



Modified Multi-walled Carbon Nanotubes Containing Firmly Interacted Venturrello Anion: An Efficient Catalyst for Olefin Epoxidation

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Selective epoxidation of olefins for industrial applications requires heterogeneous catalysts with robust structure and higher reusability performance. The choice of solid support and catalytically active species plays an important role in improving the reaction efficiency. Herein, aminopropyl-functionalized multi-walled carbon nanotubes (NH₂-MWCNTs) were used as suitable modified solid nanomaterial to immobilize efficient epoxidation active catalyst like Venturrello anion *i.e.* {PO₄[W(O)(O₂)₂]₄}³⁻, *via* strong electrostatic interaction. The appropriate physicochemical techniques confirmed successful immobilization of Venturrello anion and synthesis of desired heterogeneous catalyst. Superior catalytic activity was achieved in the epoxidation of some olefins (*e.g.* > 99 % for cyclooctene and >86% for cyclohexene) in the presence of prepared heterogeneous catalyst with utilizing H₂O₂ as green and benign oxidant. Comparison with other similar works revealed the higher catalytic activity of prepared heterogeneous catalyst. Moreover, the stability and reusability of catalyst were examined in four catalytic cycles which approves its stability without loss of its activity.

Keywords: Carbonaceous material, Peroxophosphotungstate, Heterogenization, Epoxidation

INTRODUCTION

Carbon nanotubes (CNTs) as a member of carbon materials have been found as excellent materials for several utilizations such as catalytic reactions [1], sensors [2], supercapacitors [3] and microelectronic devices [4] owing to their prominent features including high specific surface area and electrical conductivity as well as chemical, thermal, and mechanical stability. These outstanding features make the CNTs as a suitable solid support for heterogenizing the homogeneous catalytic components [5-7].

Olefin epoxidation is a well-known organic reaction for preparing different epoxides which are substantial precursors for the preparation of valuable commercial fine chemicals, surfactants, pharmaceuticals, paints, fragrances and epoxy resins [8-11]. Besides, epoxides have shown

significant reactivity in ring-opening reactions with different nucleophiles to produce 1,2-difunctionalized species, and also they have been used in other rearrangement or reduction reactions owing to their special structure containing strained triangular heterocycles [12,13].

One of the serious issues concerned with green chemistry is the development of ecofriendly catalytic approaches using cheap and non-toxic oxidants like hydrogen peroxide (H₂O₂) or molecular oxygen (O₂) instead of organic peroxides and peracids as environmentally incompatible oxidants which can generate large quantities of waste products [12,14-16]. Hydrogen peroxide is considered as promising green oxidant for industrial oxidation processes and produces water as single by-product [17]. Therefore, it surpasses other oxidants and even gaseous O₂, because handling these types of gases is much difficult and sometimes it causes perilous explosion when mixing with organic substrates [12,18]. However, the use of hydrogen peroxide alone in the epoxidation reaction is

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hindered by its limited reactivity and selectivity. Accordingly, catalysts specially those encompassing transition metals are required to generate reactive oxidation moieties [19,20].

Polyoxometalates (POMs) are metal oxide anionic clusters that usually contain some transition metals like molybdenum, tungsten, and vanadium in their high oxidation states [21,22]. Also, the existence of vast amount of oxygen atoms in POM clusters accompanied with their high redox capability have led to their widespread use as efficient catalysts in oxidation reactions [23]. In this regard, the peroxophosphotungstate species known as Venturello anion, formulated as $\{\text{PO}_4[\text{W}(\text{O})(\text{O}_2)_2]_4\}^{3-}$, have been extensively investigated after its discovery by Venturello *et al.* in 1980s, and then attracted significant attention as efficient and environmentally friendly catalyst for selective epoxidation reaction. The biphasic catalytic system containing the aqueous (H_2O_2) and organic phases, and Venturello anion as catalyst was utilized for preparing different epoxides in mild reaction conditions [24,25].

However, the almost inseparable property and environmental impacts of using such homogeneous catalysts along with high recovery expenses have led to their limited utilization in production of epoxides with high quality [26-28]. This is why numerous solid materials such as silica [29], palygorskite [30], activated carbon [31], graphene oxide [32,33] and Zn-modified SnO_2 [34] have been utilized so far to immobilize the homogeneous Venturello anion on the surface and pores of these materials in order to overcome the drawbacks associated with homogeneous systems.

