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Catalytic epoxidation of olefins and hydroxylation of alkanes with sodium periodate by water-soluble manganese(III)salen

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Abstract

The catalytic activity of a water-soluble Mn(salen)OAc complex in the epoxidation of alkenes and hydroxylation of alkanes was studied in acetonitrile, at room temperature, using sodium periodate as an oxygen source. The effect of various axial ligands as co-catalyst such as triethylamine, diethylamine, piperidine, 4-cyanopyridine, 2-methylpyridine, 4-methylpyridine, 4-*tert*-butylpyridine, 2-methylimidazole, pyrazine, quinalidine, morpholine, triphenylphosphine and dimethylformamide were investigated in the epoxidation of cyclooctene. Imidazole as a strong π-donor was the best axial ligand. The effect of different solvents was studied in the epoxidation of cyclooctene and CH_3CN/H_2O was chosen as solvent. The effect of the oxygen donors such as $NaIO_4$, Bu_4NIO_4, $KHSO_5$, H_2O_2, H_2O_2/urea, NaOCl and *tert*-BuOOH was also studied in the epoxidation of cyclooctene where $NaIO_4$ was selected as an oxygen donor.

Keywords: Alkenes; Alkanes; Mn(salen); Epoxidation; Hydroxylation; Periodate

1. Introduction

Transition metal complexes with Schiff base and porphyrin ligands have been extensively used as models for the heme containing cytochrome P-450 [1]. Cytochorome P-450 catalyzes a wide variety of reactions including oxygen transfer to heteroatoms, epoxidation of olefins, hydroxylation of aromatic hydrocarbons and oxidative degradation of chemically inert xenobiotics such as drugs and environmental contaminants [2]. On the other hand, metal complexes of salen and salophen ligands have been used as reagents and catalysts in many reactions including olefin epoxidation, nucleic acid modification, electrochemical reduction, alkane hydroxylation, Diels–Alder transformations, carboxylic acid decarboxylation, amines oxidation and medicinal studies as models for mimicing the superoxide dismutase [3–11]. For mimicing the function of cytochrome P-450, the transition-metal Schiff base systems have several advantages such as: (i) ready availability and simple preparation; (ii) low cost, and (iii) high activity.

Mn(III) complexes of salen type ligands have attracted much interest in the last few decades because of their unique catalytic activity, especially as epoxidation catalysts, in the presence of terminal oxidants like iodosylbenzene, sodium hypochlorite, *tert*-butylhydroperoxide and hydrogen peroxide [12–20]. These complexes catalyze the transfer of oxygen atoms to organic substrates and the nature of products depends on several factors such as substrate, oxidant, counterion, solvent, structure of salen ligands and the kind of axial ligand [21,22].

In biomimetic works, however, the preferred systems are oxidation in aqueous conditions. Water-soluble Schiff base complexes have mainly been used as DNA cleavage catalysts [23–27]. These water-soluble catalysts can be used as catalyst for the oxidation of pollutants in water such as phenol derivatives (lignin and DDT). Numerous salen-type complexes have been synthesized and investigated in relation to a wide variety of reactions. However, the drawback of most of the complexes has been their limited solubility in aqueous media. To over-

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Table 1
Effect of various oxidant on the epoxidation of cyclooctene with Mn(salen)

Oxidant	Solvent	Epoxide yield (%)[a] after 15 min
$NaIO_4$	CH_3CN/H_2O	98
Oxone ($KHSO_5$)	CH_3CN/H_2O	92
H_2O_2	CH_3CN	87
H_2O_2/urea	CH_3CN	44
NaOCl	CH_3CN	31
tert-BuOOH	CH_3CN	23
Bu_4NIO_4	CH_3CN	38

[a] GLC yields based on the starting cyclooctene.

come this limitation, we have synthesized a water-soluble complexes containing methyl(triphenylphosphonium chloride) substituents in the ligand and investigated its application in the epoxidation of olefins and hydroxylation of alkanes by $NaIO_4$ at room temperature for the first time. We have attempted to assess the effect of axial ligand type, the choice of the metal center and the influence of the oxygen source on catalyst activity.

