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The catalytic performance of Mn-tetraarylporphyrins in the highly selective oxidation of primary aromatic amines to azo compounds by Bu_4NHSO_5

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ABSTRACT

The catalytic activity of Mn(TPP)OAc/imidazole system in the oxidation of aromatic amines using Bu_4NHSO_5 in CH_3CN, provides a rapid and highly selective method for the preparation of symmetrical azoarenes, in high/excellent yield (78–96%) and short time (<5 min). The selectivity of the reaction depends remarkably on both the axial ligand and the nature of the solvent. Electron-deficient anilines are oxidized slowly and yield azoxy arenes as the main product. The presence of an electron-donating group on the phenyl ring of both the aromatic amine and the porphyrin ligand of the catalyst enhances the oxidative reactivity of the amine as well as the catalytic activity of the Mn-catalyst. The high turn over rates obtained for Mn-porphyrin reflect the efficiency as well as the relative stability of this biomimetic catalyst towards oxidative degradation. Some evidences for the participation of an Mn-oxo species as active oxidant in this catalytic process have been obtained.

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1. Introduction

The mild oxidation of aromatic amines, which can, if not performed with appropriate care, results in a complex mixture of different products (Fig. 1) has generated considerable debate in the literature [1,2]. In recent years there has been much effort to conduct this transformation selectively under catalytic conditions [3,4]. A possible strategy is to build biomimetic or bioinspired catalysts based on enzymatic systems that have been selected by living organisms for selective oxidation during life evolution [5]. Accordingly, porphyrin-based catalysts have been developed as synthetic models of monooxygenase enzymes like cytochrome P-450 and peroxidase, for the selective oxidation of organic compounds in combination with various oxygen sources [6–8]. While, the high selectivity and the excellent turnover numbers have been achieved for numerous oxidation processes such as alkane hydroxylation and olefin epoxidation using these biomimetic catalysts [9,10], the catalytic role of metalloporphyrins in the oxidation of primary aromatic amines still lag behind [11–13].

Among various products obtained in the oxidation of aromatic amines, azoarenes (Fig. 1D) are of extreme interest due to their vast and attractive applications. They are useful synthetic intermediates and analytical reagents [14]. They have been widely utilized as dyes [15] and in the area of organic nonlinear optics (NLO) in modern technology [16–19]. In addition, azo chromophores contained in

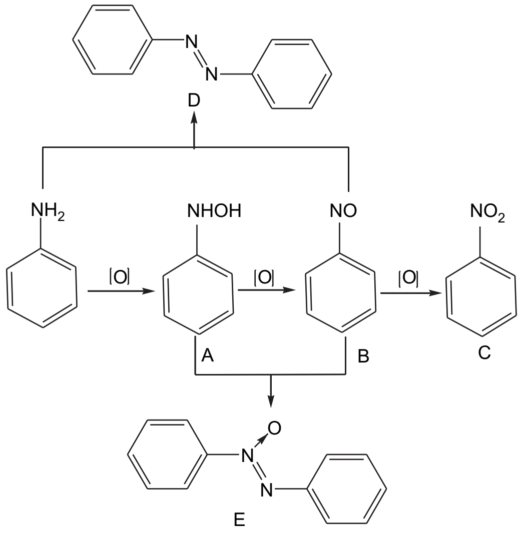

Fig. 1. The formation of different products in the oxidation of aromatic amines.

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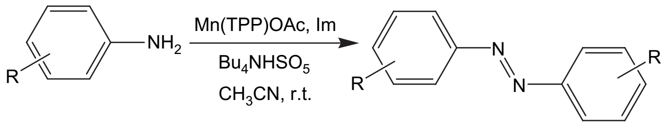


Fig. 2. Selective oxidation of aromatic amines to azoarenes using Bu_4NHSO_5:Im:Mn(TPP)OAc system in CH_3CN.

polymers may be oriented by polarized light irradiation in a process that involves both thermal- and photoisomerization of the azo linkage [20,21].

The promising employment of tetrabutylammonium peroxymonosulfate (Bu_4NHSO_5) as an organic salt of Oxone® ($2KHSO_5$, $KHSO_4$, K_2SO_4) in catalytic oxidation of hydrocarbons using Mn(TPP)OAc catalyst in the presence of imidazol co-catalyst [22], led to the development of highly selective methods for oxidation of sulfur-containing compounds [23] and alcohols [24]. We have also applied successfully this potent oxygen source for oxidation of aldimines to oxaziridines under non-catalytic conditions [25].