Considering the above mentioned importance of using heterogenized peroxophosphotungstate catalyst for its widespread application in selective epoxidation reaction under benign conditions using cheap and green oxidant like H_2O_2 , a new design of a catalytic system seems essential to prepare a highly active catalyst with robust structure which can be reused in several catalytic runs. To this end, (3-aminopropyl)triethoxysilane was used in this work as appropriate spacer group to modify the surface of hydroxylated MWCNTs in purpose of creating strong electrostatic interaction between the Venturello anion and aminopropyl-modified solid support. The as-prepared heterogeneous peroxotungstate catalyst was utilized in the

epoxidation of some olefins using hydrogen peroxide as benign oxidant.

EXPERIMENTAL

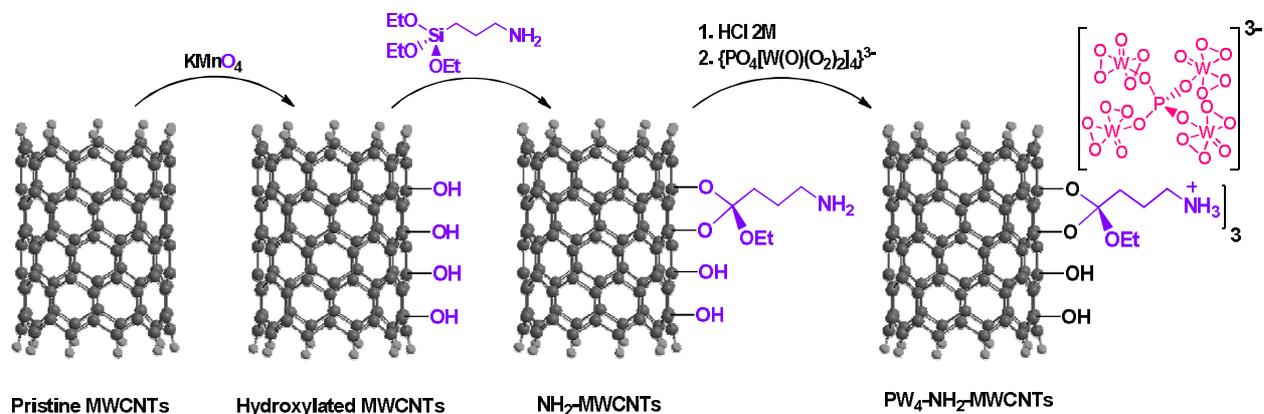
Materials and Instruments

MWCNTs was provided from Neutrino Company, Iran. The other reagents and solvents were supplied from Merck chemical company and utilized directly upon receipt. Venturello anion, $\{\text{PO}_4[\text{W}(\text{O})(\text{O}_2)_2]_4\}^{3-}$, was prepared based on reported work elsewhere [35].

FT-IR spectroscopy was performed with Perkin-Elmer Spectrum RXI FT-IR spectrometer, with pellets of materials diluted with potassium bromide. The CHN and EDX elemental analyses were accomplished with Perkin Elmer 2400 SERIES II CHN Analyzer and scanning electron microscope (FESEM, model MIRA III equipped with SAMX detector (France), TESCAN company, Czech Republic), respectively. The specific surface areas were obtained using nitrogen adsorption at $-196\text{ }^\circ\text{C}$ using Quantachrome Nova 2200, Version 7.11 instrument. The materials were outgassed at $120\text{ }^\circ\text{C}$ under high vacuum before the adsorption experiments. The transmittance electron micrograph of final catalyst was taken by Philips EM 208 S device with an accelerating voltage of 100 kV. The Agilent 6890N gas chromatograph (GC) instrument coupled with a HP-5 capillary column was used to determine the progress of catalytic epoxidation reaction.

Preparation of Venturello Anion Immobilized on Amino Propyl-modified MWCNTs, $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$

Multi-walled carbon nanotubes (MWCNTs) were initially treated in HCl (4 M) at reflux temperature for 24 h in order to dissolve metallic impurities within the MWCNTs and then centrifuged and washed with double distilled water. Afterward, 2 g of dried MWCNTs powder was suspended in CH_2Cl_2 (150 mL), followed by subsequent addition of acetic acid (50 mL), aqueous solutions of TBABr (1 g in 50 mL H_2O) and KMnO_4 (2.5 g in 50 mL H_2O). The obtained mixture was stirred at $50\text{ }^\circ\text{C}$ for 40 h. The hydroxylated MWCNTs was further washed with HCl (2 M), soxhlet washed with methanol, and finally vacuum dried at $110\text{ }^\circ\text{C}$ for 24 h.



