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Oxidation of hydrocarbons with iodobenzene diacetate catalyzed by manganese(III) porphyrins in a room temperature ionic liquid

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Abstract

Efficient methods for oxidation of alkanes and alkenes were obtained by using two manganese(III) porphyrin catalysts in combination with iodobenzene diacetate in a room temperature ionic liquid [bmim]PF₆ (1-*n*-butyl-3-methylimidazolium hexafluorophosphate). The effects of various organic solvents on the reactions and the role of axial ligands were examined. The recyclability of the catalyst remained in the oily ionic liquid phase was also investigated. A high-valent manganese-oxo porphyrin complex ($Mn^V = O$) was considered as a reactive oxidation intermediate according to investigation by stopped-flow rapid spectroscopy.

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1. Introduction

Salts that are fluid at room temperature are exciting and interesting as environment friendly solvents for a range of chemical processes. Ambient temperature ionic liquids typically consist of a heterocyclic cation based on substituted imidazole or pyridine and an inorganic anion such as $[AlCl_4]^-$, $[BF_4]^-$ or $[PF_6]^-$. Ionic liquids of this type have many benefits: (1) they can dissolve an enormous range of inorganic, organic and polymeric materials at very high concentrations; (2) they are non-corrosive; (3) they have low viscosities and no significant vapour pressures [1-3]. Our research group is interested in the utility of ionic liquids for transition metal-catalyzed oxygen atom transfers. We have shown previously that the ionic liquid $[bmim]PF_6$ can be used with iodobenzene diacetate (IBDA) and the catalyst meso-tetrakis(pentafluorophenyl) porphinato manganese(III) chloride (MnTFPPCl) to effect the epoxidation of olefins [4]. The yields of epoxide are comparable to the yields that are obtained in molecular solvents, and there seems to be a rate-enhancement compared to reactions in dichloromethane.

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+86-931-8276531; ac cn (C -G Xia) zene diacetate (PhI(OAc)₂) at phenyl)porphine (TPPS₄H₂) y MnTPPCl, MnTMOPPCl, M MnTPPS₄ were prepared act ously described [5–7] The jon

But up to now, the catalytic oxidation of alkanes by metalloporphyrins in ionic liquids had not been reported. In order to gain an insight into the catalytic selectivity of metalloporphyrins for the activation of various inert hydrocarbon bonds, we studied the catalytic activity of two manganese porphyrins including the simplest MnTPPCl as well as water-soluble MnTPPS₄ for the oxidation of alkanes and alkenes with PhI(OAc)₂.

2. Experimental

2.1. Materials and equipment

Dichloromethane (DCM), acetonitrile (ACN) and 1,2-dichloroethane (DCE) were dried and stored in 4 Å molecular sieves. Hexafluorophosphoric acid (60 wt.% in water), 5,10,15,20-tetrakis(pentafluorophenyl) - 21*H*, 23*H*-porphine (TFPPH₂), adamantane and 1-methylimidazole were obtained from Acros. Cyclooctane, 5,10,15,20-tetrakis(4-methoxyphenyl)-21*H*,23*H*-porphine (TMOPPH₂), iodobenzene diacetate (PhI(OAc)₂) and *meso*-tetrakis(*p*-sulfonatophenyl)porphine (TPPS₄H₂) were purchased from Fluka. MnTPPCl, MnTMOPPCl, MnTFPPCl, MnTMPyP and MnTPPS₄ were prepared according to procedure previously described [5–7]. The ionic liquid [bmim]PF₆ (IL) was

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prepared and purified as previously reported [8]. All alkenes were purified to use by passing through a column of basic alumina and then purities were checked by gas chromatography. All other reagents used were of AR grades of purity.

UV-Vis spectra were obtained with a Hewlett-Packard 8453 spectrophotometer. Time-resolved visible spectra were recorded on an Applied Photophysics DX-17MV Stopped-flow spectrophotometer. The structures of the reaction products were determined and analyzed using a HP 5890 II gas chromatogragh and an Agilent 6890/5973 GC/MS.