In a continuation of our ongoing research on the application of Bu_4NHSO_5 in the oxidation of different organic compounds we decided to examine its potential in the oxidation of primary aromatic amines. Very recently we have presented [26] a simple, safe and highly efficient method for selective oxidation of primary aromatic amines to azoxy arenes (Fig. 1E) which is the key material for electronic devices due to their liquid crystalline properties [27,28]. However, when we performed this oxidation process under the influence of simple Mn(TPP)OAc catalyst in the presence of an imidazol axial base in CH_3CN, a highly selective method for preparation of azoarenes in high yields and short times resulted which is the subject of this report (Fig. 2). The influence of electronic and structural requirements in the amine molecule and porphyrin ligand on the reaction rates has also been investigated in this study.

2. Results and discussion

2.1. The effects of axial ligand and solvent on the oxidation of aniline using Bu_4NHSO_5 catalyzed by Mn(TPP)OAc

To find the optimum reaction conditions, the oxidation of aniline by Bu_4NHSO_5 was used as a model reaction. Initially, the catalytic activity of simple manganese salts such as $Mn(OAc)_2 \cdot 4H_2O$ in the oxidation of aniline was examined in CH_2Cl_2 affording to a mixture of products containing azobenzene in 17% yield within 5 min. When this oxidation reaction was performed in the presence of Mn(TPP)OAc, azobenzene was obtained in 46.5% yield at the same time. It is well established, in many cases, rates and selectivity of oxidation reactions catalyzed by metalloporphyrins can be improved considerably by performing the reactions in the presence of nitrogen donors as co-catalyst [29,30]. The influence of a number of common heterocyclic nitrogen donors on the oxidation of aniline mediated by Mn(TPP)OAc was investigated (Fig. 3). It was observed that the selectivity of the reaction was dramatically dependent upon the nature of co-catalyst, in which imidazole with strong π-donating ability is the best co-catalyst in comparison with pyridines in terms of conversion and azobenzene yield [13,31]. Also an increase in the Im/Mn–porphyrin molar ratio up to 20:1 remarkably improved the selectivity of azobenzene formation (Fig. 4). However, as outlined in Fig. 4, further increase in the ratio, decreases the azobenzene yield.

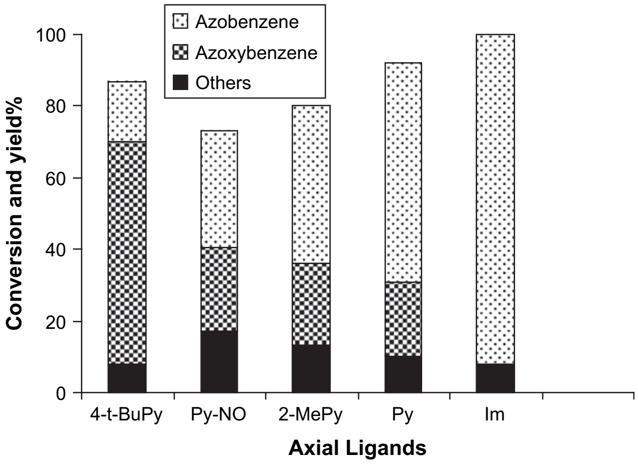


Fig. 3. The effect of different axial ligands upon oxidation of aniline catalyzed by Mn(TPP)OAc with molar ratio of 100:170:20:1 for amine:Bu_4NHSO_5:Im:catalyst in CH_2Cl_2 after 5 min.

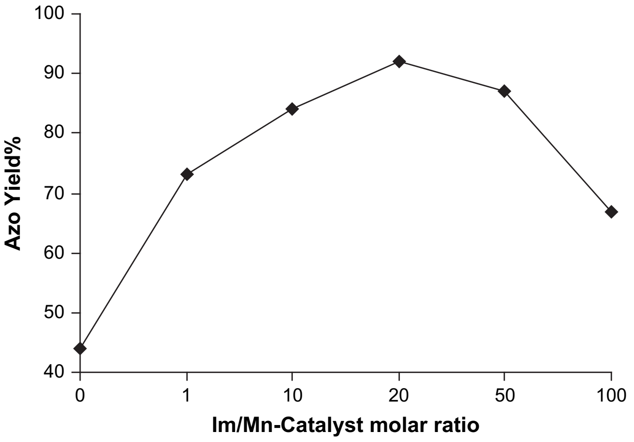


Fig. 4. The effect of Im/catalyst molar ratios upon the formation of azobenzene in the oxidation of aniline catalyzed by Mn(TPP)OAc with molar ratio of 100:170:X:1 for amine:Bu_4NHSO_5:Im:catalyst in CH_2Cl_2 after 5 min.