2. Results and discussion

2.1. The effect of terminal oxidants in the epoxidation of cyclooctene catalyzed by Mn(salen)OAc

In the catalytic epoxidation of alkenes the choice of oxygen donor and solvent is crucial. In this study we examined different oxidants such as NaOCl, $NaIO_4$, Bu_4NIO_4, H_2O_2, $KHSO_5$, *tert*-butylhydroperoxide, urea–H_2O_2 (UHP) in the oxidation of cyclooctene. The results are summarized in Table 1. When NaOCl, H_2O_2, *tert*-butylhydroperoxide and urea–H_2O_2 (UHP) were used as the oxygen source in acetonitrile or dicholoromethane, Mn(salen)OAc, produced low cyclooctene oxide. Oxone ($KHSO_5$) is a strong, cheap and versatile oxidizing agent that has previously been studied in metalloporphyrin-catalyzed oxidations [28]. It suffers from some disadvantages: buffering is needed due to its acidity and some time bleaches the metal catalysts during oxidation reactions. When sodium periodate was used as the oxygen source, higher epoxide yield and lower by-products were observed.

Table 2
Effect of solvent on the epoxidation of cyclooctene with Mn(salen)/$NaIO_4$

Row	Solvent	Yield (%)[a] after 15 min
1	CH_3CN/H_2O	98
2	CH_3COCH_3/H_2O	70
3	CH_2Cl_2/H_2O	54
4	CH_3OH/H_2O	46
5	CH_3CH_2OH/H_2O	42
6	$CHCl_3/H_2O$	37
7	CCl_4/H_2O	5

[a] GLC yields based on the starting cyclooctene.

2.2. Effect of solvent on the oxidation of cyclooctene with sodium periodate catalyzed by Mn(salen)OAc

Solubility of manganese(III) Schiff base complexes strongly depends on solvent polarity. Mn(salen)OAc containing phosphine groups is very soluble in aqueous solutions. Among the 2:1 mixture of methanol, ethanol, acetone, acetonitrile (single phase systems), chloroform, dichloromethane and carbon tetrachloride (two phase systems, in which the triphenylphosphonium groups lead to solubility of complex in aqueous and organic solvents and can act as phase transfer catalyst) with water, the 2:1 acetonitrile/water mixture was chosen as the reaction medium, because the complex was highly soluble in this solvent and higher epoxide yield was observed. The results are shown in Table 2.

2.3. The effect of the metal center on the oxidation of cyclooctene

We have investigated the influence of the metal center on the activity of catalysts. Oxidation reactions were carried out with metallosalens having the same ligand but different metals: Mn, Fe, Co and Ni. The most active catalyst was manganese complex which showed higher activity in the epoxidation of cyclooctene.

2.4. The effect of various axial ligands on the oxidation of cyclooctene

The reactivity of salen catalysts in epoxidation reactions cannot only be tuned by substitution of the salen, but also

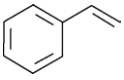
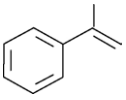
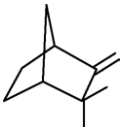
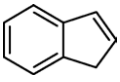
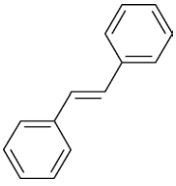
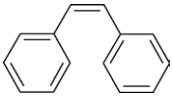
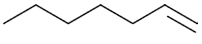
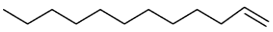
Table 3
The effect of different axial ligands in the epoxidation of cyclooctene by Mn(salen)OAc/$NaIO_4$[a]

