



Short Communication

Efficient oxidation of polycyclic aromatic hydrocarbons with H₂O₂ catalyzed by 5,10,15-triarylcorrolatoiron (IV) chloride in ionic liquids

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ABSTRACT

A simple and efficient oxidative system has been developed for the oxidation of polycyclic aromatic hydrocarbons with H₂O₂ catalyzed by 5,10,15-triarylcorrolatoiron (IV) chloride in mixed reaction media using imidazolium ionic liquids and organic solvents. The coordination effects of different organic solvents as well as anions of ionic liquids on the oxidation reactions have been examined.

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1. Introduction

Polycyclic aromatic hydrocarbons (PAHs) belong to a class of carcinogenic and mutagenic pollutant [1]. The interaction of their specific oxidative metabolites with DNA is primarily responsible for these hazardous effects. The primary oxidative metabolites of PAHs are formed by cytochrome P450 monooxygenases, a membrane bound heme enzyme system [2]. The tedious isolation and fragility of natural cytochrome P450 enzyme system are the main limiting factors for the development of their use at large scale. In this regard, metalloporphyrins have been extensively used in various biomimetic reactions to understand the reaction mechanism and to isolate the metabolites in sufficient amount for further biological studies [3–5].

Recently, the metal complexes of corroles, the ring contracted porphyrins, have generated intense interest in catalysis and the major contribution in this new emerging field has been made by Z. Gross et al. [6]. However, the reports on oxidation of hydrocarbons catalyzed by iron (IV) corroles are sparse [7,8]. The complete bleaching of iron (IV) corrole at the end of the reactions, poor yields of products and the use of toxic oxidants limit their applications in catalysis. Hence, a catalytic system with mild oxidant operating at ambient condition is highly desirable. The use of H₂O₂ as a clean and mild oxidant in biomimetic oxidation processes is of particular interest because it leads to formation of only water as the side product formed by the dissociation of hydroperoxo complex (Fe^{IV}–O–OH) [9]. Ionic liquids (ILs) have been used as a novel reaction media for various organic syntheses and transformations [4,5]. The positive effect of ILs on oxidation processes using

iron and iron complexes has also been reported in literature [10,11]. Herein, we report the oxidation of 2-methyl naphthalene (**1**), naphthalene (**2**), anthracene (**3**) and benz [a] anthracene (**4**) with H₂O₂ catalyzed by 5,10,15-triarylcorrolatoiron (IV) chloride [TACFe(IV)Cl] (**17a–17e**) in ILs (Scheme 1, Fig. 1).

2. Experimental

2.1. Synthesis of 5,10,15-triarylcorrolatoiron (IV) chlorides/ bromide (**17a–17f**)

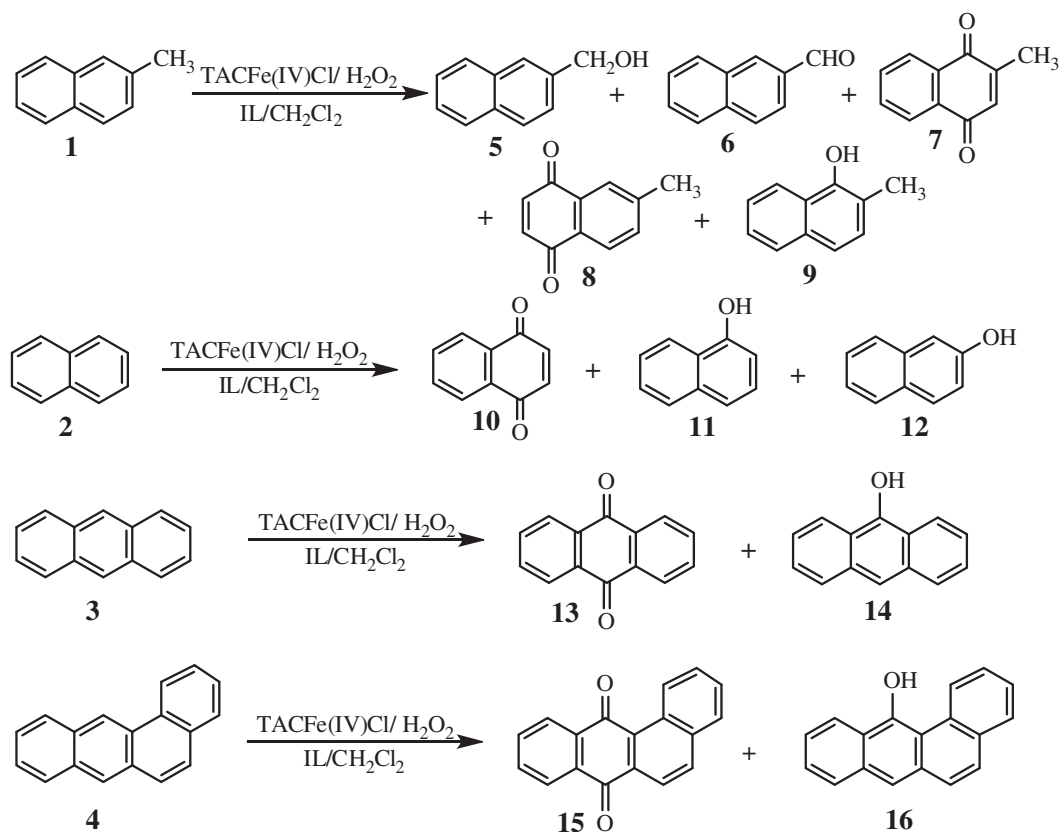
The 5,10,15-triarylcorrolatoiron (IV) chlorides (**17a–17e**) or 5,10,15-triarylcorrolatoiron (IV) bromide (**17f**) were prepared by following the literature method [12]. Further details and spectroscopic data are given in ESI.

