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Self-assembled Solvent-regulated Phase Transfer Catalyst for Epoxidation of Non-functionalized Alkenes

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Abstract Metal-organic assembly with *quasi*-salen Mn(III) active units was prepared by in situ self-assembly of linked 4,4′-methylenebis(6-(sec-butylimino)methyl) phenol ligand with manganese ion and gave comparable catalytic activity in the epoxidation of conjugated non-functionalized alkenes relative to the traditional salen Mn(III) complex. More importantly, the *quasi*-salen Mn(III) assembly could be conveniently recycled.

Keywords Self-assembled · *quasi*-salen Mn(III) complex · Alkenes · Epoxidation

1 Introduction

Epoxides of non-functionalized olefins are very important intermediates in the manufacture of drugs, vitamins, fragrances, and optical materials, because they can be readily transferred into various compounds via regioselective ring-opening or functional transfer reactions [1–5]. Consequently, there has been considerable interest in the development of highly efficient epoxidation catalysts [6, 7]. In particular, the epoxidations mediated by homogeneous nontoxic metal-organic ligand complexes, such as salen Mn(III), with various oxidants have been appreciated as one of the most efficient processes [8–12]. Owing to the difficulties in separation and/or recovery of catalyst and removal of trace amounts of metal contaminants leached from the catalysts in the products, extended efforts have been focused on immobilization of such homogeneous epoxidation catalysts on various solid/liquid supports, including inorganic porous materials [13–19], organic polymers [20], and ionic liquids [21]. Unfortunately, the large number of immobilized catalysts usually displayed less efficiency in the catalysis in comparison with their homogeneous counterparts, probably due to the inaccessibility of the reagents to the reactive centers resulted from random distribution of the reactive centers onto supports [19, 22]. Thus, it is still of academic interest and commercial importance to develop efficient and reusable catalyst for the epoxidation of non-functionalized olefins. More recently, we have reported on a novel class of solvent-regulated phase transfer salen Mn(III) catalysts [23, 24], which possess various solubility in different solvent and, consequently, the capability of "one-phase catalysis and two-phase separation". Such solvent-regulated phase transfer catalysts may open an alternative opportunity for "heterogenization" of homogeneous catalysts.

Metal-directed self-assembly of metal-organic self-assembled entities have attracted much attention in the field of catalysis [25]. Complementarity of the properties of the metal ion and the nature of organic spacer play a crucial role in self-assembly processes. Polydentate ligands can spontaneously form bridges for the control of polymerization degree and metallic ions act as catalytically active center [26]. In such way, "self-supported" catalysts can be generated by self-assembly of polydentate ligands with metal ions without using any support. More importantly, large molecular weight of metal-organic assembly would

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lower their solubility in some solvents [12, 27], making them admirable candidate for solvent-regulated phase transfer catalyst. As a part of the ongoing efforts in our laboratory to develop solvent-regulated phase transfer catalyst, herein, we reported on synthesis of a new kind of metal-organic assembly with *quasi*-salen Mn(III) active structural units via a process involving in situ self-assembly of linked 4,4′-methylenebis(6-(sec-butylimino)methyl) phenol ligand with manganese ion. The synthesized *quasi*-salen Mn(III) assembly showed typical properties of solvent-regulated phase transfer catalyst and high catalytic efficiency bearing comparison with homogeneous salen Mn(III) catalyst in the epoxidation of non-functionalized alkenes.

2 Experimental

2.1 Materials and Methods

3-chloroperoxybenzoic acid (*m*-CPBA), 4-methylmorpholine *N*-Oxide (NMO), and 4-phenylpyridine *N*-Oxide (4-PhPyNO) were purchased from Acros. Pyridine *N*-oxide (PyNO) was bought from Fluka. 2-butylamine and 2-tert-butyl phenol were purchased from Alfa Aesar. 4-(3-phenylpropyl) pyridine *N*-Oxide (4-PPPyNO) was obtained from Aldrich. Other commercially available chemicals were laboratory grade reagents from local suppliers.