2.2. Oxidation reactions

A typical procedure for the oxidation of hydrocarbons using manganese porphyrin as catalyst in ionic liquid [bmim]PF₆: the catalyst (2 μ mol) was dissolved in [bmim]PF₆ (1.5 ml). To this solution, substrate (300 μ mol) in corresponding organic solvent (1 ml) and oxidant (450 μ mol) were added. The resulting solution was stirred at room temperature for 2 h. Progress of the reaction was monitored by GC.

2.3. Stopped-flow rapid spectroscopy

In one of two equal volume syringes was placed a solution of 2.4×10^{-5} M catalyst in mixed solvent [bmim]PF₆-CH₂Cl₂ (3/2, v/v). The second syringe contained 4.8 mM PhI(OAc)₂ in the same solvent. At the termination of mixing a series of spectra were taken by triggering the Applied Photophysics instrument.

3. Results and discussion

3.1. Comparison of different manganese porphyrins

We initially set out to investigate the catalytic ability of five types of manganese porphyrins in a mixture of ionic liquid and dichloromethane using tetralin as a model substrate (Table 1). Catalytic systems consisting of MnTPPCl,

Table 1

Oxidation	of tetralin	with Pl	nI(OAc) ₂	by	different	manganese	porphyrins
in [bmim]	PF6-CH2C	l ₂ mixe	d solvent	a			

Entry	Catalyst	Conversion	^b Yield ^b (%	ol/one	
		(%)	Alcohol	Ketone	
1	MnTPPCl	76 (34)	<1 (3)	75 (27)	< 0.01 (0.1)
2	MnTMOPPCl	62	27	31	0.9
3	MnTFPPCl	96	1	95	0.01
4	MnTMPyP	48	<1	47	0.02
5	MnTPPS ₄	71 (30)	6 (<1)	64 (29)	0.1 (0.02)

^a Conditions: molar ratio IBDA:alkane:catalyst = 225:150:1; 20° C; 2 h; [bmim]PF₆-CH₂Cl₂ (3/2, v/v); solvent volume: 2.5 ml.

^b Conversion and yield based on starting alkane. Results obtained in the absence of ionic liquid are given in parentheses.

MnTMOPPCl, MnTFPPCl, MnTPPS₄, MnTMPyP and PhI(OAc)₂ exhibit different activities. It is very clear that the order of catalytic activity for the manganese porphyrins tested is MnTFPPCl > MnTPPCl \approx MnTPPS₄ > MnT-MOPPCl > MnTMPyP under our experimental conditions. One could see from the results that in the mixture solvent ketones were obtained as major products with moderate to excellent substrate conversion except for the reaction catalyzed by MnTMOPPCl, in which the ratio of alcohol/ketone was equal. The data in Table 1 indicate that even the most simple MnTPPCl showed high catalytic activity in the presence of $[bmim]PF_6$ when compared to the same reaction performed in dichloromethane (Table 1, entry 1). This result suggests the higher performance of the ionic liquid as reaction medium. However, when the reaction was performed in pure ionic liquid phase, poor yields of products were obtained due to the high viscosity of the ionic liquid and weak solubility of IBDA. MnTMPyP and MnTPPS₄ are water-soluble manganese porphyrins that are widely used as catalysts in the oxidation of hydrocarbons [9-11]. A comparison between MnTMPyP and MnTPPS₄ showed that the anionic porphyrin MnTPPS₄ was a better catalyst than MnTMPyP (Table 1, entries 4 and 5).

3.2. The influence of various organic solvent on the oxidation of alkanes

In spite of the preliminary results in Table 1 which showed MnTFPPCl was the most active catalyst among the manganese porphyrins tested, we decided to examine the oxidation of various alkanes in [bmim]PF₆ containing different organic solvent catalyzed by MnTPPCl, MnTPPS₄ and IBDA systems. The reason of this attempt is originated by the fact that MnTPPCl is a simple, easy to obtain and cheap catalyst. MnTPPS₄ showed a comparative efficacy to MnTPPCl under identical condition.