2.2. Representative procedure for the oxidation of PAHs (**1–4**) with H₂O₂ catalyzed by iron (IV) corroles (**17a–17e**) in CH₂Cl₂/ IL (1:1, v/v)

To the solution of PAHs (**1–4**) (4.0 mmole) and iron (IV) corrole (**17a–17e**) (0.04 mmole) in CH₂Cl₂: IL (1:1, v/v) (24 mL), H₂O₂ (30% in water, 12.0 mmole) was added in small aliquots and the reaction mixture was stirred for 5 h at room temperature. The progress of the reaction was examined by the analysis of thin layer chromatography (TLC). The solvent was removed under reduced pressure and the reaction mixture was extracted with ethyl acetate. The catalyst (**17a–17e**) immobilized in IL layer was dried and reused for the oxidations of PAHs. The ethyl acetate layer was concentrated under reduced pressure and all the components were separated by preparative TLC. The yield of different oxidized products was analyzed by HPLC in methanol/ water

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Scheme 1. Oxidation of PAHs with H_2O_2 catalyzed by TACFeCl (**17a–17e**).

(1:1, v/v) as solvent, by comparing the retention time with authentic samples. All the oxidized products were characterized by 1H NMR, IR and MS spectroscopic techniques which gave the satisfactory results with the reported data.

3. Results and discussion

The oxidation of **1** with H_2O_2 in absence of catalyst **17** in CH_2Cl_2 did not yield any product even after 24 h. The same reaction in presence of **17a** gave **5** and **6** from side chain oxidation and **7** from aromatic ring oxidation (Table 1, entry 1). The use of [bmim][BF_4] as reaction medium showed higher catalytic activity of **17a** as compared to CH_2Cl_2 and the yields improved further when mixed solvent system [bmim][BF_4]/ CH_2Cl_2 (1:1, v/v) was used (Table 1, entries 2 and 3). This could be ascribed to the disruption of the supramolecular ionic aggregates for

imidazolium ionic liquids by organic solvent molecules which turn imidazolium cations “naked” making them available for the interaction with the catalyst [TACFe(IV)Cl] (**17**) [13,14]. The higher performance of the mixed solvent system than the pure organic solvent was also observed in acetonitrile (ACN) and methanol (Table 1, entries 4–7). The ease of oxidation of **1** was found to increase in the line of $CH_2Cl_2 < ACN < methanol$. This remarkable effect could be correlated to the polarity of the solvent which governs the interionic interaction energy of ILs and solvation dynamics [15,16].