Scheme 1. The representative preparation steps of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst

Amino propyl-modified MWCNTs ($\text{NH}_2\text{-MWCNTs}$) was prepared according to our previous report [36]. Briefly, 1 g of hydroxylated MWCNTs was suspended in 40 mL anhydrous toluene containing 1 mL of (3-aminopropyl) triethoxysilane (APTES) and refluxed for 24 h. The prepared $\text{NH}_2\text{-MWCNTs}$ material was thoroughly washed with CH_2Cl_2 to remove unreacted reagents. In order to convert amino groups to ammonium species with positive charge, the solid was treated in HCl 2 M (50 mL) and washed with methanol. Then, 1 g of acid treated $\text{NH}_2\text{-MWCNTs}$ was dispersed in 50 ml aqueous solution of Venturello anion (2 mmol) and stirred at 40 °C for 16 h. The final product, named as $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$, was separated and rinsed many times with double distilled water and vacuum dried at 110 °C.

Epoxidation of Olefins in the Presence of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$

The catalytic epoxidation procedure was performed in 25 ml round bottomed flask. In general, 0.1 g of prepared catalyst was added to the mixture of desired olefin (8 mmol) in acetonitrile (10 mL). The reaction was immediately started by addition of 30% H_2O_2 aqueous solution (1.5 mL) as oxidant. Sampling was done at specified time intervals and further diluted with appropriate solvent for GC analysis.

RESULTS AND DISCUSSION

The step by step catalyst synthesis processes is

schematically represented in Scheme 1. First, several hydroxyl groups were introduced on the surface of MWCNTs by treating pristine MWCNTs with appropriate oxidizing reagent like KMnO_4 . Then (3-aminopropyl) triethoxysilane was covalently attached to the hydroxylated MWCNTs, followed by treating with HCl to produce ammonium species. Finally, the strong electrostatic interaction between Venturello anion and positive charged ammonium groups led to the preparation of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst.

To elucidate the successful surface modification of MWCNTs with desired functional groups, FT-IR spectroscopy was carried out and the results are presented in Fig. 1. Compared to FT-IR spectrum of MWCNTs, the presence of two new peaks at 2870 and 2950 cm^{-1} in the FT-IR spectrum of $\text{NH}_2\text{-MWCNTs}$ which are associated to CH_2 stretching vibrations [37], demonstrated the covalently binding of (3-aminopropyl)trimethoxysilane on the surface of MWCNTs. Also, based on the CHN elemental analysis data given in Table 1, the nitrogen content increased to 0.89 mmol g^{-1} upon surface modification with (3-aminopropyl) trimethoxysilane reagent which further confirmed the appropriate modification method. After electrostatic interaction of Venturello anion with surface grafted ammonium groups, four individual peaks corresponding to W-O-O (810 and 892 cm^{-1}), W=O (975 cm^{-1}), and P=O (1075) stretching vibrations were appeared [38]. On the other hand, the total amount of nitrogen was decreased due to the increasing of catalyst

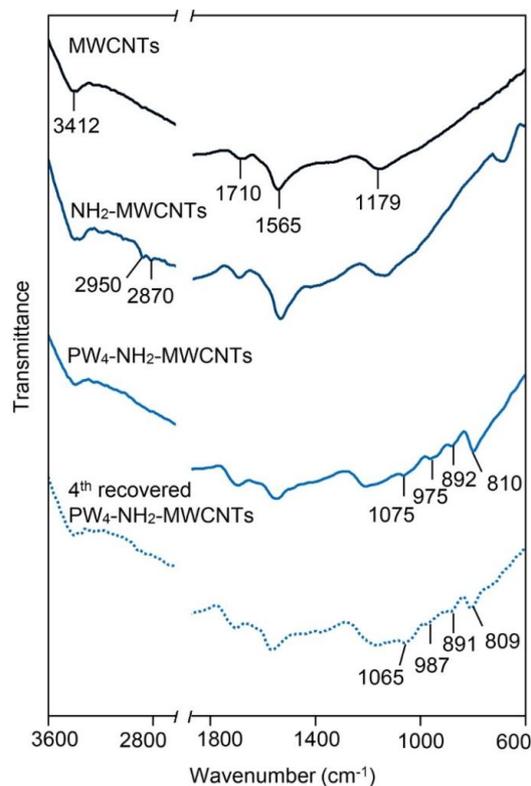


Fig. 1. The FT-IR spectra of prepared samples.