Row	Axial ligand	Epoxide yield (%)[b] after 15 min
1	None	12.5
2	Triethylamine	34
3	Diethylamine	37
4	Piperidine	48
5	Pyridine	35
6	4-Cyanopyridine	22
7	2-Methypyridine	43
8	4-Methypyridine	52
9	4-*tert*-Butylpyridine	78
10	Imidazole	98
11	4(5)-Methylimidazole	94
12	2-Ethylimidazole	85
13	Benzimidazole	96
14	2-Methylimidazole	91
15	Pyrazine	72
16	Quinalidine	31
17	Morpholine	38
18	Triphenylphosphine	32
19	DMF	21

[a] Cyclooctene (0.5 mmol), $NaIO_4$ (1 mmol), axial ligand (0.22 mmol) and catalyst (0.052 mmol) in CH_3CN (5 mL)/H_2O (2.5 mL), at room temperature.
[b] GLC yields based on the starting cyclooctene.

by adding donor ligands to the reaction mixture [29,30] to mimic the effect of the axially coordinating histidine and thiolate residue found in peroxidase enzymes and cytochrome P-450, respectively [31]. For better understanding the role of axial ligands in activating water-soluble Mn(salen)OAc catalyst, we investigated the effect of different axial ligands upon the epoxidation of cyclooctene. Pure σ-donor amines, with very large pK_a values, were relatively poor co-catalysts in the epoxidation of cyclooctene (Table 3). Pyridine and methyl-substituted pyridines, with weak π-donating ability and pK_a values which are much smaller than those of σ-donor amines, generally show co-catalytic activities similar to those of amines. The observed order of co-catalytic activities, which is 4-*tert*-butylpyridine > pyridine ≫ 4-cyanopyridine, seems to be directly related to both the σ- and π-donating abilities of these nitrogen donors. Electron-withdrawing substituents such as CN^-, essentially displays no co-catalytic activity. However, substituted pyridines, having electron-releasing methyl group such as 4-*tert*-butylpyridine, showed 78% conversion in the oxidation of cycloocetene, which is higher than the unsubstituted pyridine. Addition of Ph_3P and DMF as donor ligands have no significant change in the epox-

Table 4
Epoxidation of alkenes with $NaIO_4$ catalyzed by Mn(salen)OAc in the presence of imidazole at room temperature

Entry	Alkene	Conversion (%)[a]	Epoxide yield (%)[a]	Time (min)
1		98	98	10
2		100	100	10
3		95	93[b]	10
4		100	100	10
5		97	97	10
6		95	95	10
7		91	91 (*trans*-epoxide)[c]	20
8		78	40 (*cis*-epoxide)[c], 38 (*trans*-epoxide)[c]	20
9		70	70	15
10		54	54	15

[a] GLC yield based on starting alkene.
[b] The by-product is 2% acetophenone.
[c] Both ^1H NMR and GLC data approved the reported yields.

ide yields. Among the nitrogen bases, which are used as axial ligands, imidazole exhibited the highest activity toward epoxidation of cyclooctene with sodium periodate. Strong π-donors ImH and its derivatives are the best co-catalysts among the nitrogen donors listed in Table 3. The lower co-catalytic activity of 2-MeImH and 2-EtImH or 4(5)-MeImH are due to the steric hinfrance of 2-substituent. Bulky and flat BzImH displays similar co-catalytic activity to imidazole.

Strong coordination of imidazole should result in an increase in electron density on metal and a facile cleavage of O–IO_3 bond in $NaIO_4$. In the absence of any axial ligand, only 12.5% of cyclooctene oxide was observed.