To explore the effect of solvent on the activity of the catalyst, the UV–vis titrations of **17a** with methanol were carried out which exhibited slight red shift in the Soret band (Figs. 1S–2S). The 1H NMR spectrum of **17a** in $CDCl_3$ on addition of CH_3OH or in CD_3OD showed upfield shift in the resonances for β -pyrrolic protons (Figs. 3S–5S). These spectral changes suggested the dissociation of Fe–Cl bond in (TAC

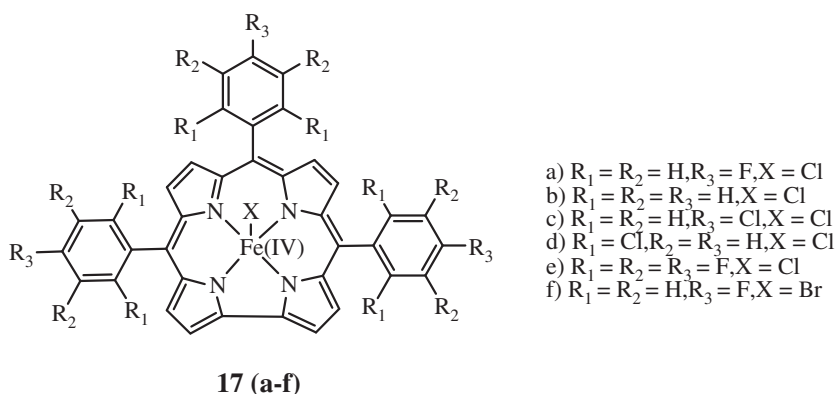


Fig. 1. Different catalysts (**17**) used in oxidation reactions.

Table 1
Oxidation of **1** with H₂O₂ catalyzed by **17a** in different solvents^a.

Entry	Solvent system	Conv. (%) ^b	Yields (%) ^c				
			5	6	7	8	9
1	CH ₂ Cl ₂	28	10	5	12	–	–
2	[bmim][BF ₄]	39	14	6	18	–	–
3	[bmim][BF ₄]/ CH ₂ Cl ₂	60	20	9	28	t	t
4	CH ₃ CN	62	19	11	28	2	t
5	[bmim][BF ₄]/ CH ₃ CN	71	22	12	28	5	2
6	CH ₃ OH	74	22	13	30	4	3
7	[bmim][BF ₄]/ CH ₃ OH	80	24	15	32	6	2
8	[bmim][Br]/ CH ₂ Cl ₂	44	19	9	12	–	t
9	[bmim][Cl]/ CH ₂ Cl ₂	33	11	6	14	–	–
10	[bmim][PF ₆]/ CH ₂ Cl ₂	65	21	10	33	3	–
11	[bmim][OAc]/ CH ₂ Cl ₂	46	19	10	14	–	t
12	[bmim][BF ₄]/ CH ₃ OH ^d	78	23	15	30	5	2

^a 1: H₂O₂: **17a** = 50:150:1.

^b Based on **1**.

^c Based on HPLC analysis.

^d Recovered catalyst was used, t = trace.

FeCl (**17**) and the stabilization of [(TAC)Fe]⁺ species by the coordination of methanol to give [(TAC)Fe(CH₃OH)]⁺ species. This is in accordance with the interaction of methanol with iron-porphyrins [17]. Similar spectral changes, though not so pronounced with ACN (Figs. 6S–9S), also suggested the generation of [(TAC)Fe(ACN)]⁺ species through –CN complexation [18]. The possibility of the formation of [(TAC)Fe^{III}(ACN)₂] species [7] by the auto reduction of **17** in presence of excess ACN was ruled out as the UV–vis spectrum of **17a** in CH₂Cl₂ on addition of ACN differs from that of **17a** in ACN (Figs. 6S–7S). The [(TAC)Fe(CH₃OH)]⁺ and [(TAC)Fe(ACN)]⁺ species are believed to be more catalytic active than (TAC)Fe^{IV}Cl (**17**) due to the axial ligand effect [19]. The catalyst **17a** regenerates after removal of methanol/ACN as indicated by the ¹H NMR spectrum of the recovered catalyst (Figs. 4S and 9S). The regenerated catalyst **17a** can be reused to catalyze the oxidation reactions (Table 1, entry 12).

The influence of anions of ILs on the oxidation of **1** was also examined. The change of IL from [bmim][BF₄] to [bmim][PF₆] improved the yields, whereas the use of [bmim][Cl], [bmim][Br] and [bmim][OAc] decreased the ease of oxidation (Table 1, entries 8–11). The UV–vis studies of **17a** with ILs exhibited a red shift in the Soret band with enhanced absorption intensity (Figs. 10S–13S). The coordination or π–π interaction between tetrapyrrolic unit and imidazolium part of ILs that is believed to play a vital role in the extraction process of cytochrome c, myoglobin, haemoglobin and horseradish peroxidase [20], is considered to cause only diminutive change in the spectra as there is little change in the spectra with [bmim][Cl] which has same cationic part as that of other ILs.

