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Multi-wall carbon nanotubes supported molybdenum hexacarbonyl: An efficient and highly reusable catalyst for epoxidation of alkenes with *tert*-butyl hydroperoxide

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ABSTRACT

The preparation, characterization and investigation of catalytic activity of $Mo(CO)_6$ supported on multi-wall carbon nanotubes modified with 4-aminopyridine is reported. The catalyst, $[Mo(CO)_5$-APy-MWCNT], was characterized by elemental analysis, scanning electron microscopy, and FT-IR and diffuse reflectance UV–vis spectroscopic methods. This new heterogenized catalyst was used as an efficient catalyst for alkene epoxidation with *tert*-butyl hydroperoxide (*tert*-BuOOH) in CCl_4 as solvent. This heterogeneous metal carbonyl catalyst showed high stability and reusability in the epoxidation reactions without loss of its catalytic activity.

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1. Introduction

Epoxides are valuable intermediates in organic synthesis for production of various chemicals such as polyethers, diols and aminoalcohols. Therefore, the catalytic epoxidation of alkenes in the liquid phase is an important area in synthetic organic chemistry [1]. Transition-metal complexes of Mo, Ti, W, and V have been found as very effective and selective catalysts for epoxidation of alkenes with hydroperoxides [2]. An important example is the Halcon process for industrial production of propylene oxide. This process is carried out by liquid phase epoxidation of propylene with alkyl hydroperoxides in the presence of a homogeneous Mo(VI) compound. A number of soluble molybdenum complexes bearing different ligands have been synthesized and employed as homogeneous catalysts for epoxidation of alkenes [3–7].

Reusable heterogeneous catalysts have attracted a great attention in organic laboratories and industries [8]. Different approaches have been used for immobilization of molybdenum complexes on various supports to obtain heterogeneous catalysts. Sherrington and coworkers have reported efficient epoxidation of alkenes with *tert*-butyl hydroperoxide catalyzed by reusable Mo(VI) supported on imidazole containing polymers [9–13]. Other organic polymers including modified polystyrenes [14–17], polyaniline [18], ion-exchange resins [19], ethylene-propylene rubber and modified poly(ethylene oxide) [20] have been used as support for immobilization of molybdenum compounds. On the other hands, several approaches have been reported for supporting of molybdenum catalysts on silica [21–25], modified MCM-41 [26–34], zeolites [35] and layered double hydroxides [36].

Carbon nanotubes (CNTs) have attracted much attention in the synthesis, characterization, and other applications because of their unique structural, mechanical, thermal, optical and electronic properties [37–39]. Since CNTs are insoluble in the most solvents, these materials can be used as catalysts support. For example, Pt nanoparticles supported on CNTs have been used for methanol oxidation [40], palladium nanoparticles supported on CNTs for semihydrogenation of phenylacetylene [41], chiral vanadyl salen complex supported on single-wall CNTs for enantioselective cyanosilylation of aldehydes [42,43] and manganese(III) porphyrin supported on MWCNTs for epoxidation of alkenes with $NaIO_4$ [44].

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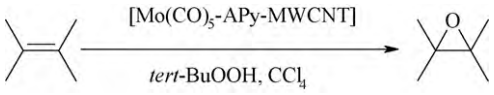


Scheme 1.

In this paper, the preparation, characterization and investigation of catalytic activity of $Mo(CO)_6$ supported on multi-wall carbon nanotubes is reported (Scheme 1).

2. Experimental

All materials were commercial reagent grade and obtained from Merck and 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 metal carbonyl. FT-IR spectra were obtained as potassium bromide pellets in the range 500–4000 cm^{-1} with a Bomen-Hartmann instrument. Scanning electron micrographs of the catalyst were taken on SEM Philips XL 30. ^1H NMR spectra were recorded on a Bruker-Arance AQS 400 MHz. Gas chromatography experiments (GC) were performed with a Shimadzu GC-16A instrument using a 2 m column packed with silicon DC-200 or Carbowax 20 M. The 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 ^1H NMR spectra. MWCNTs containing carboxylic acid groups (multi-wall carbon nanotubes with diameters between 20 and 30 nm) were purchased from Shenzen NTP Factory.

