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Green oxidation of 2-imidazolines with *tert*-butyl hydroperoxide catalyzed by supported manganese(III) porphyrin in water

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Green oxidation of 2-substituted imidazolines with *tert*-butyl hydroperoxide catalyzed by tetraphenylporphyrinatomanganese(III) chloride, [Mn(TPP)Cl], supported on polystyrene and silica bound imidazole is reported. A variety of 2-imidazolines were efficiently converted to their corresponding imidazoles by these catalytic systems in H₂O. Ultrasonic (US) irradiation enhanced the catalytic activity of these catalysts and higher product yields were observed at shorter reaction times. These catalysts were re-used several times without significant loss of their catalytic activities. The effect of reaction parameters such as catalyst amount, reaction temperature, and the effect of US irradiation on the catalytic activity was also investigated.

Keywords: Manganese(III) porphyrin; TBHP; Imidazolines; Oxidation; Supported catalyst; Ultrasonic irradiation

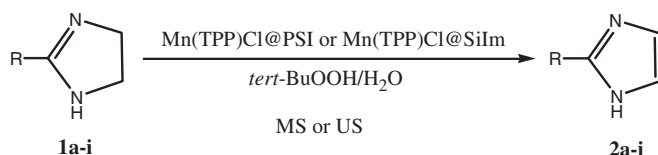
1. Introduction

Oxidation of organic substrates by mimicking the function of monooxygenase enzymes like cytochrome P-450 has attracted much attention. In these systems, oxo metalloporphyrins, in which the metal possesses a high oxidation number, are used as catalysts [1–8]. Different single oxygen donors such as iodosylbenzene [9, 10], hypochlorites [11, 12], amine *N*-oxides [13], alkyl hydroperoxides [14], hydrogen peroxide [15, 16], and sodium periodate [17, 18] in the presence of natural cytochrome P-450 and metalloporphyrin complexes are able to oxidize organic compounds.

Among metalloporphyrins, Mn porphyrins have attracted much attention both as oxidation catalyst and as therapeutics for oxidative stress-related diseases [19].

One problem associated with homogeneous metalloporphyrins is their degradation in the reaction media and formation of μ -oxo catalytically inactive dimers. One way to overcome these problems is to immobilize them on a solid organic or inorganic support [20, 21]. Although the reactivity of this heterogenized catalyst is less than the

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Scheme 1. Oxidation of imidazolines with *tert*-BuOOH catalyzed by [Mn(TPP)Cl@PSI] and [Mn(TPP)Cl@SiIm].

homogeneous counterpart, recovery and reusability of the catalyst makes it cost effective.

Manganese porphyrins have been immobilized on different supports *via* coordination, covalent anchorage, electrostatic interactions, encapsulation, and intercalation. These heterogeneous catalysts have been used for oxidation of organic compounds [22–32]. Previously, our research group reported the immobilization of manganese porphyrins on silica, polystyrene, and multiwall carbon nanotubes *via* covalent attachment and used them for oxidation of organic compounds with NaIO₄ [17, 18, 22, 24–30].

Oxidation of 2-imidazolines to their corresponding imidazoles is biologically and pharmaceutically very important, since many imidazole derivatives possess anti-hypertensive, anti-inflammatory, antibacterial, and antidiabetic activities [33, 34]. 2-Imidazolines can be easily prepared from nitriles and ethylenediamine [35, 36]. Oxidation of 2-imidazolines by an oxidizing agent should provide an efficient method for preparation of imidazole derivatives. Several reagents such as Zn–Al₂O₃ [37], Ni [38], Se [39], Pd/C [40], MnO₂ [41], BaMnO₄ [42], DMSO [43], KMnO₄@SiO₂ [44], KMnO₄@K10 [45], KMnO₄@Al₂O₃ [46], trichloroisocyanuric acid [47], KMnO₄-refluxing dioxane [48], activated carbon–O₂ [49], microwave assisted in the presence of MnO₂ [50], homogeneous manganese(III) porphyrins [51, 52], and manganese(III) salophen [53] have been previously reported for this purpose. Some of these reagents suffer from limitations, such as low yields of the products, very long reaction times, toxicity, the use of large excess of reagents, and harsh reaction conditions. Also, some of these reagents are not effective for oxidation of 2-alkylimidazolines. Therefore, development of a mild and efficient method for oxidation of 2-imidazolines to their corresponding imidazoles is of interest.