The role of ILs in the reaction could be correlated to the coordination ability of the anionic part. The yields of oxidative products increased in ILs in the order of Cl < Br < OAc < BF₄ < PF₆ which reflects the relative acidity of the C2–H caused by the coordination ability of the counter anions [21,22]. The interionic interaction energy of ILs also varies with the counter anions and is generally smaller for the

Table 2
Oxidation of **1** using different catalysts in [bmim][BF₄]/CH₂Cl₂^a.

Entry	System	Conv. (%) ^b	Yields (%) ^c				
			5	6	7	8	9
1	1/ 17b / H ₂ O ₂	45	16	7	20	–	–
2	1/ 17c / H ₂ O ₂	57	21	8	26	t	–
3	1/ TCPPFe(III)Cl/ H ₂ O ₂	50	18	7	22	t	2
4	1/ 17d / H ₂ O ₂	70	22	10	34	3	–
5	1/ 17e / H ₂ O ₂	82	25	12	37	5	2

^a 1: H₂O₂: catalyst = 50:150:1.

^b Based on substrate **1**.

^c Based on HPLC analysis, TCPPFe(III)Cl = 5,10,15,20-tetra(4-chlorophenyl)porphyrinatoiron(III) chloride.

Table 3
Oxidation of **2**, **3** and **4** with H₂O₂ using different catalyst (**17**) in [bmim][PF₆]/CH₂Cl₂^a.

Entry	Catalyst	Conv. (%) ^b	Yields (%) ^c			Conv. (%) ^b	Yields (%) ^c			Conv. (%) ^b	Yields (%) ^c	
			10	11	12		13	14	15		16	
1	17a	23	19	2	–	21	18	3	24	18	3	
2	17b	15	12	–	–	16	14	t	18	12	4	
3	17d	32	25	5	t	31	24	5	35	25	8	
4	17e	40	28	8	2	38	29	7	42	28	11	

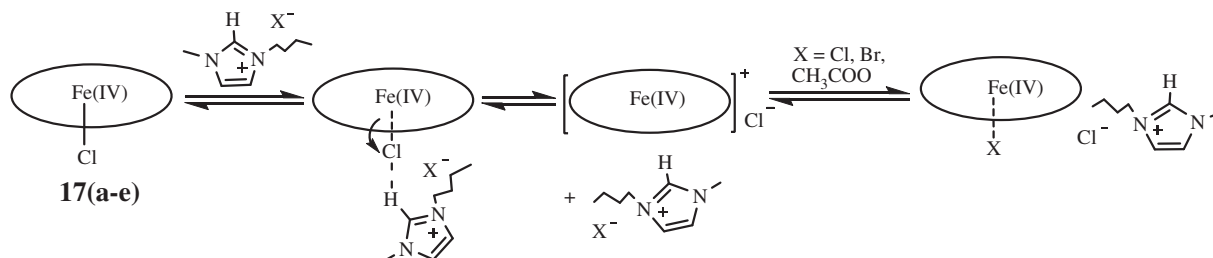
^a Substrate: H₂O₂: **17** = 50:150:1

^b Based on substrate **2**, **3** or **4**.

^c Based on HPLC analysis.

larger anion [23]. The weak nucleophilic counter anion such as PF₆ poorly coordinates and thereby C2–H preferably coordinates to axial ligand –Cl of the catalyst (Scheme 2). The downfield shift of C2–H signals in the ¹H NMR spectra of ILs in presence of **17a** explicates this interaction (Figs. 14S–17S). This H-bonding interaction could make the Fe–Cl bond in **17** weak leading to afford [(TAC)Fe]⁺ species. Furthermore, the low coordinative ability of the anions of ILs such as PF₆ makes them more available to stabilize [(TAC)Fe]⁺ intermediate [24], facilitating its formation and thereby promoting the attack of H₂O₂ on iron atom to generate reactive intermediate to give oxidative products in high yields.