2.1. Chlorination of MWCNT-COOH

In a 100 ml round-bottom flask equipped with a condenser and a magnetic stirrer bar, MWCNT-COOH (5 g), and $SOCl_2$ (30 ml) were mixed and refluxed under N_2 atmosphere for 1 h. Then, the reaction mixture was cooled and the $SOCl_2$ was evaporated. The resulting precipitate is chlorinated multi-wall carbon nanotubes, MWCNT-COCl.

2.2. Preparation of MWCNT-APy

In a 50 ml round-bottom flask equipped with a magnetic stirring bar, MWCNT-COCl (1 g) and Et_3N (1 ml) were added to a solution of 4-aminopyridine (0.5 g) in dimethyl formamide (DMF) (10 ml) and heated at 80 °C for 72 h. Then, the reaction mixture was filtered, washed with CH_3CN and dried at 60 °C. CHN analysis of *MWCNT-APy*: C: 93.14%, H: 0.31%, N: 1.16%.

2.3. Preparation of the catalyst, [Mo(CO)5-APy-MWCNT]

First, the metal carbonyl was activated by stirring a mixture of $Mo(CO)_6$ (2 g, 7.5 mmol) in tetrahydrofuran (THF) (60 ml) under UV irradiation for 15 min [45]. Then, MWCNT-APy (1 g) was added to this solution and refluxed for 1 h. At the end of the reaction, the catalyst was filtered, washed thoroughly with THF and dried in vacuum. The unreacted $Mo(CO)_6$ was recovered after evaporation of the solvent.

2.4. General procedure for epoxidation of alkenes with tert-BuOOH catalyzed by [Mo(CO)5-APy-MWCNT]

In a 25 ml round bottom flask equipped with a magnetic stirrer bar, alkene (1 mmol), *tert*-BuOOH (2 mmol, 80% solution in di-*tert*-butyl peroxide), catalyst (100 mg, 0.017 mmol) and CCl_4 (4 ml) were mixed and refluxed. The reaction progress was monitored by GC. At the end of the reaction (since different alkenes has different reactivities toward oxidation, the reactions were continued until no further progress was observed), the reaction mixture was diluted with Et_2O (20 ml) and filtered. The catalyst was thoroughly washed with Et_2O and the combined washing and filtrates were purified on a silica gel plate to obtain the pure product.

2.5. Reusability of the catalyst

The reusability of the catalyst was studied in the repeated epoxidation reaction of *cis*-cyclooctene. The reactions were carried out as described above. At the end of each reaction, the catalyst was filtered, washed thoroughly with Et_2O, dried and reused.

3. Results and discussion

3.1. Preparation and characterization of catalyst

The specification of MWCNT-COOH used in this study is presented in Table 1. Scheme 2 shows the preparation procedure of MWCNTs supported molybdenum catalyst. The modified MWCNT, APy-MWCNT, was prepared by covalent attachment of 4-aminopyridine to MWCNT-COCl via an amide linkage. The [$Mo(CO)_5$-APy-MWCNT] catalyst was synthesized by the reaction of APy-MWCNT with a solution of $Mo(CO)_5$THF (this compound was prepared by activation of $Mo(CO)_6$ in THF under UV irradiation). The [$Mo(CO)_5$-APy-MWCNT] catalyst was characterized by elemental analysis, scanning electron microscopy, and FT-IR and diffuse reflectance UV–vis spectroscopic methods. The nitrogen content of catalyst was 1.16% (0.83 mmol/g). According to this value, the degree of nitrogen, which was available for attachment of Mo, was 0.41 mmol/g of support. The metal loading of [$Mo(CO)_5$-APy-MWCNT], which was measured by ICP, was 0.17 mmol/g. Based on these values, 42% of available nitrogen sites for anchoring to Mo, have been coordinated to $Mo(CO)_6$. The FT-IR spectra of MWCNT-APy and [$Mo(CO)_5$-APy-MWCNT] are shown in Fig. 1. The C=O stretching band of the amide group appeared at 1657 cm^{-1}. The bands at 1996, 1948 and 1898 cm^{-1} are assigned to the C_4v symmetry pattern of $Mo(CO)_5$ [46]. These observations proved the coordination of $Mo(CO)_5$ to MWCNT. UV–vis spectroscopy was employed in the diffuse reflectance mode for characterization of the supported catalyst. The supported catalyst showed a strong absorption peak at 226 nm which was attributed to Mo → CO charge transfer bands (Fig. 2A). This peak is observed in the UV–vis of $Mo(CO)_6$ (Fig. 2B), while MWCNTs showed no absorption peak in this region (Fig. 2C). These observations indicated that molybdenum hexacarbonyl has been supported on MWCNTs. The SEM images of the [$Mo(CO)_5$-APy-MWCNT] showed that the nonaotubes are aggregated and has retained their nanotube nature (Fig. 3).

