

Oxidation of 2-imidazolines to 2-imidazoles with sodium periodate catalyzed by polystyrene-bound manganese(III) porphyrin

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ABSTRACT

In the present work, the dehydrogenation of 2-substituted imidazolines with sodium periodate in the presence of tetraphenylporphyrinatomanganese(III) chloride supported on polystyrene-bound imidazole, [Mn(TPP)Cl@PSI] is reported. A wide variety of 2-imidazolines were efficiently converted to their corresponding imidazoles by the [Mn(TPP)Cl@PSI]/NaIO₄ catalytic system in a 1:2 CH₃CN/H₂O mixture under agitation with magnetic stirring. Ultrasonic irradiation enhanced the catalytic activity of this catalyst in the oxidation of 2-imidazolines and this led to shorter reaction times and higher product yields. This catalyst could be reused several times without significant loss of its catalytic activity.

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1. Introduction

Cytochrome P-450 is an iron porphyrin surrounded by proteins which can selectively catalyze a wide range of oxidation reactions [1]. Synthetic metalloporphyrins, as models for cytochrome P-450, have been found as highly efficient and homogeneous catalysts in the oxidation of organic compounds [2–8] in the presence of a single oxygen atom donor, such as PhIO, ClO⁻, H₂O₂, ROOH and IO₄⁻. These reactions reveal some information about the mechanism of the cytochrome P-450 monooxygenation enzyme [9–14]. One major drawback associated with these homogeneous catalysts is their degradation in reaction media, a result of which is they cannot be recovered. One way to overcome this disadvantage is their immobilization on solid supports [15–19].

Oxidation of 2-imidazolines to their corresponding imidazoles is biologically and pharmaceutically very important, since many imidazole derivatives possess antihypertensive, antiinflammatory, antibacterial and antidiabetic activities [20]. 2-Imidazolines can be easily prepared from nitriles and ethylenediamine [21,22]. Dehydrogenation of 2-imidazolines by an oxidizing agent should provide an efficient method for the preparation of imidazole derivatives. Several reagents such as Zn–Al₂O₃ [23], Ni [24], Se [25], Pd/C [26], MnO₂ [27], BaMnO₄ [28], DMSO [29], KMnO₄@SiO₂ [30], KMnO₄@K10 [31], KMnO₄@Al₂O₃ [32], trichloroisocyanuric acid [33], KMnO₄-refluxing dioxane [34], activated carbon–O₂ [35],

microwave-assisted in the presence of MnO₂ [36] and the homogeneous Mn(TPP)Cl/NaIO₄ catalytic system [37] 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 a large excess of the reagents and harsh reaction conditions. Also, some of these reagents are not effective for the oxidation of 2-alkylimidazolines. Therefore, the development of a novel, mild and efficient method for the oxidation of 2-imidazolines to their corresponding imidazoles is of great interest.

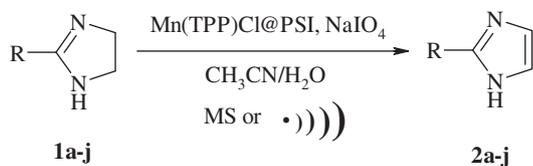
Over the last two decades, several homogeneous and heterogeneous metalloporphyrins have been developed for the oxidation of organic compounds [38–53]. Previously, we have reported the use of supported manganese(III) porphyrins in the oxidation of organic compounds with NaIO₄ [54–63]. Here, dehydrogenation of 2-substituted imidazolines to 2-imidazoles in the presence of tetraphenylporphyrinatomanganese(III) chloride supported on chloromethylated polystyrene-bound imidazole, [Mn(TPP)Cl@PSI] with NaIO₄ under mechanical stirring (MS) and ultrasonic irradiation (US) conditions is reported (Scheme 1).

2. Experimental

All materials were of commercial reagent grade. All materials were purchased from Merck. Tetraphenylporphyrin was prepared, metallated and supported according to a reported procedure [56,64]. All the 2-substituted imidazolines were synthesized by

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Scheme 1. Oxidation of imidazolines with sodium periodate catalyzed by $[\text{Mn(TPP)Cl@PSI}]$.

the reported procedures [21,22]. ^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 ultrasonic 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 to 40 °C during sonication.

2.1. General procedure for the dehydrogenation of imidazolines with NaIO_4 catalyzed by $[\text{Mn(TPP)Cl@PSI}]$ under MS or US irradiation

A solution of NaIO_4 (2 mmol) in H_2O (10 mL) was added to a mixture of the 2-imidazolines (1 mmol) and $[\text{Mn(TPP)Cl@PSI}]$ (0.012 mmol) in CH_3CN (5 mL), and the reaction mixture was stirred at room temperature or exposed to ultrasonic irradiation. The progress of the reaction was monitored by TLC (eluent: EtOAc/MeOH: 4/1). After the reaction was completed, the catalyst was filtered. Water (30 mL) was added to the filtrates and the corresponding imidazoles were extracted with CH_2Cl_2 (2×20 mL). The solvent was evaporated 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 identities of the products.

