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Selective oxidation of alcohols with hydrogen peroxide catalyzed by hexadentate binding 8-quinolinolato manganese(III) complexes

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ABSTRACT

A series of hexadentate 8-quinolinolato manganese(III) complexes were synthesized and proven to own a distorted octahedral geometry via elemental analysis, solid UV–vis spectroscopy and Hartree–Fock/3-21G+ calculation. These Mn(III) complexes were found to be more efficient than their corresponding tetradentate 8-quinolinolato manganese(II) and salen-$Mn^{III}OAc$ for the oxidation of alcohols in acetone medium, being due to their special hexadentate binding structures that could open an axial Mn–O bond to form the more active pentadentate structures in the presence of aqueous hydrogen peroxide, as supported by UV–vis spectra. The halogen substituents in ligand's aryl ring could significantly enhance the catalytic activities and 5-chloro-7-iodo-8-quinolinolato manganese(III) gave the highest turnover number (TON). A reasonable mechanism for the present catalytic system was proposed.

1. Introduction

The selective liquid-phase oxidation of alcohols to the corresponding carbonyl compounds is of significant importance in the fine chemicals and pharmaceutical industries. And these products represent important intermediates for the preparation of various other compounds [1,2]. Some strong oxidants such as $KMnO_4$, CrO_3, and HNO_3 [3,4] were applied traditionally to alcohol oxidation, which might result in much serious pollution and some potential risks in the process of operation. In terms of atom efficiency and environment friendly, after oxygen, aqueous hydrogen peroxide (H_2O_2) is a very attractive oxidant for industrial applications since water is the only by-product, and it is easy to be dealt with after reactions. The latest studies have therefore focused on the catalytic alcohol oxidations with H_2O_2 as the terminal oxidant [5–10]. Moreover, transition metal-catalyzed oxidation of alcohols is of current interest, and the various effective catalysts have been reported, such as molybdenum and tungsten [11–13], ruthenium [14], cobalt [15], manganese [16], iron [17] and rhenium [18]-based catalysts. However, based on the fact that most of the metal-containing catalysts are expensive and some of them may lead to the environmental pollution, some poisonous reagents are required as solvents [12,13] with a time-consuming process [11] and so on, it becomes more and more urgent to focus on finding out a more efficient and environmental friendly catalytic system.

In our previous works [19,20], we have reported that 8-quinolinolato manganese(III) complexes (Q_3Mn^{III}) could efficiently catalyze the epoxidation of olefins with H_2O_2 in water–acetone media containing ammonium acetate and acetic acid. More recently, we found that the Q_3Mn^{III} complexes could catalyze the selective oxidation of some primary and secondary alcohols with high TON and efficiency of H_2O_2 utility in acetone medium without any additives. Herein, we report the initial results we have obtained.

2. Experimental

2.1. Materials and apparatus

Benzyl alcohol, cyclohexanol, octanol-1, octanol-2, 1,2-propylene glycol, *trans*-2-hexen-1-ol, tetrahydrofuran (THF), ethanol, acetone, 50 wt% aqueous H_2O_2, 70 wt% aqueous *tert*-butyl-hydroperoxide (TBHP) and solvents were at least A.R. grade. C, H and N element analyses were performed on a Vario EL III CHN elemental analyzer and UV–vis spectrum were recorded from 200 to 700 nm on UV-3310 spectrophotometer.