Representative results of MnTPPCl and MnTPPS₄ catalyzed oxidation of alkanes with IBDA in different solvent systems are summarized in Table 2. In pure organic solvent, decompose of the catalyst was inevitable resulting lower oxidation yields. So the experiments were operated in mixed solvent. Oxidation of less reactive cyclohexane was not favoured in IL-DCM solvent system catalyzed by MnTP-PCl and MnTPPS₄ (Table 2, entries 1 and 4). However, when the reaction was performed in IL-DCE and IL-ACN, yields of alcohol 25 and 32% and alcohol/ketone ratios of 2.3 and 3.2, respectively were obtained with MnTPPCl as catalyst. In the case of MnTPPS₄, comparing with MnTPPCl similar conversions of cyclohexane were obtained with 27 and 10% yields of cyclohexanol and cyclohexanone in IL-DCE, respectively, and 24 and 19% yields of cyclohexanol and cyclohexanone in IL-ACN, respectively. The difference of alcohol/ketone ratio between MnTPPCl and MnTPPS4 was not very large. In the case of cyclooctane oxidation, these two manganese porphyrins exhibited similar activity with 29-46% cyclooctane conversion.

Table 2								
The influence of various	s organic solv	vents in the or	xidation of	alkanes	catalyzed b	y manganese	porphyrin-IBDA :	system ^a

Entry	Catalyst	Substrate	Solvent	Conversion ^b (%)	Yield ^b (%)		ol/one
					ol	one	
1	MnTPPCl	Cyclohexane	IL-DCM	_	_	_	
2		·	IL-DCE	36	25	11	2.3
3			IL-ACN	42	32	10	3.2
4	MnTPPS ₄		IL-DCM	_	_	_	
5			IL-DCE	37	27	10	2.7
6			IL-ACN	43	24	19	1.3
7	MnTPPCl	Cyclooctane	IL-DCM	46	29	17	1.7
8		·	IL-DCE	37	24	13	1.8
9			IL-ACN	42	25	17	1.5
10	MnTPPS ₄		IL-DCM	29	23	6	3.8
11			IL-DCE	45	27	17	1.6
12			IL-ACN	46	24	22	1.1
13	MnTPPCl	Adamantane	IL-DCM	33	32(1-ol); 1(2-ol)	_	33
14			IL-DCE	50	42(1-ol); 7(2-ol)	<1	49
15			IL-ACN	22	19(1-ol); 3(2-ol)	-	22
16	MnTPPS ₄		IL-DCM	54	52(1-ol); 2(2-ol)	_	54
17			IL-DCE	66	55(1-ol); 10(2-ol)	<1	65
18			IL-ACN	35	29(1-ol); 6(2-ol)	-	35
19	MnTPPCl	Tetralin	IL-DCM	76	<1	75	0.01
20			IL-DCE	72	31	40	0.8
21			IL-ACN	73	27	35	0.8
22	MnTPPS ₄		IL-DCM	71	6	64	0.1
23			IL-DCE	88	29	57	0.5
24			IL-CAN	87	15	71	0.2

^a Conditions as Table 1.

^b Conversion and yield based on starting alkane.

The results presented above demonstrate unambiguously that MnTPPCl and MnTPPS₄ are not very efficient catalyst for the oxidation of cycloalkanes. In the hydroxylation of bulky adamantane by IBDA, both manganese porphyrin catalysts gave 1-adamantanol (1-ol) and 2-adamantanol (2-ol) as only products (Table 2, entries 13–18). Concerning selectivity, MnTPPCl and MnTPPS₄ gave a higher 1-ol/2-ol ratio in IL-DCM than in IL-DCE and IL-ACN, showing a preferable oxidation at the tertiary C–H bond. So these results suggest a free radical activation of the C–H bonds of adamantane, as is expected for a P-450 mode.