^{1}H-NMR spectra of samples were taken on a Varian-400 spectrometer, using TMS as an internal standard. FT-IR spectra were obtained as potassium bromide pellets with a resolution of 4 cm^{-1} and 32 scans in the range of 400–4,000 cm^{-1} using an AVATAR 370 Thermo Nicolet spectrophotometer. UV–vis spectra of samples were recorded on an Agilent 8453 spectrophotometer. The solution of the sample in dichloromethane (ca. 1.0 mM) was poured into a 1 cm quartz cell for UV–vis adsorption with dichloromethane as the reference. Manganese ion content was measured by compleximetry with ethylenediamine tetraacetic acid (EDTA) [28]. The molecular weight was measured by Ubbelohde viscosimeter. Agilent Technologies 6890 gas chromatograph equipped with a flame ionization detector (FID) was used to monitor the reaction.

Density functional theory (DFT) was employed to evaluate the optimized geometries of the synthesized *quasi*-salen Mn(III) assembly in the Gaussian03 programs. The simulated structure of *quasi*-salen Mn(III) assembly **1** was performed with B3LYP hybrid functional [29], where Mn, Cl, O, and N used 6–31 g* basis set [30], C and H used 3–21 g* basis set [31]. For all DFT calculations, a convergence criterion of 1.0×10^{-6} a.u. was adopted for changes in energy and density matrix elements.

2.2 Catalyst Preparations

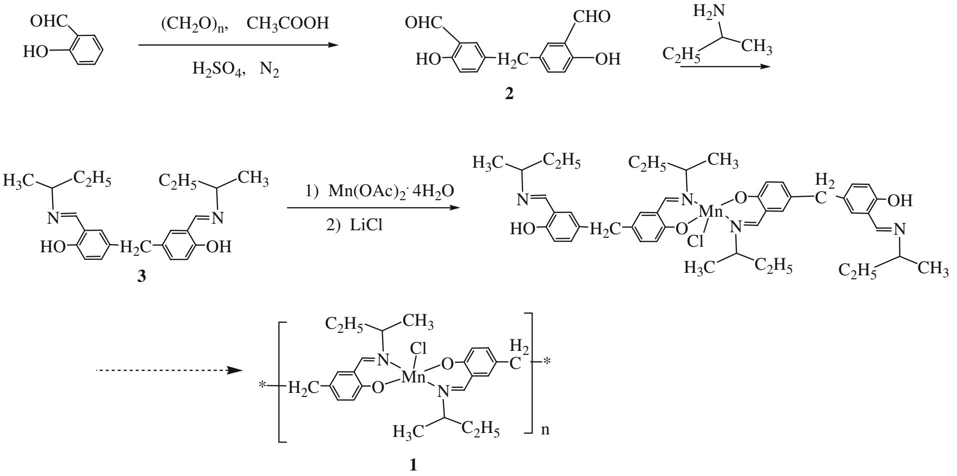
The preparation of the *quasi*-salen Mn(III) assembly **1** is outlined in Scheme 1.

2.2.1 Synthesis of 5,5′-Methylene-disalicylaldehyde (2)

5,5′-Methylene-disalicylaldehyde (**2**) was synthesized and identified according to a modified procedure reported in [32].

2.2.2 Synthesis of 4,4′-Methylenebis (6-(sec-butylimino)methyl) phenol (3)

5 mmol of **2** in 60 mL of ethanol was slowly dropwise added into 40 mL of ethanol solution containing 10.0 mmol of 2-butylamine at room temperature under nitrogen. The obtained mixture was refluxed and stirred for 8 h and then cooled in refrigerator overnight. The brilliant yellow powder of **3** was collected by filtration, followed by recrystallization from ethanol (1.41 g, 95%). ^{1}H-NMR



Scheme 1 Procedures for the synthesis of the quasi-salen Mn(III) assembly **1** ($n = \sim 265$)

(CDCl$_3$, 400 MHz): δ ppm 0.88 (m, 6H), 1.67 (m, 4H), 1.26 (d, 6 H), 3.26 (s, 2 H), 3.88 (s, 2 H), 6.57–7.18 (m, 6H), 8.20 (s, 2H), 13.75 (bs, 2H); FT-IR (KBr): 2,965, 2,925, 2,847, 1,630, 1,541, 1,458, 1,381, 1,324, 1,267, 1,220, 1,151, 1,021, 970, 921, 883, 712 cm^{-1}.