2.2. Preparation of Q_3Mn^{III} complexes

The title complexes were prepared based on the published procedures with a little modification [19]. The general producing procedure is as follows: 5 ml of $Mn(OAc)_2$ aqueous solution ($1\ mol\,L^{-1}$) was added dropwise to 50 ml of stirred THF containing 15 mmol of 8-hydroxyquinoline (8-HQ) or its derivative,

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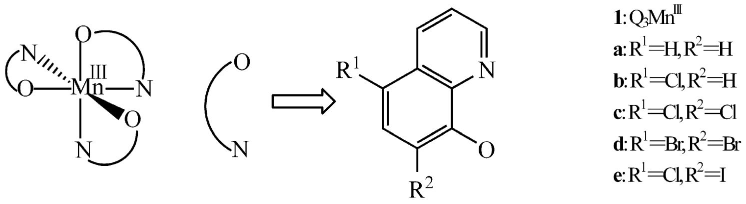


Scheme 1. The structures of Q_3Mn^{III} complexes.

followed by adding 30 wt% aqueous H_2O_2 (0.57 g, 5 mmol) into the solution. Then, the reaction mixture was refluxed for 2 h, and the resulting precipitate was filtrated and washed with ethanol for at least three times. After air-drying, a deep-yellow solid (80–92% yield) was obtained and denoted as **1a** to **1e** (see Scheme 1). The specific surface area of these Q_3Mn^{III} samples, which was measured by nitrogen volumetric adsorption-desorption method at 77 K on a Micromeritics TriStar 3000 apparatus, is very low (S_g, 10–35 $m^2\,g^{-1}$) and attributed mostly to the external porosity (>95%). For the sake of comparison, a tetradentate binding 5-chloro-7-iodo-8-quinolinolato Mn^{II} (Q_2Mn^{II}) was prepared via using two equivalents of ligand in the absence of aqueous H_2O_2. And a traditional salen-Mn^{III}OAc complex was also prepared from N,N-bis(salicylidene) ethylethylenediamine and $Mn(OAc)_2$ following reported procedures [21].

2.3. General procedure of catalytic oxidation experiments

The general procedure for alcohol oxidation is described as follows. A solution of 50 wt% H_2O_2 (3 mmol) in acetone (2 ml) was added to a stirred mixture of alcohol (9 mmol) and catalyst Q_3Mn^{III} (0.002 mmol) at room temperature (25 °C). Oxidation process for such heterogeneous mixture was carried out on a magnetic stirring apparatus with a higher stirring speed than 2500 $r\,min^{-1}$, and such stirring speed, as proven by our experiments (see entries 6 and 7 in Table 2), could efficiently eliminate the influence of mass transfer resistance on the reaction. After H_2O_2 was consumed completely via an inspection with potassium iodide-starch test paper, the catalyst was separated from the reaction mixture by filtration, and the filtrate was analyzed on an Agilent 6890N gas chromatography (GC) with a HP-5 quartz capillary column (30 m × 0.32 mm × 0.25 μm) and flame ionization detector (FID) using ultrapure nitrogen as carrier gas (rate 1.0 $ml\,min^{-1}$). Both the injector and detector temperature were 250 °C, and the column temperature was between 100 and 180 °C. The products were confirmed by GC-MS with those of the authentic samples.

3. Results and discussion

3.1. Characterizations of Q_3Mn^{III} complexes

Table 1 lists the Mn and CHN elemental contents of all the Q_3Mn^{III} complexes, wherein the found values for **1a–e** are in good agreement with those calculated from the corresponding formulae of the hexadentate structures.

Table 1
Mn and CHN elemental Contents (mass percentage) of Q_3Mn^{III} complexes.[a]

Samples		1a	1b	1c	1d	1e
Mn	Found	11.36	9.60	8.16	5.64	5.66
	Calcd.	11.29	9.32	7.93	5.72	5.68
C	Found	66.44	54.82	46.02	33.72	34.00
	Calcd.	66.50	54.90	46.68	33.71	33.45
H	Found	3.65	2.60	1.76	1.25	1.47
	Calcd.	3.70	2.54	1.73	1.25	1.24
N	Found	8.59	7.13	6.04	4.51	4.32
	Calcd.	8.60	7.12	6.05	4.37	4.34

[a] CHN elemental analyses were conducted on a Vario EL III Elemental Analyzer (Germany). Found and Calcd. list the actually measured values and the theoretically calculated ones based on a hexadentate mode, respectively. See Scheme 1 in the paper for definitions of **1a–e**.