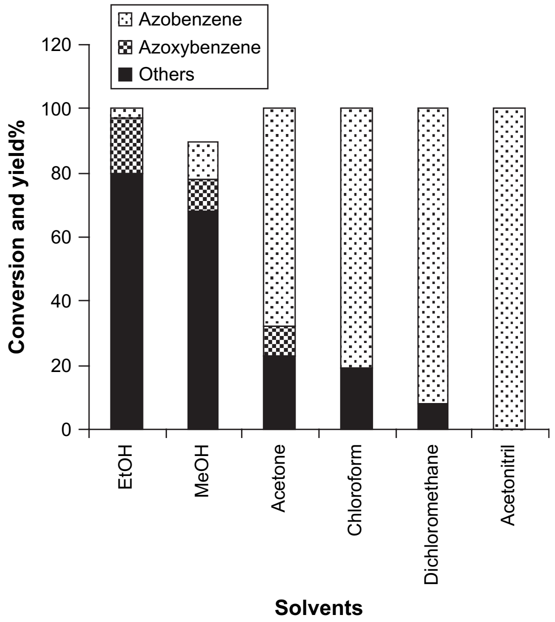
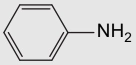
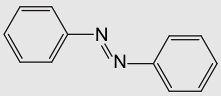
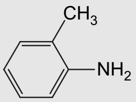
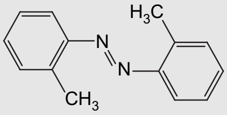
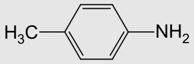
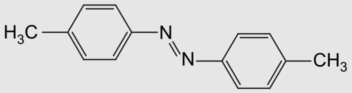
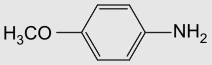
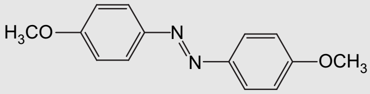
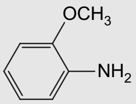
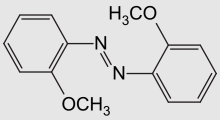
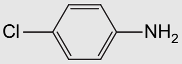
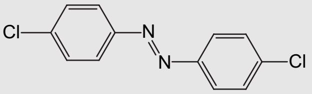
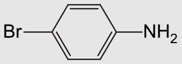
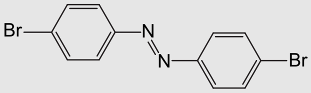
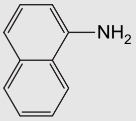
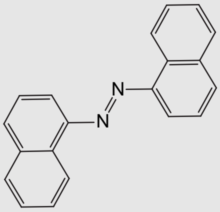
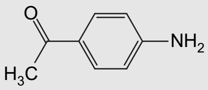
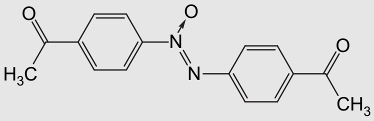
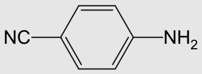
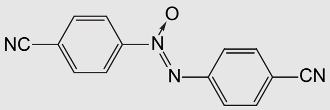
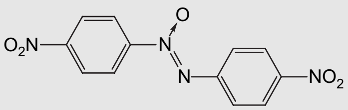


Fig. 5. The influence of solvents upon the oxidation of aniline catalyzed by Mn(TPP)OAc in the presence of Im with molar ratio of 100:170:20:1 for amine:Bu_4NHSO_5:Im:catalyst after 5 min.