2.2.3 *Synthesis of the quasi-salen Mn(III) Assembly (**1**)*

Under nitrogen protection and vigorously stirring, 10 mL of ethanol containing 6 mmol of manganese acetate was dropwise added into 10 mL of ethanol solution containing 3 mmol of the above synthesized compound of **3** at 50 °C. The mixture was refluxed for additional 5 h, and then cooled to room temperature. 10 mL of ethanol solution containing 18 mmol of lithium chloride was added into the above mixture and followed by stirring for 3 h, and then exposed to air overnight. The resulting slurry was cooled to 5 °C for 2 h, filtered and throughout washed with 40 mL of deionized water. The obtained solid was dried under vacuum at 40 °C, giving rise to light-brown powder of **1** (1.13 g, 85%). FT-IR (KBr): 2,952, 2,904, 2,867, 1,612, 1,540, 1,463, 1,433, 1,390, 1,311, 1,252, 1,200, 1,175, 1,135, 1,084, 1,036, 570, 836, 748, 484, 412 cm^{-1}; UV–vis: 510, 416, 326 nm; the average molecular weight of the complex as measured by viscometry was ca. 120,226 ($\mathrm{Mv} = \sim 120{,}226, n = \sim 265$).

2.3 Catalytic Testing

Epoxidation of non-functionalized alkenes were typically performed according to the following procedure: 0.5 mmol of non-functionalized alkenes and 1 mmol of pyridine-*N*-oxide (PyNO) were added to 1 mL of dichloromethane containing 4% of the complex **1** (based on monomeric unit of the *quasi*-salen Mn(III) assembly **1**) under stirring. The mixture was pre-cooled to 0 °C, and then 1 mmol of *m*-CPBA was added in five equal portions over 5 min periods. Gas chromatograph was employed to monitor the progress of the epoxidation reaction. After completion of the reaction, hexane was added to extract the reaction product. The catalyst was separated from the reaction system as precipitate and subsequently used without further purification; the supernatants resulted from the catalyst separation was concentrated and purified by flash chromatography on silica gel to obtain the epoxide.

3 Results and Discussions

3.1 Characterizations of the *quasi-salen* Mn(III) Assembly **1**

The synthesized *quasi*-salen Mn(III) assembly **1** and corresponding intermediates were well characterized by ^{1}H-NMR, FT-IR and UV–vis spectra. In comparison with the compound **2**, the signal at $d = 9.86$ ppm assigned to the formyl proton (HCO) disappeared and an additional imine group (HC=N) signal at $d = 8.20$ ppm was observed for the compound **3** in ^{1}H-NMR spectra. These observations imply that the condensation reaction occurs (Scheme 1), which was further confirmed by FT-IR analyses. As shown in Fig. 1, the characteristic band of HC=O at 1,654 cm^{-1} observed in the compound **2** was replaced by the stretching band of HC=N at 1,630 cm^{-1} after the condensation reaction (Fig. 1a vs. 1b). Combining ^{1}H-NMR observations and FT-IR results, it is clear that the compound **3** has been synthesized successfully.

Furthermore, after the compound **3** was chelated with manganese ion, the stretching band of C=N at 1,630 cm^{-1} shifted significantly to 1,612 cm^{-1} (Fig. 1b vs. 1c) that is typical vibration of imine group in salen Mn(III) complex. In addition, the *quasi*-salen Mn(III) assembly **1** exhibited characteristic IR bands at 1,540, 570, and 412 cm^{-1} associated with the stretching vibration modes of C–O, Mn–O, and Mn–N [33], respectively. On the other hand, the strong absorbed peak at 342 nm observed in the UV–vis spectra of the compound **3** was shifted to 326 nm, along with the presence of two additional weak absorbed peaks at 416 and 510 nm, respectively (Fig. 2a vs. 2b), which were assigned to ligand-to-metal charge transfer band and metal-to-metal charge transfer band after the compound **3** was chelated with manganese ion. The IR and UV–vis spectra characterization results indicated that the discrete organic spacer had self-assembled with manganese ion successfully, leading to *quasi*-salen Mn(III) active structure. The manganese content in the complex **1** measured by compleximetry was 2.13 mmol/g, which is close to the