As can be seen from these results, both of the two catalyst systems showed higher efficacy for oxidation of tetralin than cycloalkanes. They effected conversions of 71–88% of tetralin to 1-tetralol and 1-tetralone. Different from other alkanes, the selectivity of 1-tetralone was higher than that of 1-tetralol. Especially when the reaction was performed in the solvent of IL-DCM, tetralin afforded significant quantities of 1-tetralone in 75% (with MnTPPCl as catalyst) and 64% (with MnTPPS₄ as catalyst) yields, respectively.

In homogeneous catalytic reaction the polarity of the solvents usually play an important role. We suspect that the presence of the ionic liquid may regulate the polarity of the organic solvent thereby affecting the oxidation of alkanes.

3.3. The effect of axial ligand on the catalytic system

Since the catalytic ability of manganese porphyrins can be improved by the use of nitrogen bases as co-catalysts [12,13], the effects of two commonly used axial ligands such as imidazole and pyridine upon hydroxylation of alkanes were investigated (Tables 3 and 4). As can be seen from the data presented in Tables 3 and 4, the effect of axial ligands on the catalytic system was quite different, concerning the alkanes structure and the solvent system.

As mentioned above (Table 2, entries 1 and 4), the manganese porphyrin-IBDA systems were almost unable to oxidize cyclohexane in IL-DCM mixture. Interestingly, in this solvent mixture, fairly good yields were obtained for instance by adding of imidazole or pyridine with excellent cyclohexanone selectivity (Table 3, entries 2 and 3). Compared with MnTPPCl, under the same conditions the addition of axial ligands could not improve the catalytic activity of MnTPPS₄ in cyclohexane oxidation effectively (Table 4, entries 2 and 3). In IL-DCE and IL-ACN solvent mixtures, however, addition of imidazole and pyridine caused decrease dramatically in yields of products catalyzed by both manganese porphyrins (Tables 3 and 4, entries 4–9). In the case of cyclooctane hydroxylation for both catalyst systems, the

Table 3
The effect of axial ligand on hydroxylation of different alkanes catalyzed by MnTPPC1 with IBDA in various solvent systems

Entry	Substrate	Axial ligand	Solvent	Alcohol + ketone yield ^b (%)	Alcohol/ketone
1	Cyclohexane	No	IL-DCM	_	_
2		Imidazole		54	< 0.01
3		Pyridine		40	0.1
4		No	IL-DCE	36	2.3
5		Imidazole		17	3.3
6		Pyridine		26	3.3
7		No	IL-CAN	42	3.2
8		Imidazole		28	1.1
9		Pyridine		12	0.7
10	Cyclooctane	No	IL-DCM	46	1.7
11		Imidazole		38	1.9
12		Pyridine		48	0.6
13		No	IL-DCE	37	1.8
14		Imidazole		43	3.8
15		Pyridine		33	4.5
16		No	IL-ACN	42	1.5
17		Imidazole		31	1.8
18		Pyridine		42	2.2
19	Adamantane	No	IL-DCM	33	33
20		Imidazole		56	56
21		Pyridine		51	51
22		No	IL-DCE	50	50
23		Imidazole		21	21
24		Pyridine		16	16
25		No	IL-ACN	22	22
26		Imidazole		15	15
27		Pyridine		12	12
28	Tetralin	No	IL-DCM	76	< 0.01
29		Imidazole		90	0.3
30		Pyridine		70	1.3
31		No	IL-DCE	72	0.8
32		Imidazole		88	0.7
33		Pyridine		90	0.5
34		No	IL-CAN	73	0.8
35		Imidazole		83	0.4
36		Pyridine		90	0.3

^a Conditions: molar ratio IBDA:alkane:axial ligand:catalyst = 225:150:50:1; 20 °C; 2 h; [bmim]PF₆-organic solvent (3/2, v/v); solvent volume: 2.5 ml.

^b Conversion and yield based on starting alkane.

introduction of axial ligands showed different action. For MnTPPCl, pyridine had no effect on the yields of products. However the formation of alcohol was favoured in IL-DCE and IL-ACN (Table 3, entries 15 and 18).