Table 1. The Results of Elemental Analysis and BET Surface Area of Prepared Materials

Sample	N ^a (mmol g ⁻¹)	P ^b (mmol g ⁻¹)	W ^b (mmol g ⁻¹)	S _{BET} ^c (m ² g ⁻¹)
MWCNTs	0.18	-	-	283
NH ₂ -MWCNTs	0.89	-	-	-
PW ₄ -NH ₂ -MWCNTs	0.71	0.03	0.35	35

^aBased on CHN elemental analysis. ^bBased on EDX elemental analysis. ^cDetermined by Brunauer-Emmett-Teller (BET) method.

weight with new elements such as phosphorous and tungsten (Table 1).

The EDX spectrum was also performed for PW₄-NH₂-MWCNTs catalyst in order to qualitatively indicate the presence of all elements in catalyst structure. As shown in Fig. 2, the relative intensity of carbon is much

higher than other elements such as silicon, nitrogen, phosphorous, and tungsten owing to the carbonaceous framework of MWCNTs solid support. Also, P and W contents were determined to be 0.03 and 0.35 mmol g⁻¹, respectively, as given in Table 1.

The N₂ adsorption-desorption isotherms and specific

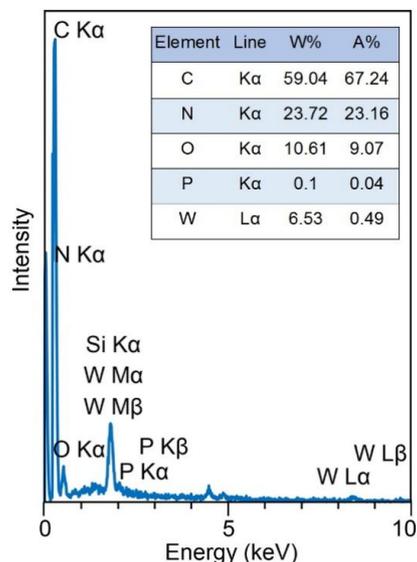


Fig. 2. The EDX spectrum of PW_4-NH_2 -MWCNTs catalyst (the inset table shows the A% and W% of each element quantitatively) .

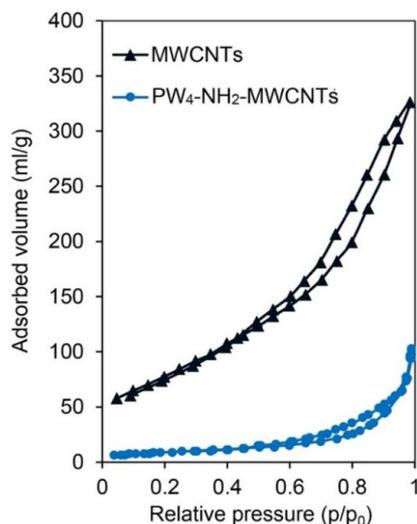


Fig. 3. The nitrogen sorption isotherms of prepared materials.

surface areas of MWCNTs and PW_4-NH_2 -MWCNTs catalyst were given in Fig. 3 and Table 1. The mesoporosity of these materials caused by agglomeration of individual MWCNTs on each other through van der waals interactions is concluded from the broad hysteresis loops in their type II isotherms [36]. Moreover, the total adsorbed nitrogen

volume was significantly decreased from MWCNTs to PW_4-NH_2 -MWCNTs catalyst. This is mainly due to the surface functionalization with APTES and Venturello anions and occupation of the large portion of created mesopores. Also, the BET surface area of PW_4-NH_2 -MWCNTs catalyst has reduced from $283 \text{ m}^2 \text{ g}^{-1}$

for MWCNTs to $35 \text{ m}^2 \text{ g}^{-1}$ for $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst, which approved the chemical surface modification with intended reagents.

