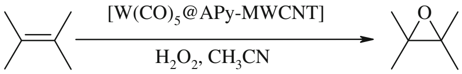
M. Nooraeipour · M. Moghadam (✉) · S. Tangestaninejad ·
V. Mirkhani · I. Mohammadpoor-Baltork
Department of Chemistry, Catalysis Division,
University of Isfahan, Isfahan 81746-73441, Iran
e-mail: moghadamm@sci.ui.ac.ir

S. Masoud Nabavizadeh
Department of Chemistry, Shiraz University, Shiraz 71454, Iran



Scheme 1 Epoxidation of alkenes with H_2O_2 catalyzed by [$W(CO)_5$@APy-MWCNT]

single-wall CNTs has been used for enantioselective cyanosilylation of aldehydes [28].

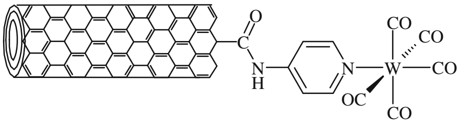
Previously, we reported manganese(III) porphyrin and manganese(III) salophen supported on MWCNTs for epoxidation of alkenes with $NaIO_4$ [29, 30] and molybdenum hexacarbonyl supported on MWCNTs for alkene epoxidation with *tert*-BuOOH [31–33].

In this article, the preparation, characterization, and catalytic activity of $W(CO)_6$ supported on amine-modified multi-wall carbon nanotubes is reported (Scheme 1).

Results and discussion

Preparation and characterization of [$W(CO)_5$@APy-MWCNT]

Scheme 2 shows the proposed structure for [$W(CO)_5$@APy-MWCNT]. First, the APy-MWCNT was prepared by covalent attachment of 4-aminopyridine to MWCNT–COCl via an amide linkage. Then, $W(CO)_6$ was activated in THF under UV irradiation to produce $W(CO)_5$THF. The [$W(CO)_5$@APy-MWCNT] catalyst was then synthesized by the reaction of APy-MWCNT with $W(CO)_5$THF. The [$W(CO)_5$@APy-MWCNT] was characterized by elemental analysis, scanning electron microscopy, and FTIR and diffuse reflectance UV–Vis spectroscopic methods. The nitrogen content of the catalyst was 1.17% (0.83 mmol/g). According to this value, the total amount of nitrogen, which was available for the attachment of W, was 0.41 mmol/g of support. The metal loading of [$W(CO)_5$@APy-MWCNT], measured by ICP, was 0.058 mmol/g. Based on these values, 14% of the available nitrogen sites were coordinated to $W(CO)_6$. Figure S1 (see the Supplementary Data) shows the FTIR spectra of APy-MWCNT and [$W(CO)_5$@APy-MWCNT]. The C=O stretching band of the amide group is present at 1654 cm^{-1}. Bands at 1,908, 1,857, and 1,801 cm^{-1} are assigned to the C_4v symmetry pattern of $W(CO)_5$ [34]. These observations confirm the coordination of $W(CO)_5$ to Apy-MWCNT. Further evidence for this was obtained by UV–Vis spectroscopy in the diffuse reflectance mode. The starting material $W(CO)_6$ showed two strong absorption peaks at 246 and 290 nm, attributed to W → CO charge transfer bands (Fig. 1A). These peaks were also observed in the diffuse reflectance UV–Vis of [$W(CO)_5$@APy-MWCNT] (Fig. 1B) and homogeneous $W(CO)_5$(Py) (Fig. 1C), while MWCNTs showed no absorption peak in this region (Fig. 1D). These observations indicate that tungsten carbonyl has been supported on the MWCNTs. The SEM images of the [$W(CO)_5$@APy-MWCNT] showed that the nanotubes are aggregated and retain their nanotube nature (Fig. 2).



Scheme 2 The proposed structure for [$W(CO)_5$@APy-MWCNT]

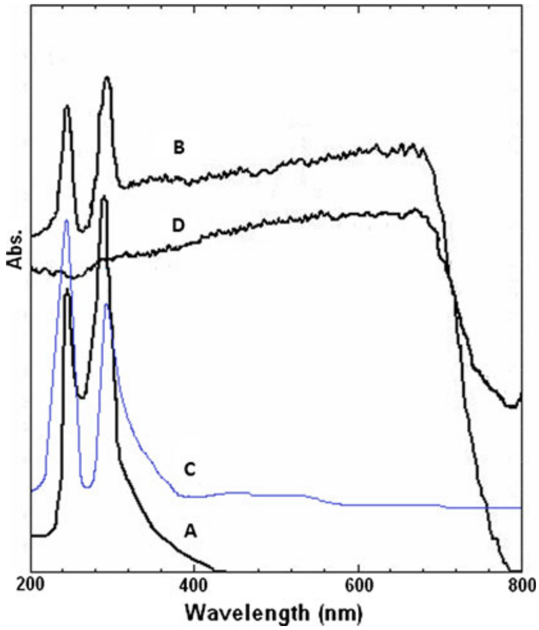


Fig. 1 UV-vis spectrum of: *A* Homogeneous $W(CO)_6$; *B* [$W(CO)_5$@APy-MWCNT]; *C* Homogeneous $W(CO)_5$(Py) and *D* MWCNT