Here, oxidation of 2-substituted imidazolines to 2-imidazoles in the presence of tetraphenylporphyrinatomanganese(III) chloride supported on chloromethylated polystyrene-bound imidazole, [Mn(TPP)Cl@PSI], and silica-bound imidazole, [Mn(TPP)Cl@SiIm], with *tert*-BuOOH in water as a green solvent under mechanical stirring (MS) and ultrasonic (US) irradiation is reported (scheme 1).

2. Experimental

All materials were of commercial reagent grade, purchased from Merck or Fluka chemical companies. All 2-substituted imidazolines were synthesized by the reported procedures [35, 36]. Tetraphenylporphyrin was prepared and metallated according to the reported procedure [54]. A new and efficient method has been reported for

the synthesis of Mn(III) metalloporphyrins [55]. The [Mn(TPP)Cl@SiIm] and [Mn(TPP)Cl@PSI] catalysts were prepared and characterized according to our reported procedures [25, 52].

¹H NMR spectra were recorded on a Bruker-Avance AQS 400 MHz. TLC monitored all reactions and all yields refer to isolated products. A UP 400S US processor equipped with a 3 mm wide and 140 mm long probe, which was immersed directly into the reaction mixture, was used for sonication. The operating frequency was 24 KHz and the output power was set at 0–400 W through manual adjustment. The total volume of the solution was 15 mL. The temperature reached 40°C during sonication.

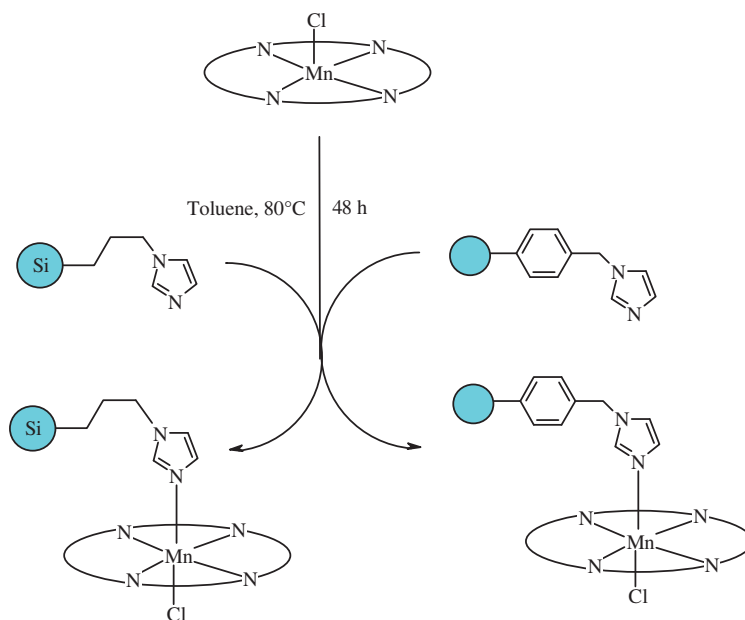
2.1. General procedure for oxidation of imidazolines with *tert*-BuOOH in water catalyzed by [Mn(TPP)Cl@PSI] and [Mn(TPP)Cl@SiIm] under MS or US irradiation

2-Imidazoline (1 mmol) was dissolved in H₂O (12 mL) and the catalyst [Mn(TPP)Cl@PSI] (0.012 mmol) or [Mn(TPP)Cl@SiIm] (0.03 mmol) was added to this solution. Then, *tert*-BuOOH (2 mmol, 70% dissolved in H₂O) was added and the reaction mixture was stirred at 80°C or exposed to US irradiation. The progress of the reaction was monitored by TLC (eluent: EtOAc/MeOH: 4/1). After the reaction was completed, the catalyst was filtered. The solvent was evaporated by rotary evaporator and the resulting crude material was purified by column chromatography on alumina to afford the pure imidazole derivatives. IR and ¹H NMR spectral data confirmed the identity of the products.