The dissociation of Fe–Cl bond in **17** was clearly observed by NMR studies of **17** with different ILs which is followed by ligand exchange process in presence of strongly coordinating anions in ILs (Scheme 2). Addition of [bmim][Br] in the solution of **17d** shifts the resonances for β-pyrrolic protons and becomes equivalent to the values for 5,10,15-tris (2,6-dichlorophenyl) corrolatoiron (IV) bromide [25] (Fig. 18S). Similarly, the signals for β-pyrrolic protons of **17a** in presence of [bmim][Br] gradually changes and finally matches with the spectrum of **17f** (Figs. 19S–20S). The UV–vis spectral study of **17a** with [bmim][Br] is also comparable to the spectrum of **17f** (Figs. 10S, 21S), supporting the dissociation of Fe–Cl bond and the formation of Fe–Br bond. Although, the ligand field strength of Cl[–] is stronger than that of Br[–] [26], the much more abundance of Br[–] in ILs compared to the Cl[–] dissociated from the (TAC)FeCl (**17**) directs the system towards



Scheme 2. Interaction of ILs with catalysts (**17a**–**17e**).

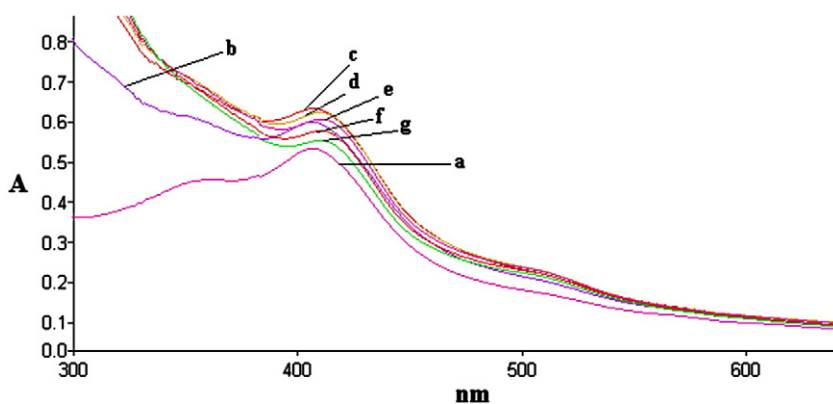
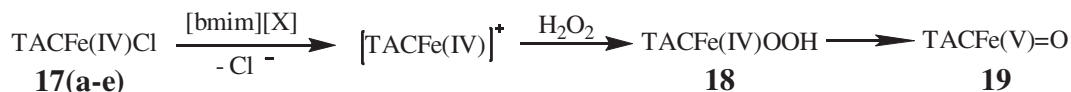


Fig. 2. UV-vis spectra of 17a (2.99×10^{-5} M) in CH₂Cl₂ (a), on addition of [bmim][PF₆] (50 μ l) (b), and H₂O₂ (10 μ l) (time interval = 2 min) (c–g).



Scheme 3. Stepwise formation of reactive intermediate.

ligand exchange process by preferential interaction of Br[−] with [(TAC)Fe]⁺. The replacement of axial ligand −Cl by chloramines-T in iron (IV) corrole [27] and −OAc by PF₆ in Mn (III) porphyrin [28] also supports the findings. However, the interaction between Br[−] and [(TAC)Fe]⁺ is relatively weak as the NMR spectra of 17a isolated from the reaction in [bmim][Br] showed mainly the presence of 17a with traces of 17f even after 24 h (Fig. 22S). The ligand exchange process was also observed with [bmim][OAc] (ESI, 23S). In titration with [bmim][Cl], no

change in the ¹H NMR spectrum of 17a was observed (Fig. 24S) which could be credited to the existence of same counter anion and axial ligand. The addition of non-coordinating [bmim][PF₆] in the solution of 17a also caused no identifiable changes in the ¹H NMR spectrum (Fig. 25S). This could be explained on the basis of high nucleophilicity of Cl[−] which forces the Cl[−] to be in proximity of [(TAC)Fe]⁺ produced by the dissociation of Fe–Cl bond of (TAC)FeCl (17). The titrations with [bmim][BF₄] leads to partial ligand exchange process in 17a when