Table 1

Oxidation of various aromatic amines using Bu_4NHSO_5 catalyzed by Mn(TPP)OAc in the presence of Im in CH_3CN[a]

Entry	Amine	Product[b]	M.p. °C (Lit.) Ref.	Isolated yield% (selectivity %)	Time (min)
1			67 (68)[c]	95 (100)	5
2			55–56 (54)[c]	78 (85)[g]	5
3			142 (143)[c]	93 (100)	5
4			158–160 (160)[c]	96 (100)	1
5			141 (143)[c]	92 (100)	3
6			186 (185)[c]	90 (100)	5
7			204–206 (205)[c]	92 (100)	5
8			191 (190)[d]	87 (94)[g]	5
9			192 (191–193)[e]	70 (79)[h]	10
10			221 (222–223)[e]	46 (70)[h]	10
11			190 (192)[f]	12 (75)[h]	10

[a] The reactions were run under air at 25 °C and molar ratio for amine:Bu_4NHSO_5:Im:Mn(TPP)OAc is 100:170:20:1.

[b] All products were identified by their IR, ^1H NMR, and MS spectral data in comparison with authentic samples prepared according to the literature [26,46].

[c] See Ref. [46].

[d] See Ref. [47].

[e] See Ref. [48].

[f] See Ref. [49].

[g] The remainder is other minor unidentified products.

[h] The remainder is essentially unreacted amines and the related azo products in 21%, 20% and 4% yields for entries 9, 10 and 11, respectively.

Table 2
The competitive reactions between aniline and other molecules using Bu_4NHSO_5 catalyzed by Mn(TPP)OAc in the presence of Im[a]

Entry	Substrates	Products	Yield%[b]
1	Aniline + styrene	Azobenzene + styrene oxide	97+ 2.5
2	Aniline + benzyl alcohol	Azobenzene + benzaldehyde	92.7 + 6.8
3	Aniline + diphenylsulfide	Azobenzene + diphenylsulfoxide + diphenylsulfone	46.5 + 3.9 + 2.5

[a] The reactions were run for 5 min in CH_3CN at 25 °C and molar ratio for amines:Bu_4NHSO_5:Im:Mn(TPP)OAc is 100:170:20:1.
[b] GC yield based on starting materials.

Finally, we examined the influence of solvent nature on this catalytic oxidation reaction (Fig. 5). From the evaluation of five reaction solvents, CH_3CN was found to be the solvent of choice in terms of azobenzene yield and selectivity. The complete oxidation of aniline under these optimized conditions afforded azobenzene as sole product in less than 5 min.

2.2. *Catalytic oxidation of primary aromatic amines by Mn(TPP)OAc/Im/Bu_4NHSO_5 system in CH_3CN*

A variety of substituted anilines were subjected to the oxidation under the optimized conditions (Table 1). Inspection of the results in Table 1 demonstrates the efficiency and also selectivity of oxidation system. Anilines with either no substituent or having an electron-donating or halogen groups, converted to the corresponding azoarenes in 78–96% yields in short times (Table 1, entries 1–7). When this system was applied to the oxidation of 1-naphthylamine, the related azo was secured in 87% yield within 5 min (entry 8). Thus, according to these results and those obtained in the absence of catalyst in absolute ethanol [26], Bu_4NHSO_5 can be used easily as a versatile oxygen source for the selective synthesis of azo- and azoxy arenes under catalytic and non-catalytic conditions, respectively.

It is noteworthy that the oxidation of less reactive anilines having electron-withdrawing groups such as –COMe, –CN and $-NO_2$ using this catalytic system required longer reaction times and produced azoxy arenes as main products along with azoarenes in low yields (Table 1, entries 9–11).

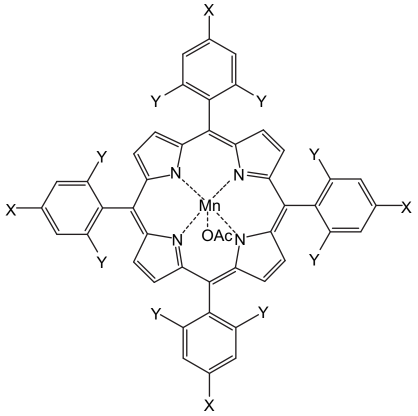
Based on the literature [32–34] it seems that an electron-donating substitute on the phenyl ring of aniline enhances remarkably the conversion rate of *N*-arylhydroxylamine (Fig. 1A) to nitrosoarene (Fig. 1B) followed by condensation with starting aromatic amine resulted in azoarene formation (Fig. 1D). The formation of azoxy arene (Fig. 1E) accompanied with azoarene in the oxidation of electron-deficient anilines, presumably, resulted from a competitive reaction between *N*-arylhydroxylamine (A) and starting amine for condensation with nitrosoarene (B) as shown in Fig. 1.