3. Results and discussion

3.1. Oxidation of imidazolines with *tert*-BuOOH in water catalyzed by [Mn(TPP)Cl@PSI] or [Mn(TPP)Cl@SiIm] under MS or US irradiation

Since a great amount of waste in the environment is attributed to utilizing organic solvents [56], there is growing demand for development of organic reactions in environmentally friendly media [57]. Water as a nontoxic, nonflammable, cheap, renewable, and environmentally friendly reaction medium has gained special interest in organic synthesis [58–61]. The remarkable properties seen in water as a result of its chemical and physical properties are very useful for selectivity/reactivity that cannot be attained in organic solvents [62, 63]. Consequently, development of highly efficient and environmentally benign procedures using reusable catalysts in water as the reaction medium is highly desirable. We decided to investigate the catalytic activity of [Mn(TPP)Cl@PSI] or [Mn(TPP)Cl@SiIm] in the biomimetic oxidation of 2-imidazoline with *tert*-BuOOH in water under agitation by magnetic stirring (MS) and under US irradiation. The preparation route of both catalysts are shown in scheme 2. First, the amount of each catalyst was optimized in oxidation of 2-phenyl imidazoline with *tert*-BuOOH under MS. As shown in table 1, the highest yield was obtained in the presence of 500 mg (12 μmol) of [Mn(TPP)Cl@PSI] and 500 mg (30 μmol) of [Mn(TPP)Cl@SiIm]. Then the effect of reaction temperature was also investigated for the same reaction. The results, which are summarized in table 2, show that the highest



Scheme 2. The preparation route for the catalysts.

Table 1. Optimization of the catalyst amounts in the oxidation of 2-phenyl imidazole with *tert*-BuOOH.^a

Entry	[Mn(TPP)Cl@PSi]		[Mn(TPP)Cl@SiIm]	
	Catalyst amount (mg)	Yield (%) ^b	Catalyst amount (mg)	Yield (%) ^b
1	0	0	0	8
2	100 (2.4 μmol)	25	100 (6.0 μmol)	32
3	200 (4.8 μmol)	36	200 (12.0 μmol)	43
4	300 (7.2 μmol)	58	300 (18.0 μmol)	65
5	400 (9.6 μmol)	63	400 (24.0 μmol)	70
6	500 (12.0 μmol)	83	500 (30.0 μmol)	90
7	600 (14.4 μmol)	83	600 (36.0 μmol)	90

^aReaction conditions: 2-phenyl imidazole (1 mmol), catalyst, *tert*-BuOOH (2 mmol), H₂O (12 mL), *T* = 80°C after 10 h.^bIsolated yield.Table 2. The effect of temperature on the oxidation of 2-phenyl imidazole with *tert*-BuOOH in water catalyzed by [Mn(TPP)Cl@PSi] and [Mn(TPP)Cl@SiIm].^a

Entry	Temperature (°C)	[Mn(TPP)Cl@PSi]	[Mn(TPP)Cl@SiIm]
		Yield (%) ^b	Yield (%) ^b
1	25	13	15
2	40	27	35
3	50	33	40
4	60	50	57
5	70	76	83
6	80	83	90
7	90	83	90

^aReaction conditions: 2-phenyl imidazole (1 mmol), [Mn(TPP)Cl@PSi] (0.012 mmol), [Mn(TPP)Cl@SiIm] (0.03 mmol), *tert*-BuOOH (2 mmol), H₂O (12 mL) after 10 h.^bIsolated yield.

Table 3. Oxidation of 2-imidazolines with *tert*-BuOOH catalyzed by [Mn(TPP)Cl@PSI] and [Mn(TPP)Cl@SiIm] in water.^a

Row	Imidazoline	Imidazole	[Mn(TPP)Cl@PSI]		[Mn(TPP)Cl@SiIm]	
			Yield (%) ^b		Yield (%) ^b	
			MS (10 h)	US (1 h)	MS (10 h)	US (1 h)
1			83	88	90	95
2			63	75	70	82
3			66	73	73	84
4			71	77	78	80
5			85	90	92	97
6			79	85	86	92
7			85	90	91	97
8			66	68	70	75
9			86	91	88	93

^aReaction conditions: 2-imidazoline (1 mmol), [Mn(TPP)Cl@PSI] (0.012 mmol), [Mn(TPP)Cl@SiIm] (0.03 mmol), *tert*-BuOOH (2 mmol), H₂O (12 mL). *T* = 80°C for MS and 40°C for US.