The nanotube shape of one individual MWCNT containing multi-layers of graphitic platelets is well illustrated in TEM image of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst (Fig. 4). The outer and inner diameter of this nanotube was estimated to be 9.1 and 6.6 nm, respectively, which consequently showed the wall thickness of about 1.3 nm.

To examine the catalytic behavior of prepared $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst, the epoxidation of cyclooctene with H_2O_2 as a green oxidant was conducted initially and the reaction progress diagram as a function of time is represented in Fig. 5a. It was found that the cyclooctene conversion was quickly reached to 89% at 2 h, then reached to 100% at a gentle rate within 24 h. This indicates the superior catalytic activity of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst with high conversion rate at shorter reaction time (2 h). This is while the blank test showed only 6% cyclooctene conversion at 24 h, showing the high activity of immobilized peroxophosphotungstate species in $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst. In addition to the type of epoxidation active species *i.e.* Venturello anion, the obtained high catalytic performance can also be referred to the morphology of selected solid support. Herein, MWCNTs with a small size in the range of nanometers (Fig. 4) provides the greater surface for functionalization (the large surface area about $283 \text{ m}^2 \text{ g}^{-1}$, Table 1), and hence more active species can immobilize on the surface of this carbonaceous material. On the other hand, the inside of these nanotubes can be functionalized with intended moieties, and epoxidation reaction can be carried out both on the surface and inside the MWCNTs, leading to enhancement of catalytic efficiency.

Considering the high activity obtained toward cyclooctene oxide production, other olefins such as cyclohexene, 1-hexene, and 1-octene were also used as substrates in the presence of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst and the epoxidation results at 24 h was shown in summary in Fig. 5b. As a result, the less olefin conversions were observed for 1-octene and 1-hexene due to the less electron density in terminal olefins compared to cyclic ones, leading to less tendency for production of desired epoxides. Similar to our previous works using heterogenized Venturello anion

[32,38], it should be noted that the above mentioned epoxidation of different olefins only produce one product, and hence the selectivity toward corresponding epoxides can be approximately determined about 100%.

In order to show the high catalytic activity of as-synthesized catalyst, the comparison with other similar catalytic systems containing Venturello anion as active species was made in the case of cyclooctene epoxidation. Based on the data gathered in Table 2, the prepared $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst exhibited much more catalytic activity within 2 h, while other reported catalysts need longer reaction time from 5-16 h to convert cyclooctene to cyclooctene oxide as the main product. Apart from reaction time as a major factor for comparing the activity of different catalytic systems, the catalyst amount and oxidant dosage are the other factors that can affect the olefin conversion. Accordingly, the less or approximately equal amounts of catalyst and oxidant used in this work have led to outstanding catalytic result. Therefore, it can be said that the prepared $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst is highly active for epoxidation of olefins and can be used for either laboratory or industrial applications.

The reusability of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst was investigated for four successive catalytic runs for epoxidation of cyclooctene. To this end, after each catalytic run, the catalyst was simply isolated with filtration, rinsed thoroughly with methanol, and vacuum dried overnight for reusing in the next run. According to the data shown in Fig. 6, the cyclooctene conversion has reduced to 97% after 4th catalytic cycle, which showed only 3% reduction in catalytic activity. This indicates the high stability and reusability of this catalyst over four successive runs. To verify the high stability of catalyst, the FT-IR spectroscopy was carried out for 4th recovered $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst, as shown in Fig. 1. All characteristic peaks of Venturello anions are well evident, reflecting the existence of these active species in catalyst structure with no observable leaching during epoxidation reaction condition.

CONCLUSIONS

In summary, peroxophosphotungstate species known as Venturello anion, $\{\text{PO}_4[\text{W}(\text{O})(\text{O}_2)_2]_4\}^{3-}$, was strongly immobilized on the surface of aminopropylated MWCNTs

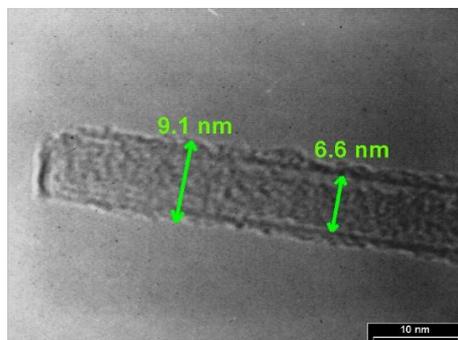


Fig. 4. The TEM image of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst.