In order to show the merit of this simple oxidation method, some competitive reactions between aniline and other molecules, which have been previously oxidized efficiently by this catalytic system were investigated (Table 2). Inspection of the results in Table 2 indicates excellent chemoselectivity for amine oxidation rather than alkene epoxidation (entry 1, 97% azobenzene *vs* 2.5% styrene oxide) and also oxidation of alcohol (entry 2, 92.7% azobenzene *vs* 6.8% benzaldehyde). However, oxidation of aniline in the presence of sulfides gave the same yields of the related oxidation products (Table 2, entry 3).

2.3. *The influence of electronic requirements of substrate on the rate of oxidation reaction*

From this strategy it is evident that the efficiency of oxidation in this catalytic system is very dependent on the electronic requirements of the substrate. Electron-donating and electron-withdrawing substitutes on the phenyl ring of aniline have a pronounced effect on the rate of oxidation and the product character. To examine the electronic influence of substitute on the reactivity of aromatic amines in the oxidation reaction, the turnover frequency (TOF) of Mn(TPP)OAc catalyst in the oxidation of different anilines was determined after 1 h. It was observed that the presence of –OMe as an electron-donating group enhances the TOF in comparison with molecule having no substitute on the ring (5760/h for *p*-anisidine *vs* 1140/h for aniline). Whereas, a substrate with a strong electron-withdrawing $-NO_2$ group on the phenyl ring displays a lower TOF than aniline (96/h for *p*-nitroaniline *vs* 1140/h for aniline) under the same conditions. Obviously, these high turnover rates of simple Mn(TPP)OAc catalyst [24] obtained in the oxidation of various anilines indicate well the efficiency and also relative stability of Mn-catalyst towards oxidative degradation [35,36].

More evidences for electronic effects of substitutes in the substrate have been obtained by performing competitive reactions between different anilines. We have found excellent selectivity among the electronically different anilines. Electron-rich *p*-anisidine converted rapidly to the related azo in 94% yield in the presence of aniline which was oxidized slightly (<5%). Also oxidation of electron-deficient *p*-nitroaniline did not occur during the oxidation of aniline to azobenzene (97%). These results demonstrate clearly the accelerating effect of an electron-donating substitute on the phenyl ring of aniline upon its oxidative reactivity.



	X	Y
Mn(TPP)OAc	H	H
Mn[T(4-OMeP)P]OAc	OMe	H
Mn[T(4-NO_2P)P]OAc	NO_2	H
Mn(TMP)OAc	Me	Me

Fig. 6. Mn-porphyrins employed in this study.

2.4. The influences of electronic and structural requirements of porphyrin ligand on the activity of Mn-catalyst

The significant influence of porphyrin nature on the catalytic activity of metalloporphyrins in the oxidation reactions is well known [37–40]. In the present oxidation method the catalytic activity of different electronically and structurally Mn-porphyrins (Fig. 6) have been compared in the oxidation of different anilines. First, to explore the effect of electronic requirements in porphyrin ligand upon the catalytic activity of Mn-porphyrins in the oxidation reaction, three *para*-substituted Mn-porphyrins having different electronic groups and with very similar steric environments at their Mn centers in the oxidation of various anilines were used (Fig. 7). The lower activity of Mn[T(4-NO_2P)P]OAc containing an electron-withdrawing –NO_2 group compared to simple Mn(TPP)OAc and especially electron-rich Mn[T(4-OMeP)P]OAc reflects the reducing influence of electron-deficient substitute upon the catalytic activity of Mn-catalyst in this oxidation method.