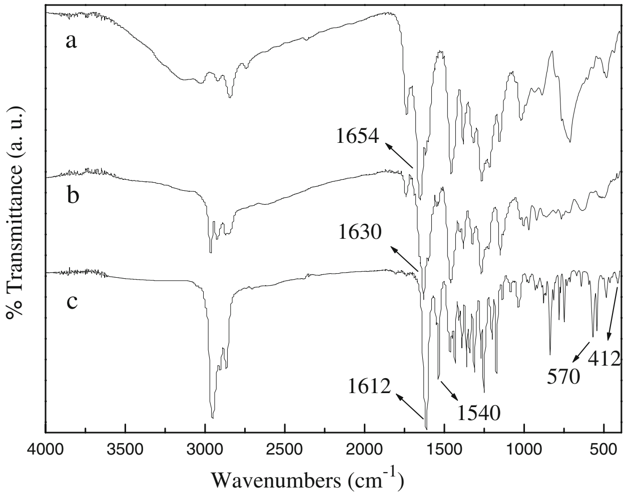


Fig. 1 FT-IR spectra of 5,5′-methylene-disalicylaldehyde **2** (**a**), 4,4′-methylenebis(6-(sec-butylimino)methyl) phenol **3** (**b**), and the *quasi*-salen Mn(III) assembly **1** (**c**)

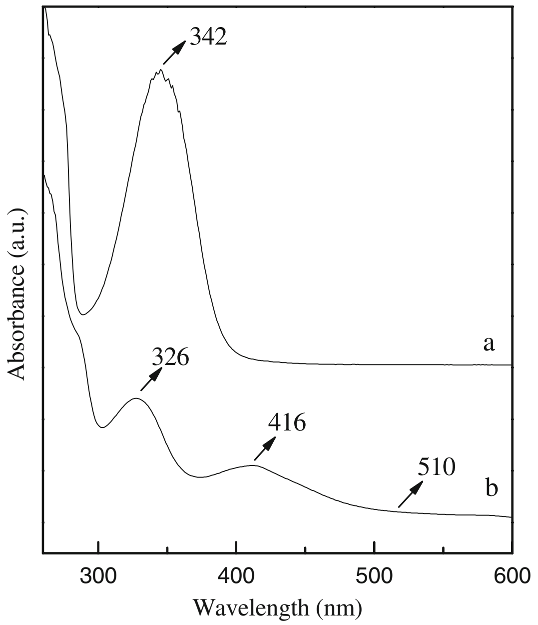


Fig. 2 UV–Vis spectra of 4,4′-methylenebis(6-(sec-butylimino)methyl) phenol **3** (**a**) and the *quasi*-salen Mn(III) assembly **1** (**b**)

theoretical value (2.20 mmol/g). The measured average molecular weight of Mv = 120,226 suggests that the complex **1** consists of ca. 265 *quasi*-salen Mn(III) active structural units.

3.2 Computational Studies

The optimized three-dimensional structure modes of the synthesized *quasi*-salen Mn(III) assembly **1** (based on monomeric unit) and the traditional salen Mn(III) complex were predicted by using the DFT at the B3LYP level in Gaussian03 programs (Fig. 3) and the corresponding central bond lengths were listed in Table 1. Clearly, the C=N, C–O, Mn–N and Mn–O bond lengths of the *quasi*-salen Mn(III) assembly **1** (mode b) were closed to those of the traditional salen Mn(III) complex (mode a). Furthermore, no imaginary frequency was observed for the *quasi*-salen Mn(III) assembly **1** in vibrational frequency analyses performed at the same level of optimization calculations. These results indicate that the structural stability of the *quasi*-salen Mn(III) assembly **1** is similar with the traditional salen Mn(III) complex.