3.2. Alkene epoxidation with tert-BuOOH catalyzed by [Mo(CO)5-APy-MWCNT]

The catalytic activity of the resulting catalyst was initially investigated in the epoxidation of *cis*-cyclooctene with *tert*-butyl hydroperoxide. Different solvents were used to find the reaction media. The results (Table 2) showed that the higher epoxide yield was observed in CCl_4. It seems that non-coordinating solvents such as chlorinated ones are the best solvents for oxidation reactions by molybdenum based catalysts. Different amounts of catalyst were used to optimize the catalyst amount. The best results were obtained by using 100 mg (0.017 mmol) of [$Mo(CO)_5$-APy-MWCNT]. During the reaction, the [$Mo(CO)_5$-APy-MWCNT] is suspended in the solvent. This is due to the bundled agglomerates of MWNT that aggregate slowly and let the catalyst to suspend in the reaction mixture for a long period of time. Control experiments in the absence of catalyst and using MWCNT-Apy as catalyst were

Table 1
The specification of MWCNT-COOH used in this study.

MWCNT-COOH				
Outside diameter	Inside diameter	Length	COOH content	Specific surface area
20–30 nm	5–10 nm	30 μm	1.5%	>110 m^2/g

Table 2
Epoxidation of *cis*-cyclooctene with *tert*-BuOOH catalyzed by [$Mo(CO)_5$-APy-MWCNT] under reflux conditions in different solvents[a].

Solvent	Epoxide (%)[b]	T (°C)
$(CH_3)_2CO$	No reaction	53
THF	No reaction	61
CH_3CN	18	77
$ClCH_2CH_2Cl$	52	78
$CHCl_3$	68	57
CCl_4	100	72
CH_2Cl_2	44	38
Neat reaction (solvent free)	37[c]	–

[a] Reaction conditions: *cis*-cyclooctene (1 mmol), *tert*-BuOOH (2 mmol), catalyst (100 mg, 0.017 mmol), 4 ml solvent.
[b] GC yield based on the starting cyclooctene after 45 min.
[c] 10 mmol of cyclooctene was used.

also performed and the results showed that the amount of epoxide was less than 5%.