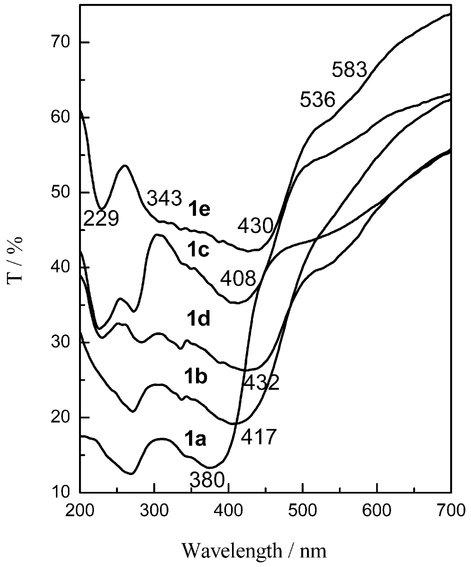


Fig. 1. Diffuse reflectance UV–vis spectra of the various solid Q_3Mn^{III} complexes (**1a–1e**).

The diffuse reflectance UV–vis spectra of all the solid Q_3Mn^{III} complexes are very similar to each other (Fig. 1), consisting of several higher energy bands in the 230–346 nm region that are probably due to the $\pi \rightarrow \pi^*$ transitions of ligands. The complexes also exhibit a strong band at ca. 394–437 nm, which should be assigned to the metal to ligand charge transfer (MLCT) absorption, as reported for other Mn(III) complexes [22]. Notably, the halogenated Q_3Mn^{III} samples commonly result in the red shift of the MLCT band, and such a red shift effect gradually strengthens with increasing the number of halogen substituents, being most likely due to the increase of electron-withdrawing effect of halogen substituents on the ligand's aryl ring. In addition, the two very weak bands in the low energy region (530–583 nm) could also be observed upon the Q_3Mn^{III} complexes, which may be attributed to the spin-allowed d–d transitions of the centered Mn ions, supporting that the Q_3Mn^{III} complexes own a distorted octahedral geometry [23].

The three dimensional structure mode of a typical sample **1e** was established by Hartree–Fock /3-21G+ with Gaussian-03 software and their Mn–O and Mn–N bond distances calculated from this mode are given in Fig. 2. The theoretical calculation results indicate that three Mn–O bonds in **1e** complex are not equal to each other, and the Mn1–O51 bond is the longest among the Mn–O bonds. A similar situation could also be noticed upon three Mn–N bonds. This should be correlated with the Jahn–Teller effect of

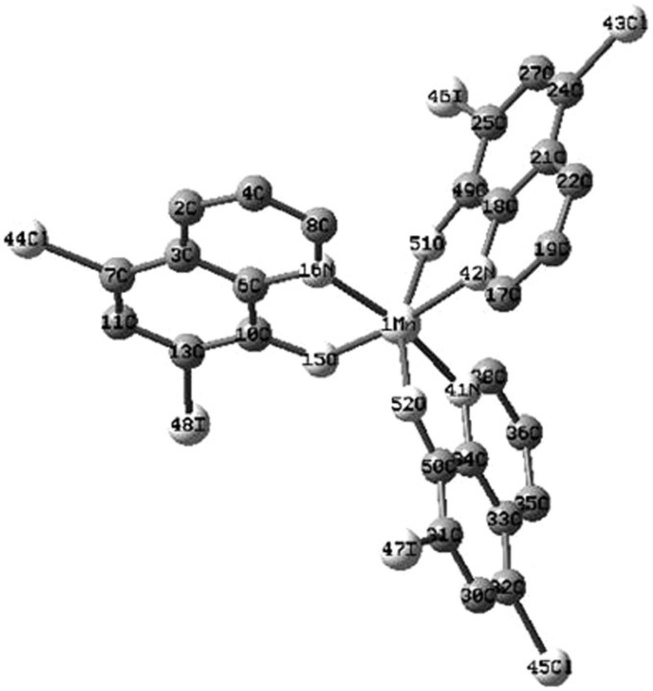
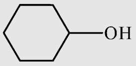
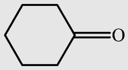
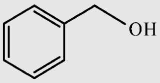
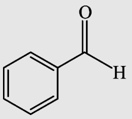
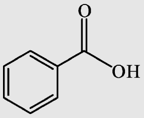
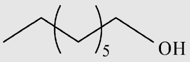
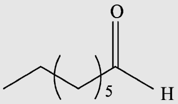
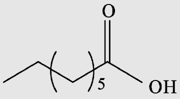
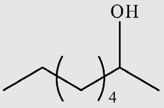
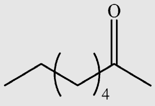
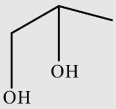
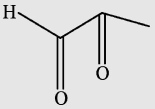
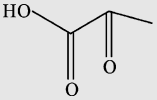
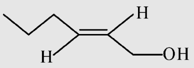
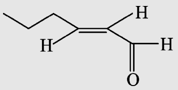
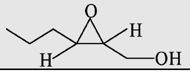