On the contrary, the use of axial ligands dramatically improved the ability of MnTPPS₄ to catalyze cyclooctane hydroxylation (Table 4, entries 10–18). In all cases, hydroxylation of adamantane only provided adamantanol (Tables 3 and 4, entries 19–27, respectively). When the reaction was performed in IL-DCE and IL-ACN the product yields were lower by using axial ligands than that of no axial ligand (Tables 3 and 4, entries 22–27, respectively). And only in IL-DCM solvent system the yield of products was improved by using axial ligands catalyzed by MnTPPCI. Whereas the same reaction wascarried out in the presence of MnTPPS₄ under identical conditions, product yields werealmost unchanged (Table 4, entries 19–21). From the data in Tables 3 and 4, it seemed that tetralin was a more reactive substrate than other alkanes investigated. By comparison. in the solvent mixture of IL-DCE and IL-ACN MnTPPCl showed a higher tetralin oxidation activity with significant tetralin conversion of 72–90% associated with the axial ligands than that of MnTPPS₄. Moreover, the former gave a high 1-tetralone selectivity in the presence of both two axial ligands.

3.4. Epoxidation of several alkenes by MnTPPCl-IBDA and MnTPPS₄-IBDA in [bmim]PF₆-CH₂Cl₂

The catalytic activities of the MnTPPCI-IBDA and MnTPPS₄-IBDA catalytic system were also tested for epoxidation of alkenes using IL-DCM solvent system, Table 5. The results indicated that MnTPPS₄ was a more efficient and selective catalyst than MnTPPCI for epoxidation of alkenes.

Table 4
The effect of axial ligand on hydroxylation of different alkanes catalyzed by MnTPPS4 with IBDA in various solvent systems

Entry	Substrate	Axial ligand	Solvent	Alcohol + ketone yield $^{b}(\%)$	Alcohol/ketone
1	Cyclohexane	No	IL-DCM	_	_
2		Imidazole		16	1.7
3		Pyridine		5	2
4		No	IL-DCE	37	2.7
5		Imidazole		6	5
6		Pyridine		25	0.7
7		No	IL-CAN	43	1.3
8		Imidazole		14	2.5
9		Pyridine		26	0.7
10	Cyclooctane	No	IL-DCM	29	3.8
11		Imidazole		68	1.3
12		Pyridine		78	0.6
13		No	IL-DCE	45	1.6
14		Imidazole		46	1.9
15		Pyridine		66	0.9
16		No	IL-CAN	46	1.1
17		Imidazole		41	0.9
18		Pyridine		66	0.4
19	Adamantane	No	IL-DCM	54	54
20		Imidazole		45	45
21		Pyridine		55	55
22		No	IL-DCE	65	65
23		Imidazole		21	21
24		Pyridine		24	24
25		No	IL-ACN	35	35
26		Imidazole		23	23
27		Pyridine		11	11
28	Tetralin	No	IL-DCM	71	0.09
29		Imidazole		29	1.4
30		Pyridine		68	1.5
31		No	IL-DCE	88	0.5
32		Imidazole		52	2
33		Pyridine		52	2
34		No	IL-ACN	87	0.2
35		Imidazole		36	0.9
36		Pyridine		43	0.9

^a Conditions as Table 3.
^b Conversion and yield based on starting alkane.

Table 5					
Epoxidation of various	alkenes wit	h manganese	porphyrin-IBDA	systems in	IL-DCM ^a

Entry	Catalyst	Substrate	Conversion ^b (%)	Epoxide		
				Yield ^b (%)	Selectivity (%)	
1	MnTPP	Styrene	97	95	97	
2		α -Methyl-styrene	56	22	40	
3		Cyclooctene	37	37	100	
4		1-Decene	57	54	94	
5		1,2-Dihydronaphthalene	37	23	61	
6	MnTPPS ₄	Styrene	99	98	99	
7		α -Methyl-styrene	81	65	80	
8		Cyclooctene	99	99	100	
9		1-Decene	74	70	95	
10		1,2-Dihydronaphthalene	94	72	77	

^a Molar ratio PhI(OAc)₂:substrate:catalyst = 225:150:1; 20 °C; 2 h; [bmim]PF₆-CH₂Cl₂ (3/2, v/v); solvent volume: 2.5 ml. ^b Conversion and yield based on starting alkene.