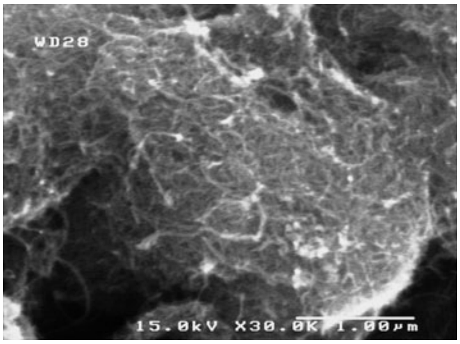


Fig. 2 SEM image of [$W(CO)_5$@APy-MWCNT]

Catalyzed epoxidation of alkenes

The activity of the prepared catalyst, [$W(CO)_5$@APy-MWCNT], was initially investigated in the epoxidation of

Table 1 Optimization of catalyst amount in the epoxidation of cyclooctene with H_2O_2 after 3 h

Entry	Catalyst amounts (mg, mmol)	Yield (%)[a]
1	0	10
2	50 mg (0.003 mmol)	78
3	100 mg (0.006 mmol)	90
4	150 mg (0.009 mmol)	93
5	200 mg (0.012 mmol)	98
6	250 mg (0.015 mmol)	98

Reaction conditions: cyclooctene (1 mmol), H_2O_2 (0.5 mL), catalyst, MeCN (5 mL)

[a] GC yield based on the starting cyclooctene

Table 2 Epoxidation of *cis*-cyclooctene with H_2O_2 catalyzed by [W(CO)$_5$@APy-MWCNT] under reflux conditions in different solvents

Solvent	Epoxide yield after 3 h (%)[a]	T (°C)
$(CH_3)_2CO$	7	53
THF	11	61
CH_3CN	98	77
$ClCH_2CH_2Cl$	33	78
$CHCl_3$	27	57
CCl_4	18	72
$CHCl_2$	24	38

Reaction conditions: cyclooctene (1 mmol), H_2O_2 (0.5 mL), catalyst (200 mg, 0.012 mmol), solvent (5 mL)

[a] GC yield based on the starting cyclooctene

cyclooctene with hydrogen peroxide. Different amounts of [W(CO)$_5$@APy-MWCNT] were investigated and the best results were obtained in the presence of 200 mg (0.012 mmol with respect to W) of the catalyst (Table 1). Different solvents were investigated and the results (Table 2) showed that the best epoxide yield was observed in acetonitrile. During the reaction, the [W(CO)$_5$@APy-MWCNT] is suspended in the solvent. This is due to the bundled agglomerates of MWCNT that aggregate slowly and allow the catalyst to remain suspended in the reaction mixture for long periods of time. Control experiments in the absence of catalyst or using MWCNT-Apy as catalyst were also performed and the results showed that the amount of epoxide was about 10%.

Under the optimized conditions, this catalytic system was applied for epoxidation of a range of alkenes (Table 3). Both cyclic and linear alkenes were efficiently converted to their corresponding epoxides. Thus, cyclooctene and cyclohexene as representative cyclic alkenes and 1-heptene and 1-dodecene as linear alkenes were efficiently converted to their corresponding epoxides. In the case of *trans*-stilbene, only the *trans*-epoxide was produced, while in the case of *cis*-stilbene, a mixture of epoxides with a high *cis/trans* ratio (9.5/1) was detected (Table 3). During the oxidation reactions, no acetamide due to the oxidation of acetonitrile was observed.

The results obtained with [W(CO)$_5$@APy-MWCNT]/H_2O_2 are compared with some previously reported epoxidation catalysts in Table 4. These comparisons show that the present catalytic system is more efficient than most of the others, except for molybdenum hexacarbonyl supported on polystyrene and MWCNTs. This is consistent with the generally higher catalytic activity of molybdenum compared to tungsten. Comparison of the catalytic activity of tungsten hexacarbonyl supported on polystyrene [35] and MWCNTs shows the effect of support in which the TOF increases from 18.8 for polystyrene to 27.2 for MWCNTs. This can be attributed to the higher surface area of MWCNTs compared to polystyrene.