Then, the steric requirements in this catalytic system were examined. The catalytic activity of the sterically more demanding Mn(TMP)OAc in the oxidation of aniline and 2-Me-aniline was compared to Mn(TPP)OAc (Fig. 8). When the bulky Mn(TMP)OAc catalyst was used, the steric factors clearly became dominant and aniline displays a greater reactivity than the hindered 2-Me-aniline under the same conditions (53% *vs* 34%). While, the same yields of the related azo products were obtained under the influence of Mn(TPP)OAc (63% *vs* 62%). The observed steric hindrance at the oxygenation site of Mn(TMP)OAc probably corresponded to an Mn-oxo species into the cavity of the catalyst. We confirmed this point by monitoring the UV–vis spectral changes during the oxidation of aniline by Bu_4NHSO_5 in the presence of Mn(TMP)OAc and imidazole (Fig. 9). The addition of Bu_4NHSO_5 (10 μmol) to a solution of MnTMP(OAc) (0.05 μmol) (Soret, $\lambda_{max} = 478$ nm), in CH_2Cl_2 (3 ml), containing ImH (5 μmol), yield an intense Soret band, $\lambda_{max} = 408$ nm, presumably due to the formation of MnTMP(Im)(O) species at room temperature [29]. Addition of a large excess of aniline (10 μl) immediately gave the original Soret ($\lambda_{max} = 478$ nm) clearly indicating the mediation of an Mn-oxo species as active oxidant in this catalytic process. Further investigation on dramatic influence of axial ligands and porphyrin structures upon the selectivity of reaction is underway in this area.

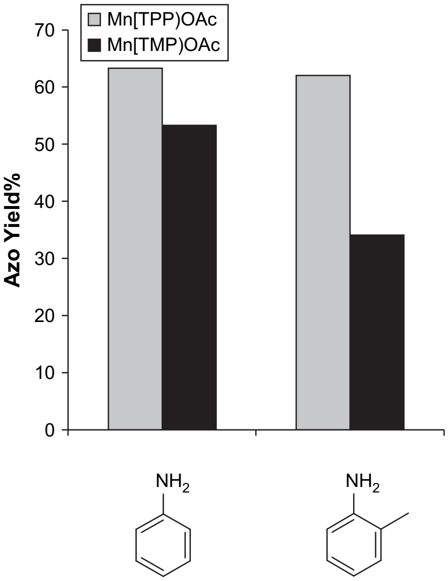


Fig. 8. The comparison of catalytic activity of Mn(TMP)OAc and Mn(TPP)OAc in the oxidation of aniline and 2-Me-aniline with molar ratio of 100:100:20:1 for amine:Bu_4NHSO_5:Im:catalyst in CH_3CN after 5 min.

3. Conclusion

In conclusion, Mn(TPP)OAc in combination with imidazole is an excellent biomimetic catalyst for activation of Bu_4NHSO_5 in rapid oxidation of electron-rich aromatic amines to the related azoarenes in high to excellent yields and excellent selectivity under mild conditions. Whilst, all substituted anilines converted selectively to

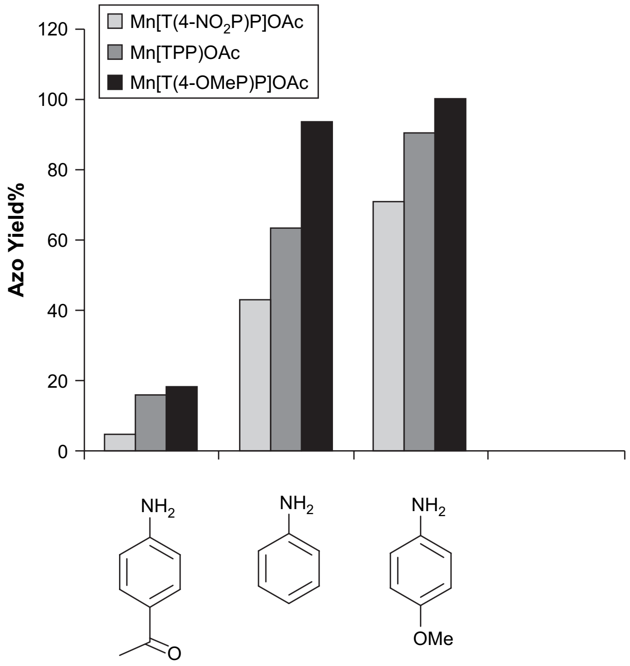


Fig. 7. The comparison of catalytic activity of Mn(TPP)OAc, Mn[T(4-OMeP)P]OAc, and Mn[T(4-NO_2P)P]OAc in the oxidation of different anilines with molar ratio of 100:100:20:1 for amine:Bu_4NHSO_5:Im:catalyst in CH_3CN after 5 min.