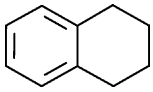
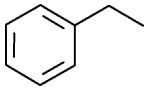
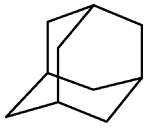
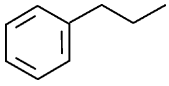
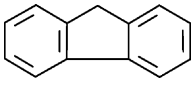
2.5. Catalytic alkene epoxidation by water-soluble Mn(salen)OAc

Reactions were performed at room temperature under air in CH_3CN/H_2O containing the alkene, oxidant, imidazole and Mn(salen)OAc in 1:2:0.22:0.104 molar ratio, respectively. This catalytic system led to the epoxidation of various alkenes (Table 4) in high yields (54–100%). Epoxidation of *trans*-stilbene proceeded in a stereospecific manner with complete retention of configuration. In contrast epoxidation of *cis*-stilbene was associated with some loss of stereochemistry and affords 40% *cis*-stilbene and 38% *trans*-stilbene oxides, respectively. Apparently, formation of the thermodynamically more stable *trans*-stilbene oxide requires a free rotation about the alkene C–C bond at some intermediate steps. Such a rotation is expected to be more feasible when catalysts with less steric strain are used [32]. The linear, cyclic and phenyl-substituted olefins were used as substrates in this system. The results are shown in Table 4. Electron-rich cyclic olefins are more reactive than the electron-poor terminal olefins. This reflects the electrophilic nature of oxygen transfer from manganese-oxo intermediate to the olefinic double bond. The epoxidizing systems were optimized using cyclooctene as the standard substrate. This alkene was selected as it is reactive towards metalloporphyrin-based epoxidizing agents, is not prone to allylic oxidation [33] and has been widely used in previous epoxidation studies with both homogeneous and heterogeneous catalysts.

3. Oxidation of alkanes with $NaIO_4$ catalyzed by Mn(salen)OAc

Direct oxidation of hydrocarbons is also one of the typical reactions of cytochrome P-450 [2]. The catalytic oxidation of alkanes with oxygen sources under mild conditions is especially a rewarding goal, since direct functionalization of unactivated C–H bonds in saturated hydrocarbons usually requires drastic conditions such as high pressure and high temperature. As shown in Table 5, we have found that water-soluble Mn(salen)OAc system is an efficient catalyst for the biomimetic oxidation of saturated hydrocarbons.

Table 5
Hydroxylation of alkanes with $NaIO_4$ catalyzed by Mn(salen)OAc in the presence of imidazole at room temperature

Entry	Alkane	Conversion (%)	Ketone (%)[a]	Alcohol (%)[a]	Time (min)
1		65	35	30	40
2		64	56	8	40
3		85[b]	85	–	40
4		32[c]	32	–	40
5		74	10	64	40
6		31[d]	31	–	40
7		58	58	–	40

[a] GLC yield based on starting alkane.
[b] Only α-position was oxidized.
[c] The product is acetophenone.
[d] The product is ethylphenyl ketone.

Cyclooctane, cyclohexane, 1,2,3,4-tetrahydronaphthalene and adamantane were converted in high yields to their corresponding alcohols and ketones. In contrast, ethylbenzene, propylbenzene, and fluorene only produced the corresponding ketones. In the case of adamantane, the only 1-adamantanol and 2-adamantanone were produced in the, reaction media as reported previously [34].

The effect of molecular oxygen was investigated in the oxidation of cyclooctane. The results showed that molecular oxygen was less efficient in oxidizing the cyclooctane and the conversion is negligible (2%). On the other hand, the oxidation of alkanes under argon atmosphere confirmed the obtained data.

This phosphonium substituted catalyst showed higher activity than unsubstituted Mn(III)salen complex in the oxidation of cyclooctene and cyclooctane. Under the same reaction conditions, the oxidation of cyclooctene in the presence of unsubstituted Mn(III)salen gave only 56% cyclooctene oxide. Cyclooctane oxidation with sodium periodate in the presence of unsubstituted catalyst produced 13% cyclooctanol and 3% cyclooctanone.

4. Oxo-manganese(V) species as the reactive intermediate

Although we have assumed the active manganese species to be reactive (salen)Mn(V) oxo intermediate, [(salen)Mn^{v} = O], by comparing the present spectral observation with the previous reports [35], we could not isolate this active species. It is pertinent to point out that to date no (salen)Mn(V) oxo species have yielded to structural characterization, although Groves et al. have characterize oxo manganese(V) porphyrin complexes in recent years [36].