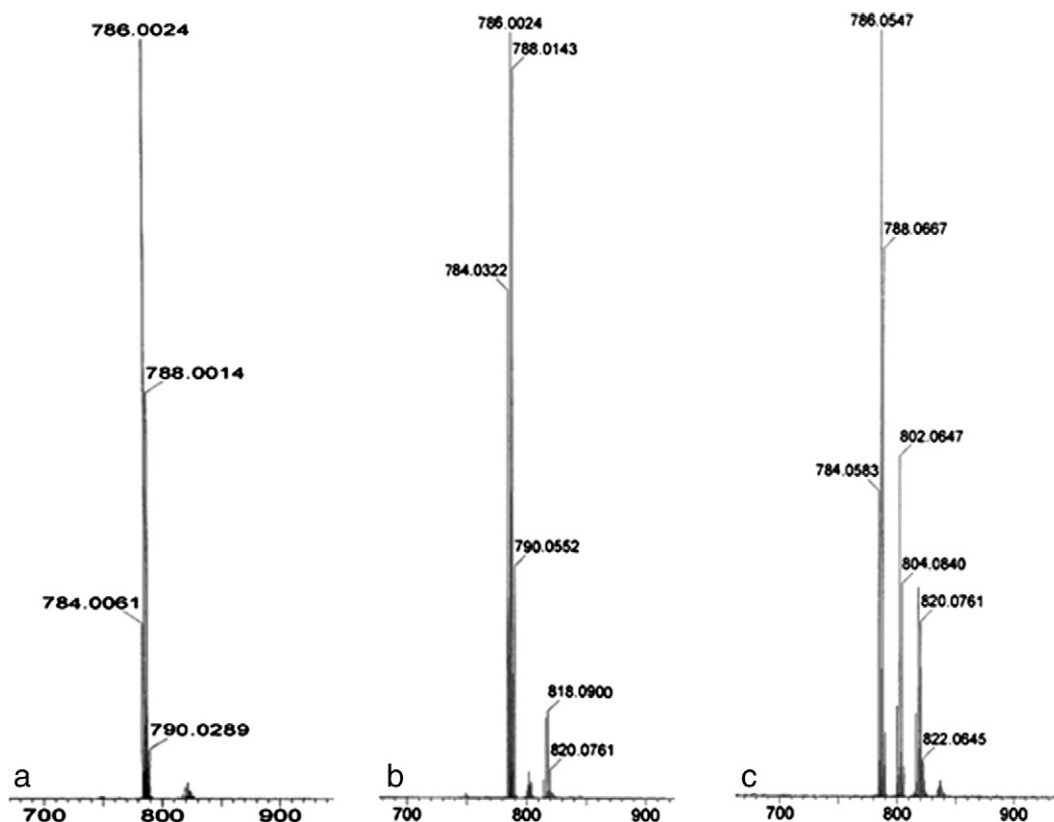
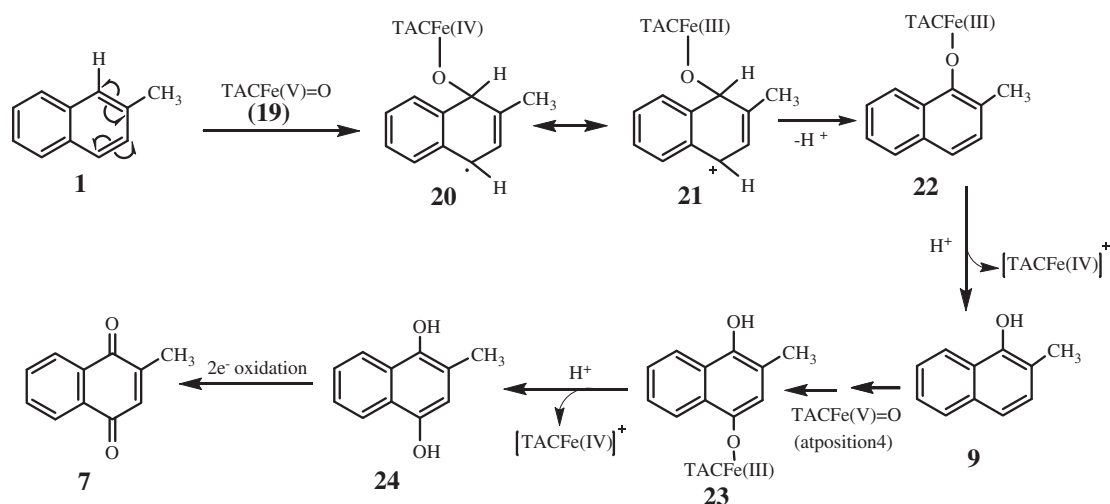


Fig. 3. ESI-MS spectrum of 17d (a), on addition of H₂O₂ (immediately) (b), after 5 min (c).



Scheme 4. Proposed mechanism of oxidation.

excess of BF_4^- is added (Fig. 26S). The broadening of the peaks of catalyst and ILs in ^1H NMR spectra can attribute to π - π interaction of ILs with catalyst.