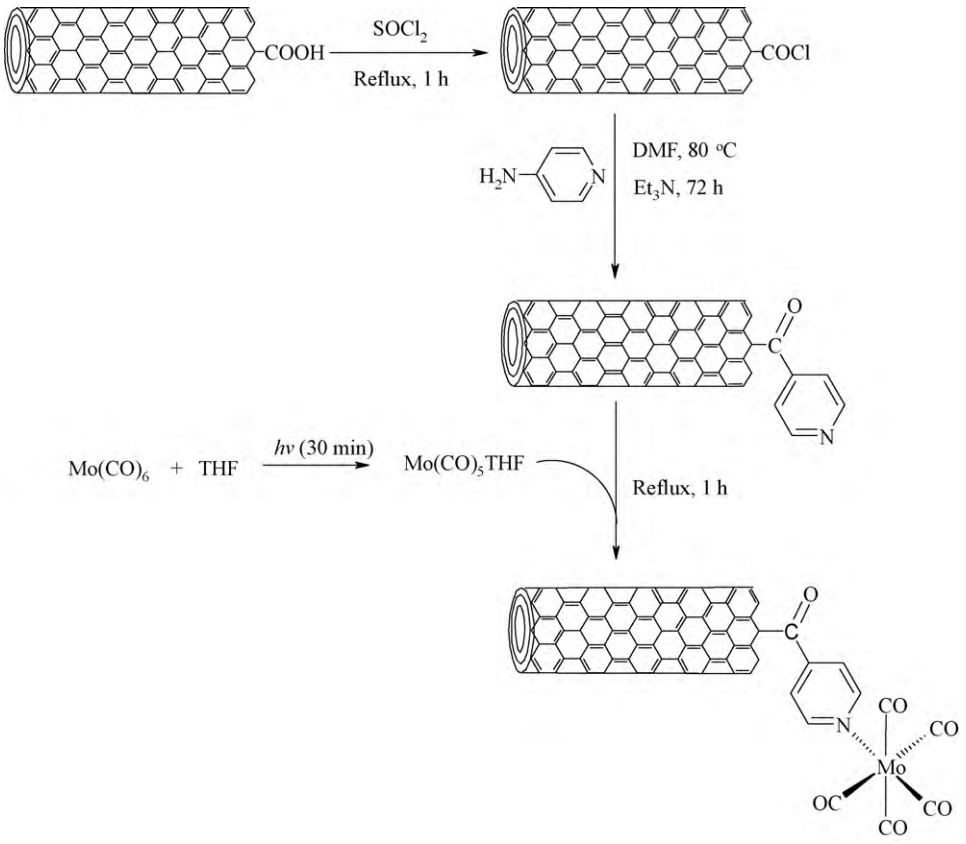
Under the optimized conditions, this catalytic system was applied to epoxidation of a wide range of alkenes (Table 3). This catalyst efficiently converted both cyclic and linear alkenes to their corresponding epoxides by using *tert*-BuOOH as oxidant. 1-Octene and 1-dodecene as linear alkenes were efficiently converted to their corresponding epoxides by [$Mo(CO)_5$-APy-MWCNT]. Stilbenes (both *cis* and *trans*) were also epoxidized by this heterogenized catalyst in high yields. *trans*-Stilbene gave only *trans*-epoxide and *cis*-stilbene produced a mixture with a high *cis*/*trans*-epoxide ratio (15/1) (Table 3). In the epoxidation reactions, about 1.7–1.85 mmol of initial *tert*-BuOOH were consumed and *tert*-butyl alcohol was produced as by-product.

In order to compare the reactivity of structurally different alkenes in the presence of this new catalytic system, the yields after 45 min are also shown in Table 3. As can be seen, cyclooctene, cyclohexene and α-methylstyrene are more reactive than the others.

In comparison with the data reported in our previous works for epoxidation of alkenes using similar catalysts [14–17], this catalytic system showed higher activity in the oxidation of alkenes (by comparison of their TOFs). One reason for this behavior may be related to the isolation of available Mo active sites which in turns increases the catalytic activity.

3.3. Catalyst recovery and reuse

Transition-metal complexes are very effective catalysts for a variety of organic reactions but one major problem, which has been associated with these homogeneous catalysts, is the recovery of catalysts from reaction medium. Therefore, the design of different functionalized supports carrying catalytically active metal species has generated considerable interest. On the other hand, the reusability of a supported catalyst is of great importance from economical and environmental points of view, because transition-



Scheme 2.