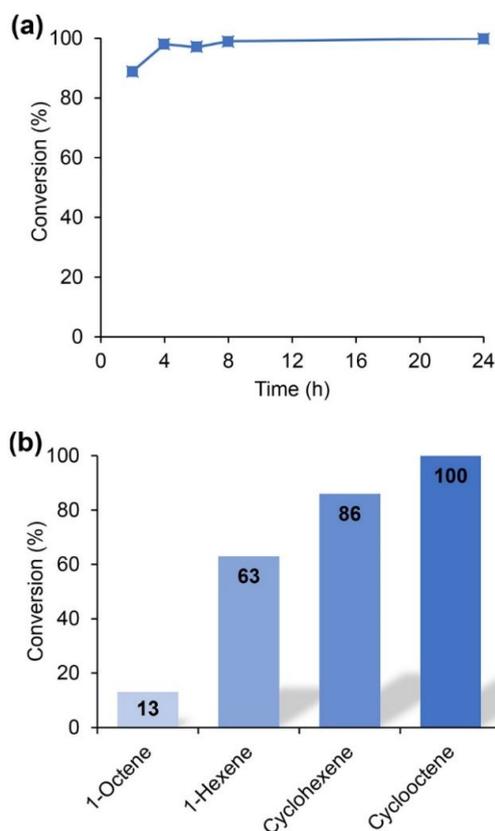


Fig. 5. (a) The kinetic profile of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst in the epoxidation of cyclooctene, (b) the epoxidation of some olefins in the presence of $\text{PW}_4\text{-NH}_2\text{-MWCNTs}$ catalyst at 24 h. Reaction conditions: olefin (8 mmol), H_2O_2 (1.5 mL), catalyst (0.1 g), acetonitrile (10 mL), reflux.

in order to achieve an efficient and stable catalyst for epoxidation of some olefins. High olefin conversion of 89% was achieved in cyclooctene epoxidation at short reaction

time (2 h) due to the presence of more electron density in this cyclic olefin compared to other cyclic and terminal olefins. The catalyst reusability after four catalytic runs

Table 2. The Efficiencies of Cyclooctene Oxide Production over $PW_4-NH_2-MWCNTs$ Catalyst in Comparison with other Different Catalytic Systems

Entry	Heterogeneous catalyst	Reaction conditions ^a	Conversion (%)	Ref.
1	$PW_4-NH_2-MWCNTs$	1:1.5, 0.44, 2	89	This work
2	PW4-C18	1:5, 6.00, 5	96	[39]
3	GO-SCMNP _s -Cyst-PW	1:1.5, 0.27, 8	~82	[40]
4	GO-SCMNP _s -APTSPW	1:1.5, 0.22, 8	~78	[40]
5	GO/MNP _s /PW	1:1.5, 0.29, 8	83	[38]
6	MCM-N ⁺ / $\{PO_4[WO(O_2)_2]_4\}^{3-}$	1:1.2, 1.00, 16	20	[41]
7	Amberlite/ $\{PO_4[WO(O_2)_2]_4\}^{3-}$	1:1.2, 1.00, 16	85	[41]

^aCyclooctene/oxidant molar ratio, catalyst amount (mol%), Time (h).

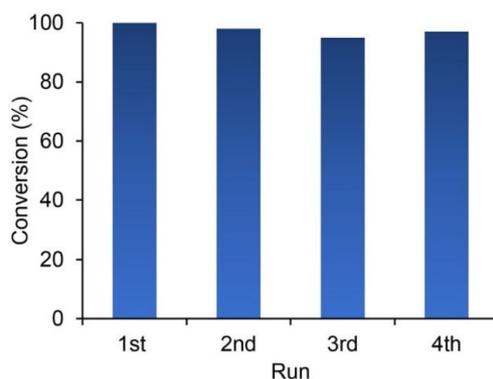


Fig. 6. The catalytic reusability of $PW_4-NH_2-MWCNTs$ catalyst in cyclooctene epoxidation at 24 h. Reaction condition: olefin (8 mmol), H_2O_2 (1.5 mL), catalyst (0.1 g), acetonitrile (10 mL), reflux.

demonstrated the maintenance of catalytic efficiency with no catalyst deactivation.

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