When a clear brown solution of Mn(III)salen in acetonitrile was treated with sodium periodate, it immediately was turned dark. The appearance of a new absorption band (around 470 nm, Fig. 1) is strongly reminiscent of the spectral change obtained during the conversion of the Mn(III)salen cation to the corresponding oxo-manganese(V) species. Upon standing, the dark brown solution faded to the original light brown within 15 min. When the same experiment was carried out in the presence of cyclooctene the dark brown solution immediately changed to its original light brown color.

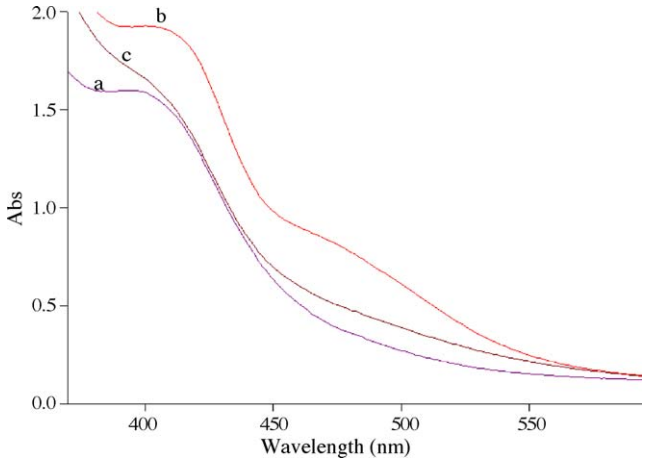
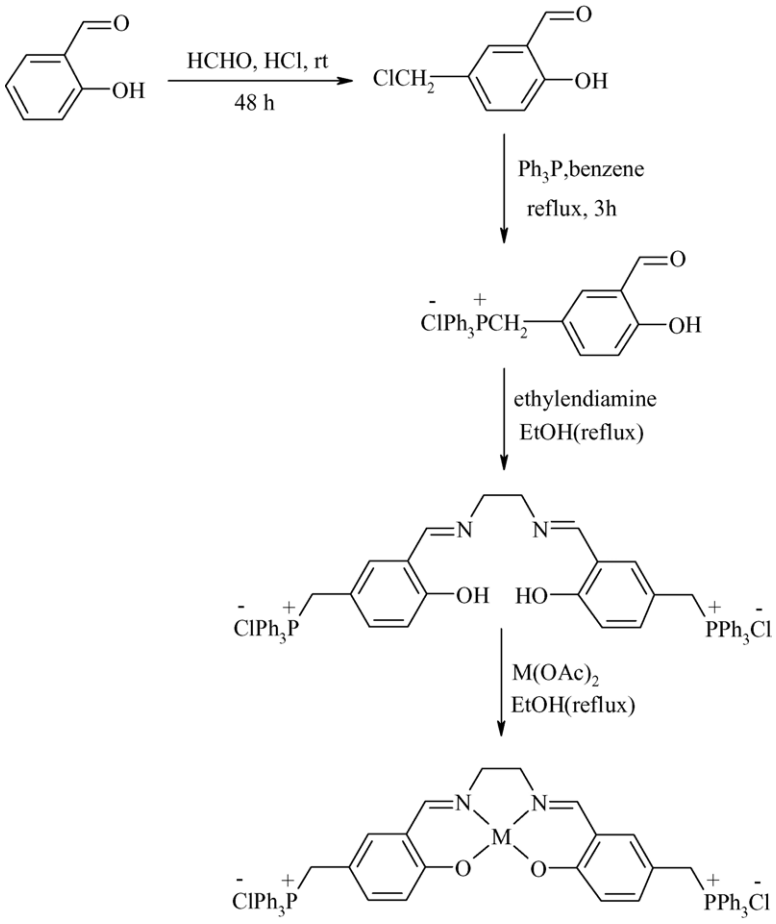


Fig. 1. (a) Absorption spectrum of Mn(salen)OAc in CH_3CN (2.0×10^{-3} M) at 25 °C; (b) absorption spectrum of Mn(salen)OAc (2.0×10^{-3} M) at 25 °C in the presence of excess $NaIO_4$; (c) absorption spectrum after addition of cyclooctene.