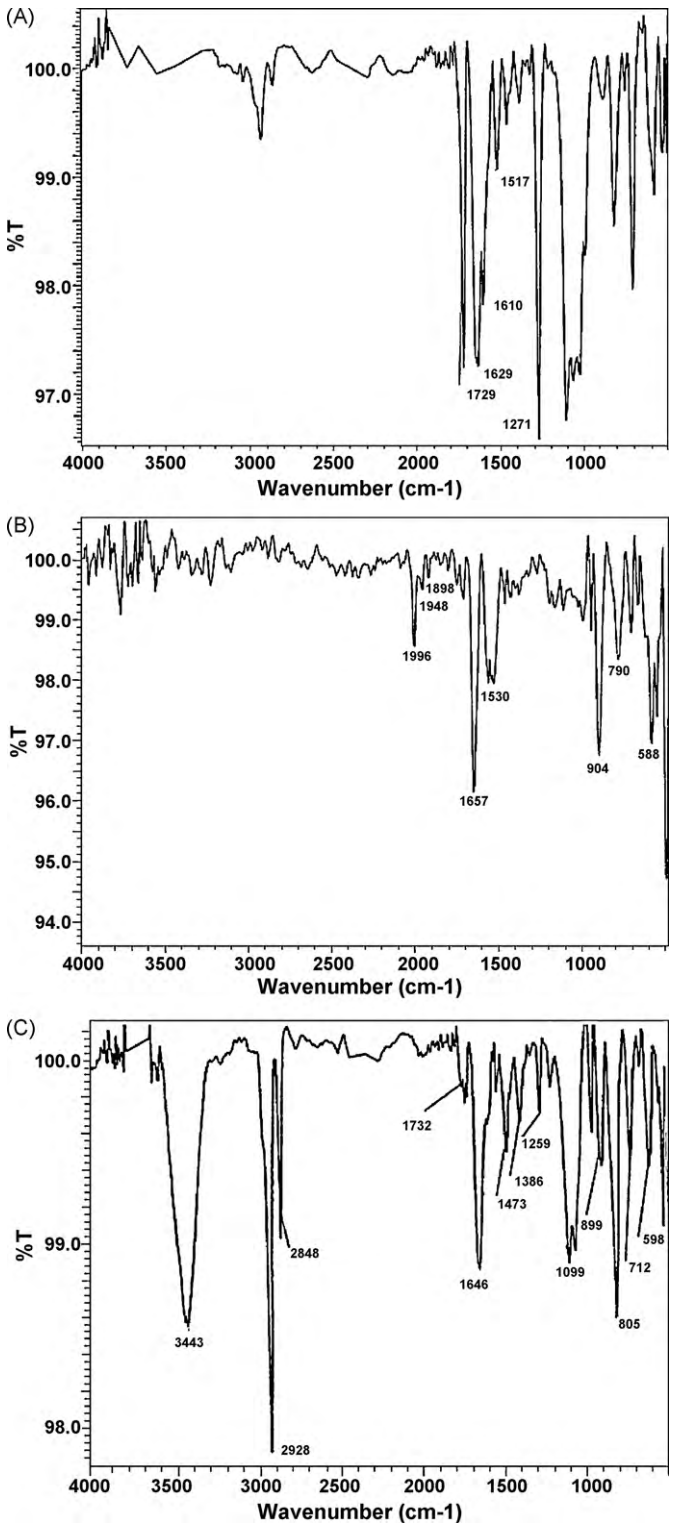


Fig. 1. The FT-IR spectrum of (A) MWCNT-APy; (B) [$Mo(CO)_5$-APy-MWCNT] and (C) recovered [$Mo(CO)_5$-APy-MWCNT].

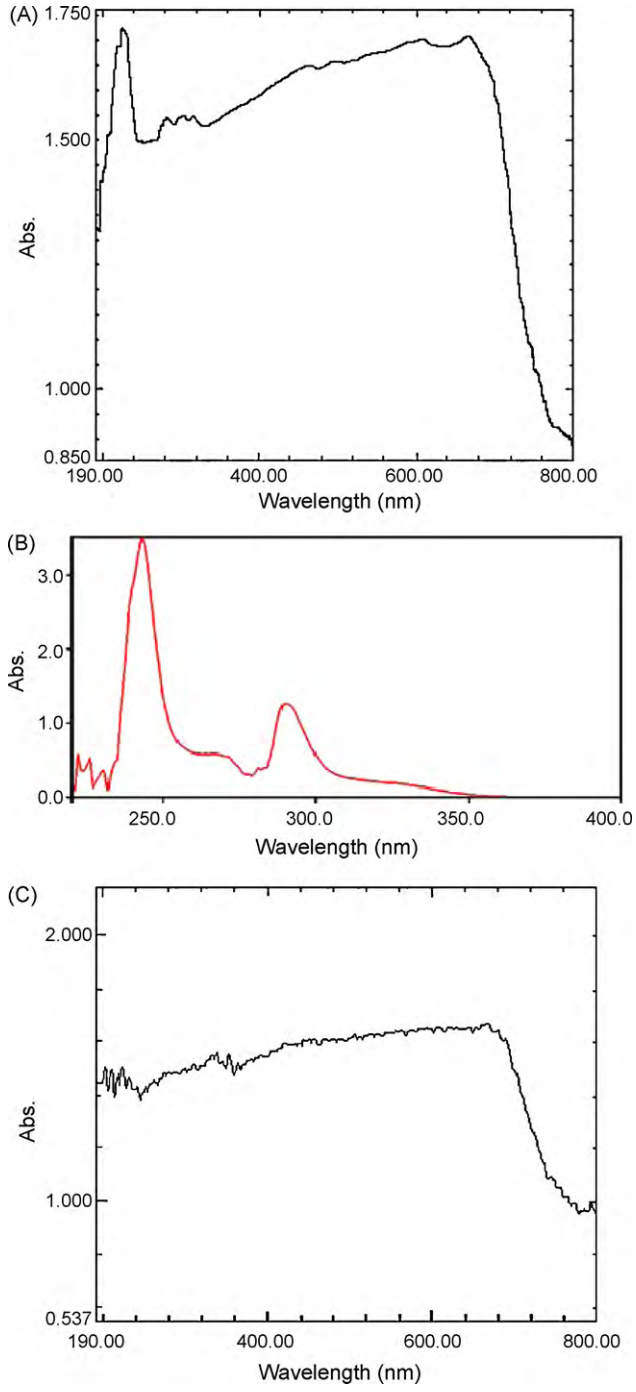
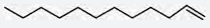
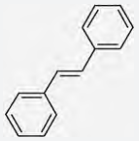
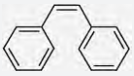