Catalyst recovery and reuse

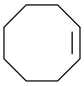
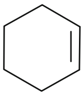
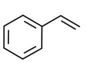
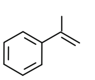
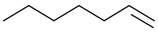
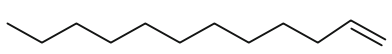
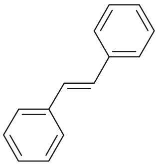
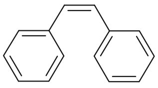
Since transition metal complexes are often expensive and toxic, the reusability of a supported catalyst is of great importance from economical and environmental points of view. Therefore, the reusability of [W(CO)$_5$@APy-MWCNT] was monitored by means of multiple sequential epoxidations of cyclooctene with H_2O_2 (Table 5). The catalyst was reused for eight consecutive experiments without loss of activity. The amount of tungsten detected in the filtrates from the first two runs was low, and after the third run, no tungsten was detected in the filtrates. These results demonstrate the strong attachment of W to the MWCNT. The catalytic behavior of the separated liquid was also tested by addition of fresh cyclooctene and H_2O_2 to the filtrates after each run. Execution of the oxidation reaction under the same reaction conditions as for the catalyst showed the same as obtained for blank experiments.

The nature of the recovered catalyst was studied by FTIR spectroscopy. No band corresponding to terminal C=O was observed in the FTIR spectrum of the recovered catalyst. This is in accordance with the mechanism previously suggested [47]. In this mechanism, C=O ligands are eliminated and W=O species are produced (Fig. S2). Therefore, $W(CO)_6$ can be considered as a catalyst precursor.

Conclusion

In conclusion, tungsten hexacarbonyl supported on MWCNTs modified with 4-aminopyridine was found to be an efficient catalyst in the epoxidation of alkenes with H_2O_2. This supported catalyst, [W(CO)$_5$@APy-MWCNT] was reactive in the epoxidation of a wide range of linear and cyclic alkenes. The catalyst was highly reusable and

Table 3 Epoxidation of alkenes with H_2O_2 catalyzed [$W(CO)_5$@APy-MWCNT] in refluxing CH_3CN

Entry	Alkene	Conversion (%)[a]	Epoxide (%)[a]	Time (h)
1		98 63[b]	98 72	3 3
2		100	100	3
3		77[c]	74	4
4		77[d]	57	3
5		100	100	3
6		25	25	4
7		90[e]	90 (*trans*-epoxide)	10
8		95[e]	86 (*cis*-epoxide) 9 (*trans*-epoxide)	10

Reaction conditions: alkene (1 mmol), H_2O_2 (0.5 mL), catalyst (200 mg, 0.012 mmol), MeCN (5 mL)

[a] GC yield based on starting alkene

[b] The reaction was carried out in the presence of $W(CO)_5(py)$ (0.012 mmol)

[c] The by-product was benzaldehyde

[d] The by-product was acetophenone

[e] Both ^{1}HNMR and GC data approved the reported yields

could be recycled eight consecutive times without loss of activity.

Experimental

All materials were commercial reagent grade and purchased from Merck or Fluka. All alkenes were passed through a column containing active alumina to remove peroxide impurities. A 400-W Hg lamp was used for activation of the metal carbonyl. FTIR spectra were obtained as KBr pellets in the range 500–4,000 cm^{-1} with a Bomen–Hartmann instrument. Scanning electron micrographs of the catalyst were taken on a SEM Philips XL 30 instrument. ^{1}H NMR spectra were recorded on a Bruker-Avance AQS 400 MHz spectrometer. Gas chromatography experiments were performed with a Shimadzu GC-16A instrument using a 2 m column packed with silicon DC-200 or Carbowax 20 M. ICP analyzes were performed on an ICP-Spectrociros CCD instrument. The products were identified by comparison of their retention times with known samples and also with their ^{1}H NMR spectra. MWCNTs containing carboxylic acid groups (the COOH content, 1.5%) (multi-wall carbon nanotubes with inside diameters between 20 and 30 nm, outside diameters between 5 and 10 nm, length 30 μm, and specific surface area more than 110 m^2g^{-1}) were purchased from Shenzen NTP Factory.

Chlorination of MWCNT-COOH

MWCNT-COOH (5 g) and $SOCl_2$ (30 mL) were mixed and refluxed under N_2 for 1 h. The reaction mixture was cooled, and the $SOCl_2$ was evaporated. The resulting precipitate is designated as chlorinated multi-wall carbon nanotubes, MWCNT-COCl (yield 92%, 4.7 g).