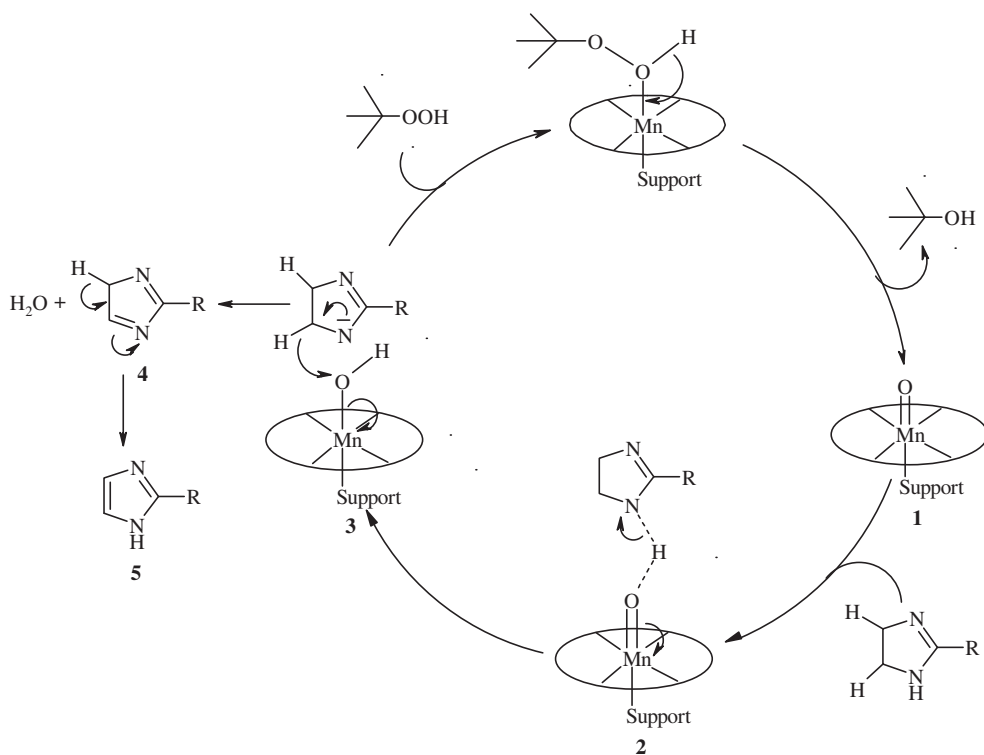
^bIsolated yield.

yield was observed at 80°C. The effect of other oxidants such as NaIO₄ and H₂O₂ was also investigated in the model reaction. The results showed that these oxidants were not as efficient as *tert*-BuOOH, and in both cases the amounts of oxidized product was less than 40%. Under these conditions, a wide range of 2-aryl-2-imidazolines were oxidized to their corresponding imidazoles with *tert*-BuOOH in good to excellent yields (entries **1a–h**) under MS at 80°C (table 3). The results showed that in the absence of catalyst only 8% of imidazoles were produced.

Some reported reagents such as Pd/C [40] and DMSO [43] were not able to oxidize 2-alkylimidazolines; one advantage of this catalytic system is its ability to oxidize 2-methyl imidazoline (entry **9**).

The exact mechanism of the reaction is not clear. However, a plausible explanation is that the catalyst is first transformed to the corresponding oxo compound **1** by *tert*-BuOOH. The oxo compound reacts with imidazoline to afford **2** which in turn converted to **3** by proton transfer. The intermediate **3** is converted to **4** by elimination of water and releases the catalyst for the next catalytic cycle. The intermediate **4** is finally converted to the desired product **5** by hydrogen shift (scheme 3).

The main effect of US irradiation in liquids is the cavitation phenomenon, which involves numerous tiny gas bubbles called cavitation bubbles. The collapse of the bubbles generates high temperatures and pressures at the center of the bubbles. These



Scheme 3. The proposed mechanism.

local effects produce a variety of radicals and highly active intermediates, which initiate secondary chemical reactions in the bulk solution. These radicals can react with contaminants to form byproducts.

The most successful applications of ultrasound have been found in heterogeneous chemistry involving solids and metals. This is due to the mechanical impact of ultrasound on solid surfaces. The mechanical effects of ultrasound offer an opportunity to overcome problems associated with conventional solid/metal reactions: break-up of the surface structure allows penetration of reactants and/or release of materials from the surface, degradation of large solid particles due to shear forces induced by shock waves, microstreaming leads to reduction of particle size and increase in surface area, and accelerated motion of suspended particles leads to better mass transfer [64].