Fig. 2. The UV–vis spectrum of (A) [$Mo(CO)_5$-APy-MWCNT]; (B) $Mo(CO)_6$ and (C) MWCNT-APy.

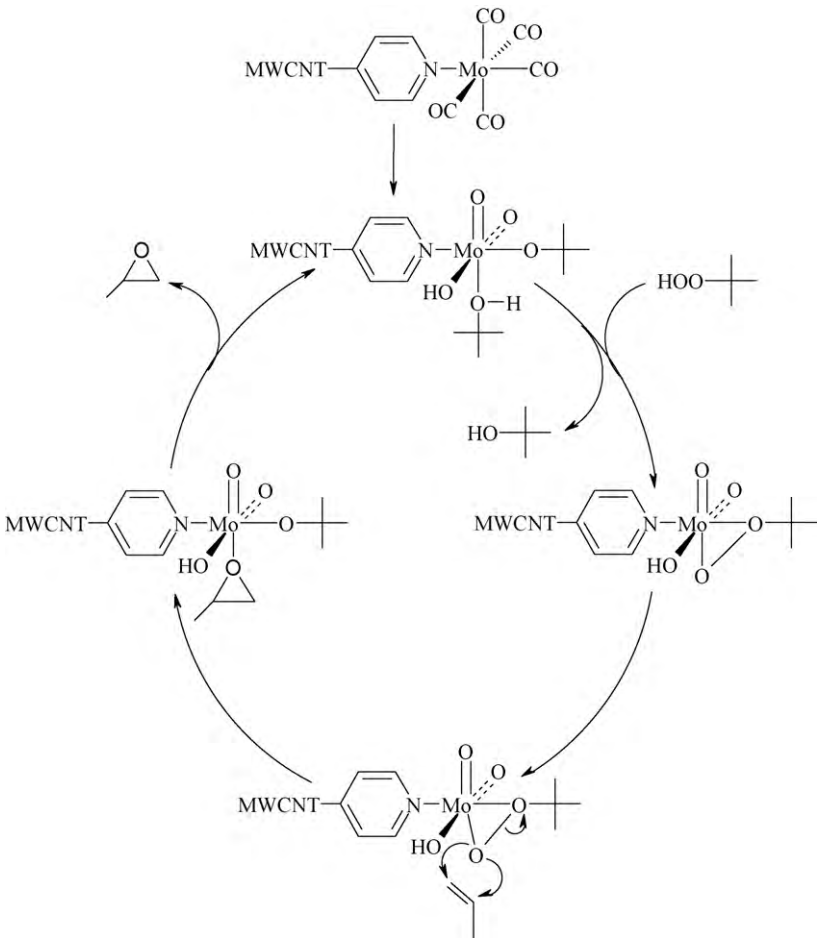
metal complexes are often expensive and very toxic. Therefore, heterogenization of homogeneous catalysts makes them useful for commercial applications. The reusability of [$Mo(CO)_5$-APy-MWCNT] was monitored by using multiple sequential epoxidation of cyclooctene with *tert*-BuOOH (Table 4). The catalyst was consecutively reused several times (10 times were checked) without loss of its initial activity. The amount of molybdenum, which was detected in the filtrates in first two runs, was low and after 3rd run no molybdenum was detected in the filtrates. These results demonstrated the strong attachment of molybdenum to the MWCNT. The catalytic behavior of the separated liquid was also tested by addition of fresh cyclooctene and *tert*-BuOOH to the filtrates after each run. Execution of the oxidation reaction under the same reaction conditions, as with catalyst, showed that the obtained results were the same as the result of blank experiments.