The effect of nature of substituent present on the ring of **17** on the ease of oxidation of **1** was also examined (Table 2). The presence of electron withdrawing groups favoured the oxidation of **1** and 82% conversion of **1** was observed with **17e** catalyst. The reaction using **17f** gave poor yields of products (results not shown) and the decomposition of **17f** was also observed. The oxidation of **1** was also compared using **17c** and structurally similar analogue 5,10,15,20-tetra(4-chlorophenyl) porphyrinatoiron (III) chloride [TCPPFe(III)Cl] as catalyst under the experimental conditions (Table 2, entries 2–3). The results explicit the higher catalytic activity of **17c** than [TCPPFe(III)Cl] which may be ascribed to much larger rate of oxygen atom transfer from oxo-metal corroles to alkanes than that of analogous oxo-metal porphyrins [29]. The catalytic system **17**- H_2O_2 also worked well for the oxidation of **2**, **3** and **4** (Scheme 1, Table 3).

To investigate the generation of reactive intermediate in the reaction, time-dependent UV–vis studies of **17a** with H_2O_2 were performed. The addition of H_2O_2 in the solution of **17a** in CH_2Cl_2 and [bmim][PF₆] initially enhanced the absorption intensity of the Soret band (Fig. 2). This could be attributed to the attack of H_2O_2 at the iron centre of $(\text{TACFe})^+$ produced by the H-bonding effect of IL, to give hydroperoxy intermediate (**18**) (Scheme 3). Further, the decay of the Soret band with time indicates the homolytic cleavage of $-\text{OOH}$ bond to give transient oxo-iron (V) intermediate (**19**) [30]. The ESI-MS (+) spectra of **17d** on addition of H_2O_2 also exhibited peaks at m/z 785.89, 818.89 and 801.88 corresponding to $[\text{MH}^+ - \text{Cl}]$, $[(\text{MOOH})\text{H}^+ - \text{Cl}]$ and $[(\text{MO})\text{H}^+ - \text{Cl}]$ (Figs. 3 and 27S–29S), supporting the generation of hydroperoxy and iron-oxo corrole intermediates in the reactions. Recently, the formation of iron (V)oxo reactive intermediate in an iron-complex catalyzed oxidation process has been proposed on the basis of mass spectroscopic and cyclic voltametric data [31].

The oxidation of PAHs with H_2O_2 catalyzed by **17** in ILs is believed to proceed with the dissociation of Fe–Cl bond to give $(\text{TACFe})^+$ species which on reaction with H_2O_2 gives **18** and then **19** intermediate (Scheme 3). In presence of methanol and ACN, $(\text{TACFe})^+$ could be present in the form of $[(\text{TAC})\text{Fe}(\text{ACN})]^+$ and $[(\text{TAC})\text{Fe}(\text{CH}_3 - \text{OH})]^+$. The species **19** attacks at the aromatic ring of **1** at position 1 creating a new carbon-oxygen bond forming σ -adduct (**20**) (Scheme 4). The e^- -transfer leads to the carbocation **21** and then to **22** after elimination of the proton. The cleavage of Fe–O-aromatic bond in **22** gives rise to **9** and $(\text{TACFe})^+$ species regenerates [32]. The addition of a second iron-oxo species (**19**) at position 4 gives **23** in several steps.

The Fe–O bond present in **23** cleaves to afford **24** which is quickly oxidized to quinone (**7**). The rapid oxidation of **11** to **10** under the given reaction condition (results not shown) also justifies the reaction pathway. The oxidation of aromatic ring of other PAHs (**2**, **3** and **4**) and the side chain in **1** can also be explained on this basis. Similar mechanism has been proposed for the oxidation of PAHs using iron-porphyrins [33] and iron-phthalocyanines [34]. On going from neat IL to IL co-solvent mixtures, the enhancement in the electron transfer rate could be responsible for the better catalytic activity [35].

4. Conclusion

The oxidation of PAHs (**1**–**4**) with H_2O_2 catalyzed by **17** in IL co-solvent mixture gives the oxidized products, particularly quinones in high yields. The presence of electron-withdrawing groups at the aromatic ring of **17** and, the use of non-coordinating IL and coordinating organic solvent improves the yields. The present method offers various advantages such as high yield of product, milder conditions and reusability of the catalyst.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.catcom.2012.09.011>.

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