Fig. 2. The optimum three dimensional structures of Q_3Mn^{III} complex **1e**, selected bond distances (Å): Mn1–O15, 1.889; Mn1–O51, 1.919; Mn1–O52, 1.887; Mn1–N16, 2.046; Mn1–N41, 2.016; Mn1–N42, 2.061.

Mn^{3+} ions with d^4 [24], which can induce the distortion of the octahedral Q_3Mn^{III} complexes.

Table 2
The oxidation of alcohol with aqueous H_2O_2 catalyzed by Q_3Mn^{III} complexes.[a]

Entry	Catalyst	Substrate	Products	Time (h)	TON	Sel.%[g]
1	**1a**			5	630	>99
2	**1b**			5	887	>99
3	**1c**			5	988	>99
4	**1d**			5	1076	>99
5	**1e**			5	1320	>99
6[b]	**1e**			5	855	>99
7[c]	**1e**			5	1322	>99
8[d]	**1e**			5	130	>99
9[e]	Q_2Mn^{II}			5	882	>99
10[f]	Salen-Mn^{III}OAc			5	216	>99
11	**1e**			3	1205	82
						18
12	**1e**			8	610	59
						41
13	**1e**			8	915	>99
14	**1e**			8	565	91
						9
15	**1e**			8	958	56
						44

[a] Reaction condition: alcohol 9 mmol, catalyst 0.002 mmol, hydrogen peroxide 3 mmol, acetone 2 ml, stirring speed 3000 $r\,min^{-1}$ at 25 °C.

[b] Stirring speed 1000 $r\,min^{-1}$.

[c] Stirring speed 2500 $r\,min^{-1}$.

[d] TBHP as oxidant.

[e] Tetradentate binding 5-chloro-7-iodo-8-quinolinolato manganese(II) (Q_2Mn^{II}) as a catalyst.

[f] Traditional Salen-Mn^{III}OAc as a catalyst.

[g] The selectivity to alcohol oxide was based on alcohol.

3.2. Catalytic tests

Cyclohexanol oxidation with aqueous H_2O_2 was used to check the catalytic performances of Q_3Mn^{III}, Q_2Mn^{II} and salen-Mn^{III}OAc complexes, and the results are listed in Table 2. To our delight, the hexadentate Q_3Mn^{III} complexes exhibited modest to high TON and excellent selectivity for cyclohexanone in acetone medium, in which the TON followed an increasing sequence of **1e** > **1d** > **1c** > **1b** > **1a**, indicating that the 5- and 7-halogenated Q_3Mn^{III} catalysts (**1b–1e**) significantly improved the catalytic efficiency (entries 2–5). This is most likely due to the increase of electron-withdrawing effect of halogen substituents on the ligand's aryl ring, and the high resistance of halogenated 8-HQ ligands against oxidative degradation of the Q_3Mn^{III}, as reported for chlorinated porphyrin-Mn^{III} [25]. Notably, using TBHP instead of H_2O_2 as an oxidant results in a poor TON and the rapid deactivation of catalyst **1e** (entry 8). Using a tetradentate Q_2Mn^{II} gave a relative low TON compared to the corresponding hexadentate **1e** (entry 9), and a much lower TON was obtained using a traditional salen-Mn^{III}OAc as catalyst (entry 10).