The nature of recovered catalyst was studied by FT-IR spectroscopy. No band corresponding to terminal C=O was observed in the FT-IR spectra of the recovered catalyst. This is in accordance with the mechanism previously reported [47]. In this mechanism,

Table 3
Epoxidation of alkenes with *tert*-BuOOH catalyzed [$Mo(CO)_5$-APy-MWCNT] in refluxing CCl_4[a].

Entry	Alkene	Conversion (%)[b,c]	Epoxide (%)[b]	Time (min)
1		100 (100)	100	45
2		90 (75)	90	75
3		97[d] (40)	90	210
4		90[e] (93)	60	35
5		75 (34)	75	160
6		85 (27)	85	240
7		100[f] (48)	99[f,g] (*trans*)	150
8		100[f] (52)	96 (*cis*), 3 (*trans*)[f,g]	150

[a] Reaction conditions: alkene (1 mmol), *tert*-BuOOH (2 mmol), catalyst (100 mg, 0.017 mmol), CCl_4 (4 ml).
[b] GC yield based on starting alkene.
[c] The numbers in the parenthesis refer to conversion after 45 min.
[d] The by-product is benzaldehyde (7%).
[e] The by-product is acetophenone (30%).
[f] Both ^{1}HNMR and GC data approved the reported yields.
[g] The by product is benzaldehyde (1%).



Scheme 3. Proposed mechanism for epoxidation of alkenes with *tert*-BuOOH catalyzed by [$Mo(CO)_5$-APy-MWCNT].

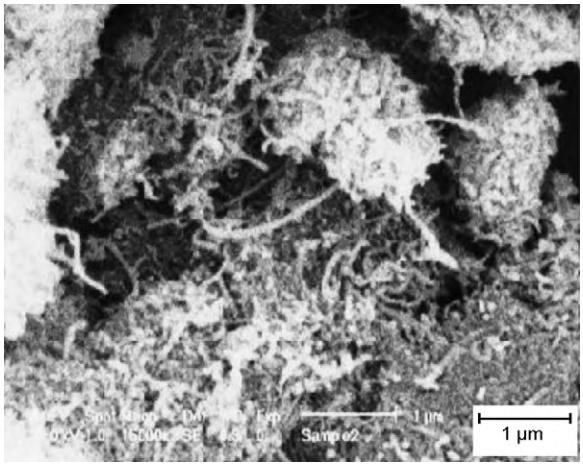


Fig. 3. The SEM image of [$Mo(CO)_5$-APy-MWCNT].

Table 4
Reusability of [$Mo(CO)_5$-APy-MWCNT] in the epoxidation of *cis*-cyclooctene with *tert*-BuOOH under reflux conditions[a].

Run	Cyclooctene oxide (%)[b]	Time (min)	Mo leached (%)[c]
1	100	45	0.6
2	100	45	0.3
3	100	45	0
4	100	45	0
5	100	45	0
6	100	45	0
7	100	45	0
8	100	45	0
9	100	45	0
10	100	45	0

[a] Reaction conditions: *cis*-cyclooctene (1 mmol), *tert*-BuOOH (2 mmol), catalyst (100 mg, 0.017 mmol), CCl_4 (4 ml).
[b] GC yield based on starting alkene.
[c] Determined by ICP.

C=O ligands are eliminated and the MoO_2 species are produced (Scheme 3). The bands at 899–1100 cm^{-1} (Mo=O) are a good indication for this explanation (Fig. 1C).

4. Conclusion

We immobilized molybdenum hexacarbonyl on MWCNTs modified by 4-aminopyridine and found that this supported catalyst was active in the epoxidation of alkenes with *tert-BuOOH*. This supported catalyst is highly reactive in the epoxidation of a wide range of alkenes such as linear and cyclic ones. The catalyst was highly reusable and recycled 10 times without appreciable decrease in its initial activity.

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