3.3 Catalytic Tests

The synthesized *quasi*-salen Mn(III) assembly **1**, together with the traditional homogeneous salen Mn(III) complex for comparison purpose, was submitted to epoxidation of styrene as a catalyst and the corresponding results were listed in Table 2. Dichloromethane was used as a solvent in the reaction. It is found that the *quasi*-salen Mn(III) assembly **1** can be dissolved in CH_2Cl_2 solvent. The catalytic activity (turnover frequency, TOF) of the *quasi*-salen Mn(III) assembly **1** showed comparable activity than that of the traditional salen Mn(III) complex under identical conditions, which is due to the fact that the *quasi*-salen Mn(III) assembly **1** can be miscible with the reaction media (CH_2Cl_2) and, indeed, performs as homogeneous catalysis in the reaction system presented here. One of the most remarkable advantages of solvent-regulated phase transfer catalyst is the ability of "one-phase catalysis and two-phase separation" by the control of solvent. The assembly **1** presented here is immiscible with hexane, consequently, it could be easily precipitated from the reaction system by adding hexane into reaction mixture. The recovered *quasi*-salen Mn(III) assembly **1** was dissolved in dichloromethane

Fig. 3 The optimized molecular structure of the traditional salen Mn(III) complex (**a**) and the monomeric unit of the *quasi*-salen Mn(III) assembly **1** (**b**)

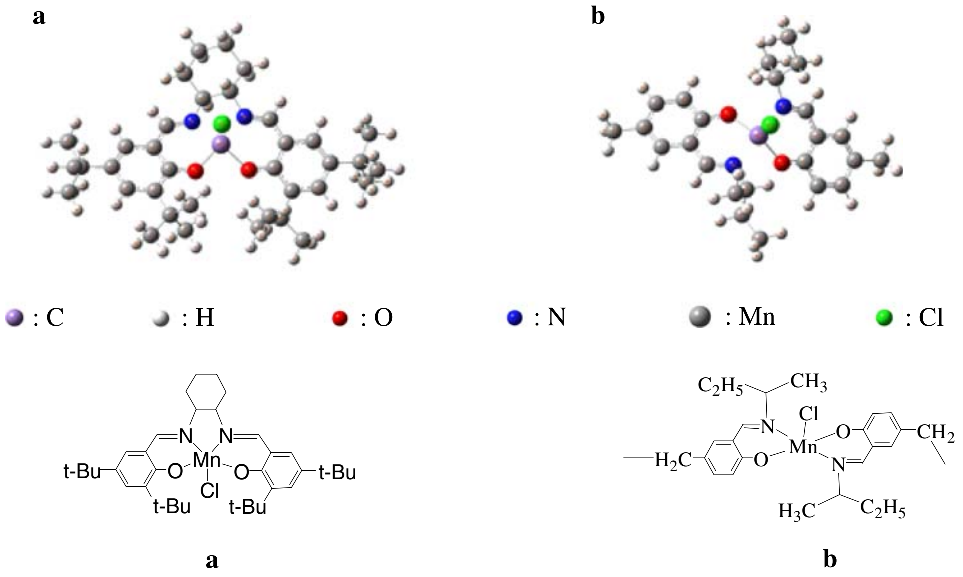


Table 1 The central bond lengths of the modes a (the traditional salen Mn(III) complex) and b (the *quasi*-salen Mn(III) assembly **1**)

Modes	$d^1_{(C=N)}$ (Å)	$d^2_{(C=N)}$ (Å)	$d^1_{(C-O)}$ (Å)	$d^2_{(C-O)}$ (Å)	$d^1_{(Mn-N)}$ (Å)	$d^2_{(Mn-N)}$ (Å)	$d^1_{(Mn-O)}$ (Å)	$d^2_{(Mn-O)}$ (Å)
a	1.297	1.298	1.319	1.312	2.028	1.983	1.866	1.902
b	1.296	1.296	1.315	1.314	2.008	2.008	1.911	1.912

The superscript of d^1 and d^2 indicated the left and right bond, respectively

Table 2 The results of the epoxidation of styrene over different catalysts

Catalyst	Run times	Yield (%)[a]	TOF × 10^{-3} (s^{-1})[b]
No	/	42	/
The traditional salen Mn(III) complex	Fresh	92	3.19
The *quasi*-salen Mn(III) assembly **1**	Fresh	97	3.30
	2nd	97	3.30
	3rd	97	3.30
	4th	97	3.30
	8th	95	3.27
The supernatants resulted from the catalyst separation	/	41	/

Catalyst (4% in 1 mL CH_2Cl_2), styrene (0.5 mmol), PyNO (1 mmol), *m*-CPBA (1 mmol), 2 h, 0 °C

[a] Yield of the epoxide

[b] Turnover frequency (TOF) is calculated by the expression of [product]/[catalyst] × time (s^{-1})

again for the subsequent catalytic runs without any further purification. As shown in Table 2, the *quasi*-salen Mn(III) assembly **1** could be reused at least 8 times without significant loss of reactivity in the epoxidation of styrene.