Entries 11–15 further illustrate that the **1e** was also very effective for the oxidations of other alcohols. Among the various alcohols verified (Table 2), benzyl alcohol was found to be the most reactive and gave high TON (ca. 1205 in entry 11). Entries 12 and 13 illustrate that secondary aliphatic alcohol was more reactive than the corresponding primary one and gave a higher TON (915 vs 610). Notably, primary alcohols were inevitably oxidized to form their secondary oxidation products carboxylic acids (entries 11, 12, 14); whereas secondary alcohols were oxidized to only form the corresponding ketones without C–C chain cleavage (entries 5 and 13). Entry 14 shows that the primary and secondary hydroxyls in 1,2-propandiol could be simultaneously and equiprobably oxidized to form pyruvic aldehyde (91%). Finally, entry 15 illustrates that the hydroxyl and C=C bond groups of trans-2-hexene-1-ol could be nearly equiprobably oxidized to form the corresponding aldehyde and epoxide respectively, but the simultaneous oxidations of the two groups did not occur.

In the following experiment, the effect of reaction time on the catalytic efficiency and goal product selectivity was investigated using **1e**-catalyzed benzyl alcohol oxidation and the results are illustrated in Fig. 3, Wherein the TON gradually increases be-

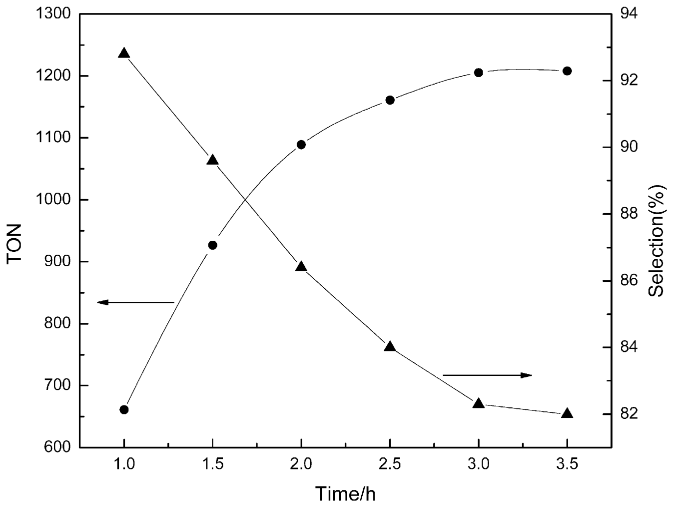


Fig. 3. The time-dependent TON and benzaldehyde selectivity for **1e**-catalyzed the oxidation of benzyl alcohol with Hydrogen peroxide.