When the same reactions were carried out under US irradiation, the reaction times were reduced to 1 h (table 3). As mentioned previously, the effect of US irradiation on the catalytic activity enhancement may be due to the break-up of the agglomerates during sonication. A part of the US irradiation effect is due to this phenomenon. To stress this point, the catalytic activity of a sonicated sample of both catalysts was studied in oxidation of 2-phenyl imidazole under mechanical stirring with reaction time reduced from 10 h to 7.5 h for [Mn(TPP)Cl@PSI] and from 10 h to 7 h for [Mn(TPP)Cl@SiIm] for complete oxidation of 2-phenyl imidazole to its

corresponding 2-phenyl imidazole. These results show that in addition to break-up of the agglomerates, other factors such as thorough mixing of the reactants and producing hot spots are the reasons for catalytic enhancement by US irradiation. When the oxidation reactions were carried out under reflux, the reaction times decreased, confirming the effect of producing hot spots on the enhancement of catalytic activity (table 3).

3.2. Catalyst reusability

The stability of the supported catalysts was monitored using multiple sequential oxidation of 2-phenyl imidazoline with sodium periodate under agitation with magnetic stirring or under US irradiation. For the repeated reactions, the catalyst was recovered, washed exhaustively with water, and diethyl ether, respectively, and dried before being used with fresh 2-phenyl imidazoline and *tert*-BuOOH. In both cases, the catalysts were consecutively reused five times without significant loss of activity (table 4).

Also, the catalytic behavior of the separated liquid was tested by addition of fresh 2-phenyl imidazoline and *tert*-BuOOH to the filtrates after each run. Oxidation under the same reaction conditions as with catalyst showed that the obtained results were the same as blank experiments.

The nature of recovered catalysts was confirmed by monitoring their DR UV-Vis spectra. The results showed that no change was observed after consecutive catalytic cycles (see "Supplementary material," figures S1 and S2). These observations indicate that the catalysts are stable during the catalytic experiments.

In order to show the effectiveness of the present method in dehydrogenation of imidazolines, the obtained results were compared with some reported systems. The results showed that these two catalytic systems have the following advantages in the oxidation of imidazolines: mild reaction conditions, high to excellent yield, reusability of catalysts, using water as a green solvent, and solubility of the oxidant in water (table 5).

Table 4. The results of [Mn(TPP)Cl@PSI] and [Mn(TPP)Cl@SiIm] after catalyst recovery and the manganese leached in the oxidation of 2-phenyl imidazoline with *tert*-BuOOH under magnetic stirring and US irradiation.

Run	[Mn(TPP)Cl@PSI]				[Mn(TPP)Cl@SiIm]			
	Yield (%) ^a		Mn leached (%) ^b		Yield (%) ^a		Mn leached (%) ^b	
	MS	US	MS	US	MS	US	MS	US
1	83	88	1.6	2.2	90	95	1.4	2
2	80	85	1.2	1.5	87	92	1.1	1.4
3	80	83	0	0.7	87	90	0	0.5
4	77	82	0	0	84	89	0	0
5	76	82	0	0	83	88	0	0

^aIsolated yield.

^bDetermined by atomic absorption spectroscopy.

Table 5. Comparison of the results in the dehydrogenation of 2-phenylimidazoline with some reported methods.

Entry	Reagent	Conditions	Time (h)	Yield (%)	Ref.
1	[Mn(TPP)Cl@PSI]/TBHP	80°C	10	83	Present method
2	[Mn(TPP)Cl@SiIm]/TBHP	80°C	10	90	Present method
3	Pd/C (10%)	Reflux	40	75	[40]
4	BaMnO ₄	Reflux	24	96	[42]
5	DMSO	120°C	48	51	[43]
6	TCCA/DBu	-15°C	3 min	93	[47]
7	KMnO ₄ /Dioxane	Reflux	16	40	[48]
8	O ₂ /Activated carbon	120°C	24	84	[49]
9	[Mn(TPP)Cl]/NaIO ₄	RT	5	84	[51]
10	[Mn(Salophen)Cl]/NaIO ₄	RT	0.5	90	[52]
11	[Mn(TPP)Cl@PSI]/NaIO ₄	RT	10	81	[53]

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