Preparation of MWCNT-APy

MWCNT-COCl (1 g) and Et_3N (1 mL) were added to a solution of 4-aminopyridine (0.5 g) in DMF (10 mL) and heated at 80 °C for 72 h. The mixture was then filtered and

Table 4 Comparison of the results obtained for epoxidation of cyclooctene catalyzed by [$W(CO)_5$@APy-MWCNT] with some of those reported in the literature

Entry	Catalyst	Support	Oxidant	Conditions (°C)/solvent	TOF (h^{-1})	References
1	Tungsten hexacarbonyl	MWCNT	H_2O_2	Reflux/CH_3CN	27.2	This work
2	Tungsten hexacarbonyl	Polystyrene	H_2O_2	Reflux/CH_3CN	18.8	[35]
3	Molybdenum hexacarbonyl	Polystyrene	TBHP	Reflux/CCl_4	31.3	[36]
4	Molybdenum hexacarbonyl	MWCNT	TBHP	Reflux/CCl_4	78.4	[32]
5	MTO	–	H_2O_2	RT/CH_2Cl_2	5.0	[37]
			H_2O_2	RT/–	3.3	
6	$WO_2Cl_2(OPPh_2CH_2OH)_2$	–	H_2O_2	70/Ethanol	13.8	[38]
7	[Mn(salophen)Cl]	Silica	$NaIO_4$	RT/CH_3CN–H_2O_2	7.4	[39]
8	[Mn(salophen)Cl]	Zeolite	$NaIO_4$	RT/CH_3CN-H_2O_2	2.1	[40]
9	[Mn(salen)]	Dowex MSC1	$NaIO_4$	RT/CH_3CN–H_2O_2	41.9	[41]
10	$MoO_2(acac)_2$	Silica	TBHP	Reflux/DCE	16.0	[42]
11	$MoO_2(acac)_2$	MCM-41	TBHP	Reflux/DCE	14.7	[43]
12	[Ru(salophen)Cl]	Polystyrene	$NaIO_4$	RT/CH_3CN–H_2O_2	4.7	[44]
13	[Ru(salophen)Cl]	Silica	$NaIO_4$	RT/CH_3CN–H_2O_2	4.6	[45]
14	[Mn(salophen)Cl]	MWCNT	$NaIO_4$	RT/CH_3CN–H_2O_2	8.0	[30]
15	$Mn(TNH_2PP)Cl$	MWCNT	$NaIO_4$	RT/CH_3CN–H_2O_2	9.5	[29]
16	Mn(TPP)Cl	Silica	$NaIO_4$	RT/CH_3CN–H_2O_2	8.3	[46]
	$Mn(Br_8TPP)Cl$				4.8	

Table 5 Reusability of [$W(CO)_5$@Apy-MWCNT] in the epoxidation of cyclooctene with H_2O_2 under reflux conditions after 3 h

Run	Yield (%)[a]	W leached (%)[b]
1	96	1.5
2	96	0.7
3	96	0
4	96	0
5	96	0
6	96	0
7	96	0
8	96	0

Reaction conditions: cyclooctene (1 mmol), H_2O_2 (0.5 mL), $W(CO)_5$@Apy-MWCNT (200 mg), MeCN (5 mL)

[a] GLC yield based on starting alkene

[b] Determined by ICP

the solid washed with MeCN and dried at 60 °C (yield 90%, 0.95 g). CHN analysis of *MWCNT-APy:* C: 93.1%, H: 0.3%, and N: 1.8%.

Preparation of [$W(CO)_5$@APy-MWCNT]

To activate the $W(CO)_6$, a mixture of $W(CO)_6$ (1.5 g, 4.26 mmol) in THF (50 mL) was stirred under UV irradiation for 15 min [48]. Then, MWCNT-APy (1 g) was added to the solution and refluxed for 1 h. At the end of the reaction, the catalyst was filtered off, washed thoroughly with THF and dried under vacuum (yield 94%, 0.99 g). The unreacted $W(CO)_6$ was recovered from the filtrate after evaporation of the solvent.

General procedure for epoxidation of alkenes

The appropriate alkene (1 mmol), H_2O_2 (0.5 mL) mmol), catalyst (200 mg, 0.012 mmol), and MeCN (5 mL) were mixed together and refluxed. Since different alkenes have different reactivities toward oxidation, the reactions were continued until no further progress was observed by GC. After completion of the reaction, the reaction mixture was diluted with Et_2O (20 mL) and filtered. The catalyst was thoroughly washed with Et_2O, and the combined washings and filtrate were purified on TLC on silica gel using n-hexane/Et_2O (3/1) to obtain the pure product.

Reusability of the catalyst

The reusability of the catalyst was studied by multiple sequential epoxidations of cyclooctene. The reactions were carried out as described earlier. At the end of each reaction, the catalyst was filtered off, washed thoroughly with Et_2O, dried, and reused.

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