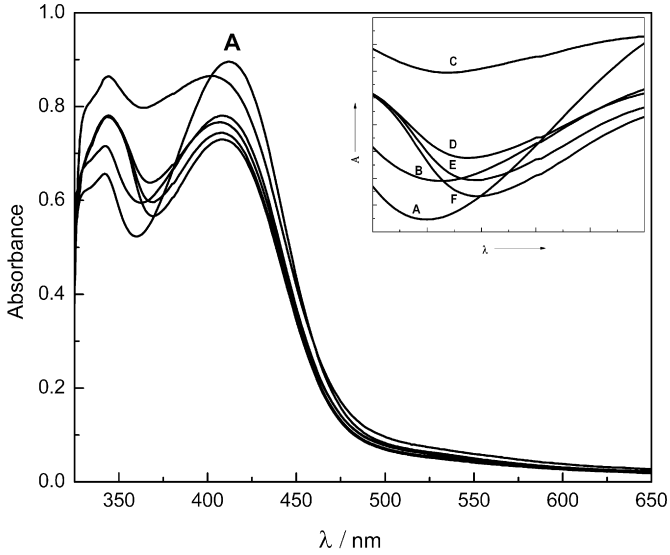


Fig. 4. UV–vis spectra of (A) a solution of **1e** in acetone (6.0×10^{-5} M, 5 ml) at 10 °C; (B) 12 h later of adding 50 wt% H_2O_2 (10 equiv relative to **1e**) to (A); (C) 12 h later of adding cyclohexanol (200 equiv relative to **1e**) to (B); (D) 24 h later; (E) 36 h later; (F) 48 h later. Inset was the amplified traces.

fore 3 h and reaches a plateau after 3 h; while benzaldehyde selectivity slowly decreases in whole reaction process, in consequence of benzaldehyde to be uninterruptedly oxidized to benzoic acid.

3.3. Catalytic mechanism

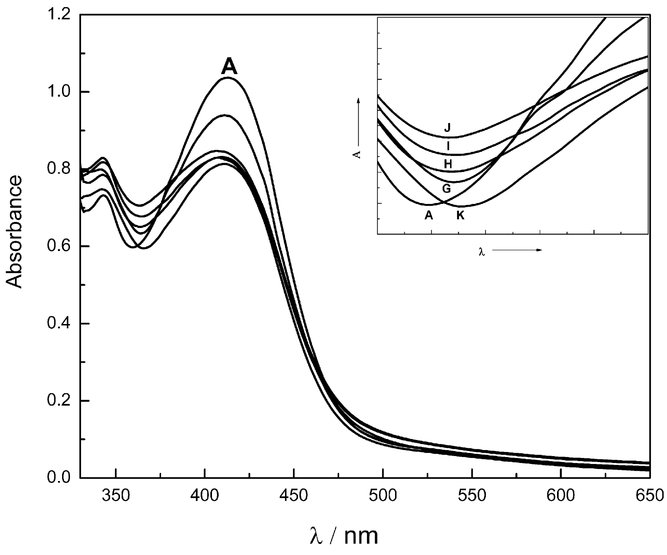


Fig. 5. UV–vis spectra of (A) a solution of **1e** in acetone (8.0×10^{-5} M, 5 ml) at 10 °C; (G) 6 h later of adding 50 wt% H_2O_2 (10 equiv relative to **1e**) and cyclohexanol (200 equiv relative to **1e**) to (A); (H) 12 h later; (I) 24 h later; (J) 36 h later; (K) 48 h later. Inset was the amplified traces.