Scheme 1. Complexes a–d used as catalysts. M = Mn, Co, Ni, Fe.

5. Experimental

The Schiff base ligand was prepared by the standard procedure of refluxing ethanolic solutions of the corresponding ethylenediamine and salicylaldehyde derivative in a 1:2 molar ratio. A four-step synthetic strategy was followed for the synthesis of catalysts a–d (Scheme 1) [37,38]. Synthesis and characterization of these complexes have been reported in the literature; therefore, their purity was only checked by ^{1}H NMR and infrared spectroscopy.

5.1. Physical and spectral data for catalyst

Complex mp (130–133 °C), ligand mp (191–193 °C); δ_H ($CDCl_3$) 0.86 (3H, t, EtOH), 1.08 (3H, t, EtOH), 2.02 (3H, t, EtOAc), 2.45 (4H, brs, H_2O), 3.34 (2H, q, EtOH), 3.78 (4H, s, CH_2N), 4.09 (2H, q, EtOAc), 5.31 (4H, d, CH_2P), 6.59 (2H, d, Ar), 6.9 (2H, d, Ar), 7.13 (1H, s, Ar), 7.59–7.78 (30H, m, Ph), 8.09 (2H, s, CH = N); FT-IR, ν (cm^{-1}) 339 (s), 2919 (m), 2851 (m), 1622 (vs), 1543 (s), 1434 (s), 1300 (s), 1165 (m), 1112 (s), 998 (m), 823 (s), 689 (s), 514 (s); λ_{max} ligand (nm) (MeOH): 323, 260 (infl), 250 (infl); λ_{max} complex (nm) (MeOH): 415 (sh), 341 (sh), 275 (sh), 245.

The alkenes were purchased from Merck or Fluka and passed through a column of neutral alumina immediately prior to use. Gas chromatography experiments (GC) were performed with a Shimadzu GC-16A instrument using a 2m column packed with silicon DC-200 or carbowax 20M. The electronic absorption spectra were recorded on a Varian Cary NIR. ^{1}H NMR were recorded on a Bruker AQS 300 MHz. FT IR was recorded on a Bomen–Hartmann spectrometer and was obtained as potassium bromide pellets in range 400–4000 cm^{-1}.

5.2. Catalysis experiments

The reaction were carried out in CH_3CN/H_2O, at room temperature with constant stirring and the composition of the reaction medium was 0.5 mmol of alkene or alkane, 0.052 mmol of Mn(III)salen complex as catalyst, 1 mmol of sodium periodate in H_2O (2.5 mL) and imidazole (0.22 mmol) in CH_3CN (5 mL).

Progress of the reactions was monitored by GLC. After the reaction was completed, the reaction products were extracted with CH_2Cl_2 and were purified by a silica gel plate or a silica gel column. The identities of the products were confirmed by IR and ^{1}H NMR spectral data. Blank experiment under the same conditions in the absence of catalyst or in the absence of oxidant were also performed.

6. Conclusions

We have demonstrated for the first time, the effectiveness of a water-soluble cationic manganese(III)salen complex as catalyst in olefin epoxidation and alkane hydroxylation by sodium periodate. On the basis of these preliminary results, a catalyst based on manganese(III)salen complex exhibiting high activity has been developed. The presence of a nitrogen base is always necessary to obtain efficient catalytic systems. Imidazole as an axial ligand with π-donor capability is much more effective co-catalysts than other nitrogen donors. Other applications of this new catalytic system and the heterogenization of this water-soluble manganese(III)salen complex by supporting it on various supports is now in progress in our laboratory.

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