The maintenance of catalytic activity observed for the recovered *quasi*-salen Mn(III) assembly **1** should origin from the unique solubility and the stable structure of the metal-organic self-assembled entity. More importantly, the possibility of formation of undesired inactive μ-oxomanganese (IV) dimmers, a main reason for deactivation of homogeneous salen Mn(III) complex, can be avoided because of the severe restriction of active site in the one-dimension distribution [34]. After completion of reaction, the *quasi*-salen Mn(III) assembly **1** could be precipitated by the addition of hexane and facilely separated by decantation. The molecular weight of the recovered catalyst (Mv = 120,217) was measured which was very similar to the fresh *quasi*-salen Mn(III) assembly **1** (Mv = 120,226). And also, the chemical analysis of manganese of the recovered catalyst gave manganese content identical to that of the fresh one. The Mn content in the supernatants was further detected and no Mn leaching was found for the catalytic system. FT-IR spectra and UV–vis spectra of the *quasi*-salen Mn(III) assembly **1** with fresh and reused 8 times (Fig. 4a vs. 4b, Fig. 5a vs. 5b) indicated that no significant changes took place even after reuse for 8 times. The supernatants resulted from the catalyst separation after 8 recycles were also characterized by FT-IR spectra (Fig. 4c) and UV–vis spectra (Fig. 5c), respectively. The characteristic IR bands at 1,612, 1,540, 570, and 412 cm^{-1} of the *quasi*-salen Mn(III) assembly **1** were not observed in the spectra of the supernatants (Fig. 4c). The characteristic UV–vis absorbed peaks of the *quasi*-salen Mn(III) assembly **1** near 326, 416, and 510 nm were also absent in the spectrum of the supernatants (Fig. 5c). In addition, the supernatants were submitted to the styrene epoxidation under the same conditions, the yield of epoxide was 41%, similar to that observed in the blank reaction (Table 2). These observations suggested that there is no extraction metal complex in the supernatants, which clearly indicated that the *quasi*-salen Mn(III) assembly **1** was insoluble completely in *n*-hexane and was stable during the epoxidation reaction with no degradation of manganese complex.

It is reported that axial base plays a crucial role in the epoxidation of alkenes catalyzed by salen Mn(III) complex using *m*-CPBA as oxidant [35]. The influence of various axial bases viz PyNO, NMO, 4-PPPyNO, and 4-PhPyNO on the epoxidation of styrene were investigated. In the absence of axial bases, only 58% yield of styrene epoxide was obtained at 0 °C for 2 h even in the *quasi*-salen Mn(III) assembly **1**. Dramatic increases of the yield (97%)

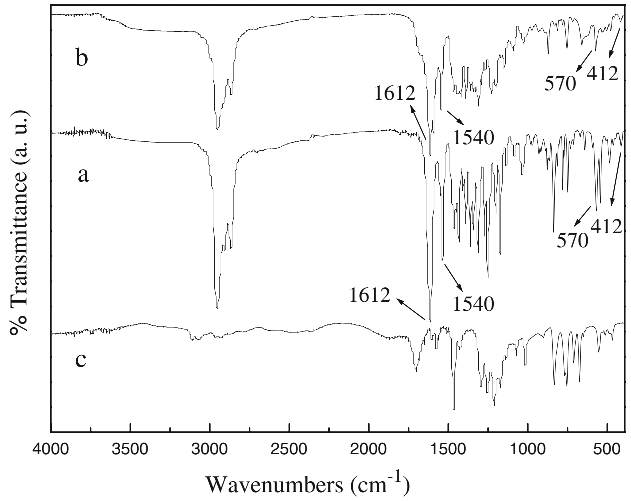


Fig. 4 FT-IR spectra of the fresh quasi-salen Mn(III) assembly **1** (**a**), after the 8th reaction (**b**) and the supernatants by addition of *n*-hexane (**c**)