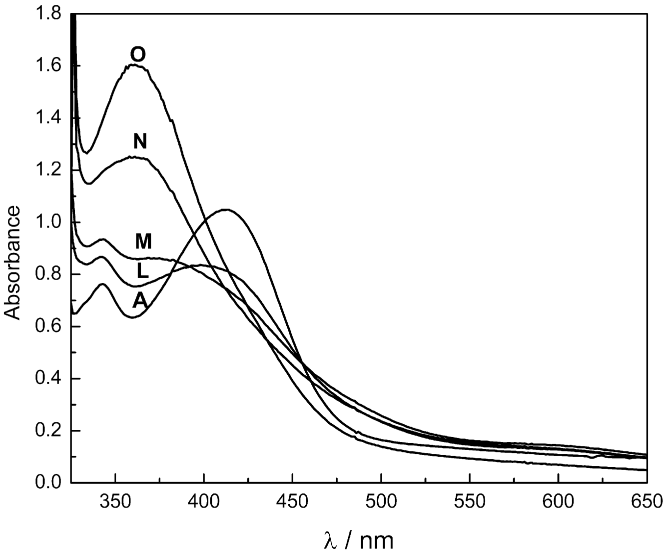
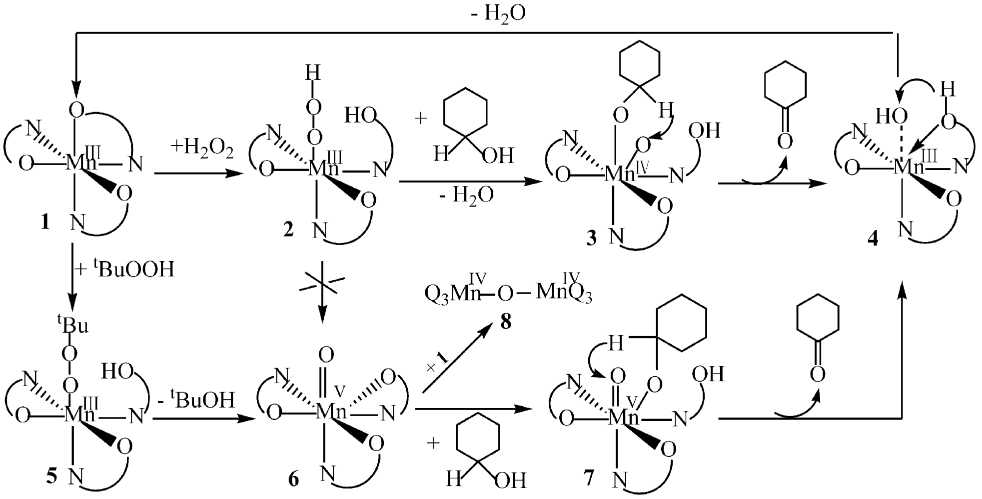


Fig. 6. UV–vis spectra of (A) a solution of **1e** in acetone (8.0×10^{-5} M, 5 ml) at 10 °C; (L) 6 h later of adding TBHP (10 equiv relative to **1e**) to (A); (M) 12 h later; (N) 1 h later of adding cyclohexanol (200 equiv relative to **1e**) to (C); (O) 36 h later.

The **1e** oxidation system was further examined by UV–vis spectroscopy in order to investigate the catalytic mechanism for the Q_3Mn^{III}-catalyzed alcohol oxidation, and the results are illustrated in Fig. 4, Wherein A is the spectrum of **1e** in acetone where an absorption band with $\lambda_{max} \sim 412$ nm is assigned to the MLCT [22]. When the A solution was treated with aqueous H_2O_2 for 12 h, the MLCT absorbance decreased, whereas the trace in 365-382 nm ran up (see B and its inlet in Fig. 4), which may be due to the cleavage of an axial Mn–O bond of **1e** to form an intermediate. Then, when adding cyclohexanol to the B solution further, the trace in 350–390 nm continuously ran up in 24 h (C), implying that cyclohexanol interacted with this intermediate to form a new intermediate. Finally, after 48 h, the trace in 350–390 nm fell back toward the trace A (F vs A), being likely due to the disappearance of such a new intermediate. We must note that: there is no clear absorbance around 500 nm designed to the ligand to metal charge transfer of the $Q_3Mn^V{=}O$ in the whole process, indicating that the $Q_3Mn^V{=}O$ should not be responsible for the oxidation of alcohols using H_2O_2 as an oxidant. If cyclohexanol and H_2O_2 were added to the A solution simultaneously, the spectral change in the simulation of real oxidation system (see Fig. 5), as expected, is very similar to that of the above described, thus indicating that the spectral information given in Fig. 4 can present the real situation of the present oxidation system.