Fig. 1. Catalytic activity of the recovered MnTPPS₄ for epoxidation of styrene and oxidation of tetralin in [bmim]PF₆-DCM (3/2,v/v), molar ratio PhI(OAc)₂:substrate:catalyst = 150:150:1; 20 °C; 2h.

3.5. Catalyst reuse and stability

The remaining reactants and the products are both easily removed from the reaction mixture via extraction with *n*-hexane, which is immiscible with the ionic liquid used in this work. The recyclability of the catalyst remained in the oily ionic liquid phase was investigated. Unfortunately for MnTPPCl a significant loss of activity was observed in the examination due to decomposition. However, MnTPPS₄ kept to its catalytic activity during the reaction.

Fig. 1 shows the catalytic activity of the reused MnTPPS₄ in epoxidation of styrene and hydroxylation of tetralin. One could see from the results that the recovered catalyst gave a comparable catalytic activity. In the case of styrene epoxidation, both catalytic activity and selectivity to epoxide were unchanged after five reuse. For tetralin, the reused catalyst showed the same efficiency (activity and selectivity) with that of fresh catalyst.

After the catalytic reaction, the UV-Vis spectrum of the ionic liquid containing catalyst showed the presence of $MnTPPS_4$ by the presence of unchanged Soret band at 472 nm, which indicates that the catalyst is stable during the reaction (Fig. 2).



Fig. 2. UV-Vis spectrum of the MnTPPS₄ in [bmim]PF₆-DCM.



Fig. 3. Difference spectra defined for the first 1 s of the reaction of $12 \,\mu$ M MnTPPS₄ with 2400 μ M IBDA in [bmim]PF₆-CH₂Cl₂ (3/2, v/v) at 20 °C.

3.6. Stopped-flow rapid spectroscopy of manganese porphyrin reacted with PhI(OAc)₂ in [bmim]PF₆-CH₂Cl₂

In the reaction of Mn(III) porphyrin with various oxidizing reagents, the nature of the active oxygen complex, which is able to transfer its oxygen atom to hydrocarbons, although not completely established, is generally considered to be a high-valent Mn-oxocomplex, at least formally, a $Mn^{V} = O$ species [14–16]. According to the above discussion, we assumed that in our case the active oxygen intermediates in these reactions are high-valent oxo-manganese species.

In order to confirm the proposed manganese-oxo reactive intermediate in these oxidation reactions, the reaction of MnTPPS₄ with PhI(OAc)₂ in mixed [bmim]PF₆-CH₂Cl₂ was analyzed by stopped-flow spectrophotometry at 20 °C. An intermediate with the characteristic Soret maximum at 424 nm could be observed in 1 s after mixing by difference spectroscopy with clear isosbestic points at 446 and 382 nm (Fig. 3). Then the Soret band of MnTPPS₄ at 468 nm was fully restored. The band at 424 nm has been reported in the literature and assigned to a Mn^V = O intermediate species [17–19].

4. Conclusions

In summary, we have shown that oxidation of various alkanes can be efficiently catalyzed by two manganese porphyrin in an ionic liquid-organic solvent mixture system. It is also shown that the efficiency and selectivity of the catalysts are sensitive to the nature of organic solvent and axial ligand. The catalyst system can efficiently epoxidize alkenes to corresponding epoxides. And the water-soluble MnTPPS₄ can be reused five times with the activity remaining. From the studies of stopped-flow spectra, we speculate that the active intermediate in the reaction is $Mn^{V} = O$ porphyrin.

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