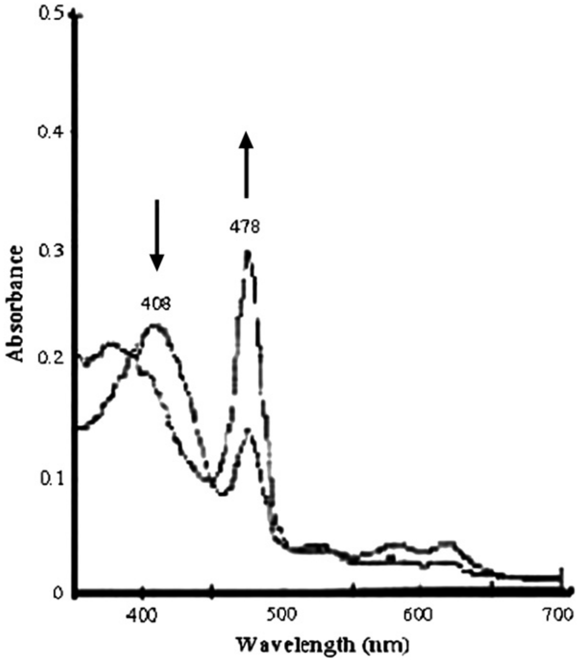


Fig. 9. The UV–vis spectra represent conversion of Mn-oxo species (MnTMP(ImH)(O), Soret, $\lambda_{max} = 408$ nm) to the original Mn(TMP)OAc (Soret, $\lambda_{max} = 478$ nm) immediately after addition of aniline at 25 °C.

azoarenes, it was observed that electron-withdrawing groups on the phenyl ring of aniline turned the selectivity of reaction to the formation of azoxy products. The presence of an electron-donating group on the phenyl ring of both arylamine and porphyrin ligands increases the reactivity of substrate as well as catalytic activity of Mn-porphyrin catalyst. The applicability of this simple methodology to a wide variety of primary aromatic amines with excellent selectivity combined with high turnover rates and relative stability of Mn-catalyst are the strong points of this catalytic method.

4. Experimental

4.1. General remarks

$Mn(OAc)_2 \cdot 4H_2O$, Oxone®, tetrabutylammonium hydrogen sulfate, nitrogen bases and amines were purchased from Merck or Fluka Chemical Companies. The free base porphyrins: $TPPH_2$ [41], $T(4\text{-}OMeP)PH_2$ [41], $T(4\text{-}NO_2P)PH_2$ [42], and $TMPH_2$ [43], were prepared and purified by methods reported previously. Mn(Por)OAc complexes were obtained using the $Mn(OAc)_2 \cdot 4H_2O$ according to the procedure of Alder et al. [44]. The freshly prepared Bu_4NHSO_5 [45] was a much stronger oxidant than commercially available samples. Since the oxidizing ability of Bu_4NHSO_5 samples would reduce with time, in order to obtain reproducible results, the freshly prepared oxidant was refrigerated within three days.

Caution: Bu_4NHSO_5 should be considered as a potential explosive [45].

4.2. Instrumentation

Purity determinations of the products were accomplished by GC on a Shimadzu GC-16A instrument using a 25m CBP1-S25 (0.32 mm ID, 0.5 μm coating) capillary column. IR spectra were recorded on a Perkin Elmer 780 instrument. UV–vis spectra were recorded on a 160 Shimadzu spectrophotometer. NMR spectra were recorded on a Brucker Avance DPX 250 MHz instrument. Mass spectra were recorded on a Shimadzu GC–MS-QP5050A.

4.3. Typical procedure for oxidation of aniline using Bu_4NHSO_5 catalyzed by Mn(TPP)OAc/Im system in CH_3CN

To a mixture of aniline (1 mmol, 0.093 g), Mn(TPP)OAc (0.01 mmol, 7.26 mg) and imidazol (0.2 mmol, 0.0136 g) in CH_3CN (5 ml) was added freshly prepared Bu_4NHSO_5 (1.7 mmol, 0.07 g). Reaction mixture was stirred at 25 °C for appropriated reaction time, which was monitored by GC (Table 1, entry 1). After 5 min, the desired product was isolated by column chromatography eluted with *n*-hexane/ethyl acetate (10:1). Evaporation of the solvent gave azobenzene in 95% yield (0.086 g). Red plates (M.p. 67 °C). IR (KBr) cm^{-1} 1585, 1484, 1460, 1438, 1397; ^1H NMR (250 MHz) δ (ppm) 7.5 (m, 3 H), 7.90 (d, 2H); ^{13}C NMR (63 MHz) δ (ppm) 122, 129, 131, 153; MS m/z 182 $[M]^+$.

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