The above experiments were repeated using TBHP instead of H_2O_2 and the obtained UV–vis spectra are illustrated in Fig. 6. Trace L is the spectrum of adding TBHP to a solution of **1e** in acetone, in which a new absorption band centered around 500 nm



Scheme 2. Possible catalytic cycle for the Q_3Mn^{III}-catalyzed cyclohexanol oxidation.

was developed and the maximum of which is obscured by the tailing of strongly absorbing species at $\lambda_{max} > 500$ nm due to the formation of the $Q_3Mn^V{=}O$, as observed similarly in the epoxidation of olefins catalyzed by salen-Mn^{III} with using PhIO and TBHP as oxidants [26]. On the other hand, another absorption band centered at 360–370 nm increased continuously (traces L and M) even after adding cyclohexanol to this solution (trace N), and was finally developed to a very strong absorbance at $\lambda_{max} \sim 364$ nm correlated to the disappearance of the $Q_3Mn^V{=}O$ species (trace O). Notably, the MLCT band of Q_3Mn^{III} at $\lambda_{max} \sim 412$ nm continuously decreased and finally disappeared in the whole process. Correlating with the poor results obtained using TBHP (entry 8 of Table 2), we assume that the absorption band at $\lambda_{max} \sim 364$ nm should be assigned to the MLCT absorption of μ-oxomanganese(IV) dimer, which is directly responsible for catalyst deactivation. In addition, the formation of such deactived species (Eq. (1)) probably undergoes a pathway proposed by Srinivasan et al. for the salen-Mn^{III}-catalyzed epoxidation of olefins [26].

$$Q_3Mn^V{=}O + Q_3Mn^{III} \rightarrow Q_3Mn^{IV}\text{–}O\text{–}Mn^{IV}Q_3. \qquad (1)$$

Based on the above findings, a mechanism for the Q_3Mn^{III}-catalyzed alcohol oxidation with H_2O_2 (using cyclohexanol as an example) is proposed as follows (see Scheme 2). Wherein the Q_3Mn^{III} (**1**) can easily interact with H_2O_2 to form a pentadentate Q_3Mn^{III}–OOH species **2** with pendant hydroxyl group through opening an axial Mn–O bond, which corresponds to the decrease and slight blue-shift of the MLCT band observed in the UV–vis spectra. Then, the **2** reacts with cyclohexanol to give a corresponding adduct **3**, which may result in the obvious blue-shift and increase of the MLCT band due to the **3** belonging to a Mn^{IV}-coordinated complex. The **3**, on the loss of the water molecules, eventually gives cyclohexanone and the regenerated catalyst **1** after undergoing the pathway to form an intermediates **4**, as reported by Chaudhari and Sawant for tungstic acid-catalyzed benzyl alcohol oxidation with aqueous H_2O_2 [27]. Notably, in the case of using TBHP as an oxidant, the Q_3Mn–O–OR (R = *tert*-butyl) **5** formed from the reaction of the **1** with TBHP may react infrequently with cyclohexanol due to the stereo-hindrance effect of its *tert*-butyl. Therefore, it has to form the $Q_3Mn^V{=}O$ (**6** in Scheme 2) through the heterolytic cleavage of O–O bond, as observed in the UV–vis spectra. In the following process, the **6** as a strong oxidant [28], may directly oxidize cyclohexanol to cyclohexanone and simultaneously give the regenerated catalyst after undergoing the pathways to form the intermediates **7** and then **4** (see Scheme 2). On the other hand, the **6** may react with another Q_3Mn^{III} to form the deactivated species **8**, as supported by the above reaction and characterization results.

4. Conclusions

In summary, for the first time we have developed the hexadentate Q_3Mn^{III} complexes as effective catalysts for the oxidation of alcohols to the corresponding carbonyl compounds in acetone medium. Besides, the present catalytic system has some distinct characteristics as follows: (a) using only acetone as a solvent without any additives; (b) employing very small amount of catalyst (0.02 mol%); (c) giving high H_2O_2 efficiency; (d) facile operation. Having found this kind of Q_3Mn^{III} complexes, we are interested in exploiting the preparations of other 8-quinolinolato metal complexes and their application in organic oxidations.

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