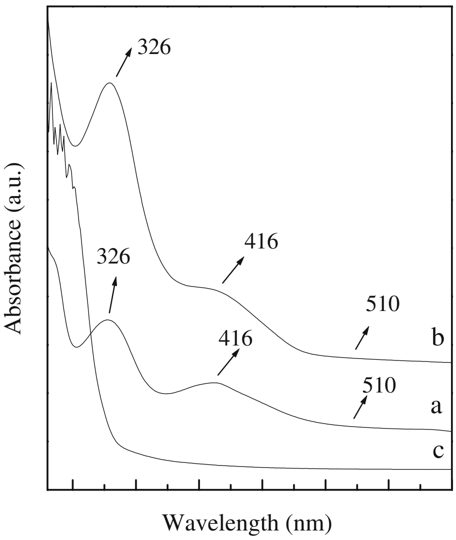


Fig. 5 UV–Vis spectra of the fresh quasi-salen Mn(III) assembly **1** (**a**), after the 8th reaction (**b**) and the supernatants by addition of *n*-hexane (**c**)

was achieved by addition of PyNO, which suggested that the axial bases were essential to the attainment of high activity in the presence of *m*-CPBA oxidant. This observation was in consonance with the report that O-coordinating axial bases could increase the activity and stability of the salen Mn(III) complex [11]. Various axial bases of NMO, PyNO, 4-PhPyNO and 4-PPPyNO were further compared in the catalytic system. It is found that the change of axial base had no significant effects on the reaction time and the yield of the styrene epoxides. Two hours should be needed to offer the most excellent yield (97%) no matter what kind of axial bases was used.

In the goal to examine the applicability of the synthesized *quasi*-salen Mn(III) assembly **1**, the catalyst was employed to catalyze the epoxidation of α-methoxyl styrene, α-methyl styrene, or cyclohexene. As shown in Table 3, the *quasi*-salen Mn(III) assembly **1** was very efficient for the epoxidations of α-methoxyl styrene and α-methyl styrene with the results comparable to that of styrene carried out under the same reaction conditions. In the case of cyclohexene, however, the yield of the epoxide was only 61%. These observations suggest that the *quasi*-salen Mn(III) assembly **1** presented here is more effective to the electron-rich conjugated alkenes, which is similar to the traditional salen Mn(III) complexes. It is perhaps due to that those electron-rich conjugated substrates are more reactive towards the active oxygen transfer species compared to the non-conjugated olefins since the salen Mn(III)-catalyzed epoxidation is a electrophilic reaction [36, 37]. Thus, the electron-rich methyl alkenes, such as styrene, α-methoxyl styrene would be expected to accelerate the epoxidation reaction rather than the electron-poor cyclohexene.

Table 3 The results of the epoxidations of various alkenes over the *quasi*-salen Mn(III) assembly **1**

Substrates	Yield (%)[a]	TOF[b] × 10^{-3} (s^{-1})
α-Methoxyl styrene	97	3.30
α-Methyl styrene	97	3.30
Cyclohexene	61	2.08

Catalyst (4% in 1 mL CH_2Cl_2), alkene (0.5 mmol), PyNO (1 mmol), *m*-CPBA (1 mmol), 2 h, 0 °C

[a] Yield of the epoxide

[b] Turnover frequency (TOF) is calculated by the expression of [product]/[catalyst] × time (s^{-1})

4 Conclusions

The synthesis of a new kind of metal-organic assembly with *quasi*-salen Mn(III) active structural units was successfully achieved via a process involving in situ self-assembly of linked 4,4′-methylenebis(6-(sec-butylimino) methyl) phenol ligand with manganese ion. Comparable catalytic activity over the *quasi*-salen Mn(III) assembly **1** relative to traditional salen Mn(III) complex was observed. More importantly, such *quasi*-salen Mn(III) assembly possessed typical properties of solvent-regulated phase transfer catalyst and can be conveniently separated from reaction system by the control of solvent. Reuse results showed the *quasi*-salen Mn(III) assembly **1** could repeatedly be used at least eight times without loss of catalytic activity.

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