3. Results and discussion

3.1. Preparation of polystyrene-bound imidazole

Scheme 2 shows the preparation route for the catalyst. First, chloromethylated polystyrene (cross-linked with 2% divinylben-

zene, 4–5% Cl content, 1.14–1.40 mmol/g Cl) was treated with an appropriate quantity of imidazole in refluxing acetonitrile for 48 h [65]. The PSI was characterized by elemental analysis. The nitrogen content of this resin was 2.06%. According to this value, the degree of imidazole, which was introduced into the polymer composition, was 0.736 mmol/g of support. This shows that only 53–64% of the total chlorines were substituted by imidazole.

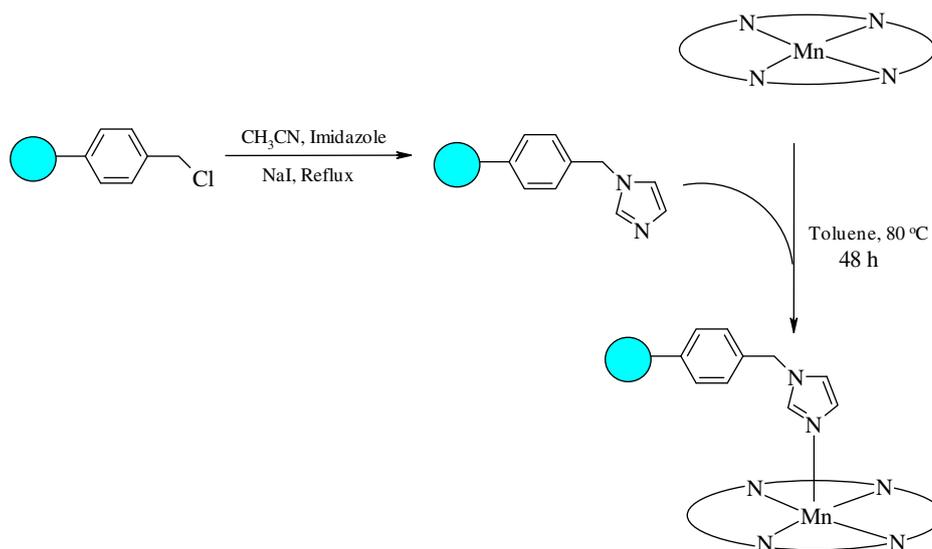
3.2. Preparation and characterization of the catalyst, $[\text{Mn(TPP)Cl@PSI}]$

The supported catalyst was prepared by stirring a suspension of the support (PSI) in a solution of Mn(TPP)Cl in toluene, because of better solvation and swelling of the polymer in it. The free manganese porphyrin was removed by subsequent washing of the catalyst with toluene, dichloromethane and acetone, respectively. The reflectance of the $[\text{Mn(TPP)Cl@PSI}]$ clearly indicated the presence of manganese porphyrin on the polymer surface. The manganese content of the supported catalyst, determined by neutron activation analysis (NAA), showed a value of about 0.024 mmol/g.

In order to ascertain the attachment of the metalloporphyrin on the support, the IR spectra of PSI and $[\text{Mn(TPP)Cl@PSI}]$ were recorded. The sharp C–Cl peak (due to $-\text{CH}_2\text{Cl}$ groups) at 1264 cm^{-1} in the starting polymer was practically omitted or was seen as a weak band after introduction of imidazole and manganese(III) porphyrin on the polymer. The C=N vibration band of imidazole group appeared at 1601 cm^{-1} , which is an indication of the introduction of the catalyst on the support (Fig. 1). The reflectance of the polymer-bound porphyrin resembles the solution counterpart spectrum, with only a slight shift and a Soret band at 478 nm and Q bands at 583 and 618 nm, which clearly indicates the presence of the metalloporphyrin on the surface (Fig. 2). The SEM image of the catalysts showed that the polystyrene has retained its spherical nature and was not altered during the catalyst preparation (Fig. 3).

3.3. Dehydrogenation of imidazolines with NaIO_4 catalyzed by $[\text{Mn(TPP)Cl@PSI}]$ under MS or US irradiation

The successful application of $[\text{Mn(TPP)Cl@PSI}]$ in the oxidation of organic compounds [52,53], prompted us to investigate its catalytic activity in the biomimetic dehydrogenation of 2-imidazoline with NaIO_4 under agitation with magnetic stirring (MS) and under ultrasonic irradiation (US). First, the amount of catalyst was optimized in the dehydrogenation of the 2-phenyl imidazolines



Scheme 2. Preparation route for $[\text{Mn(TPP)Cl@PSI}]$.

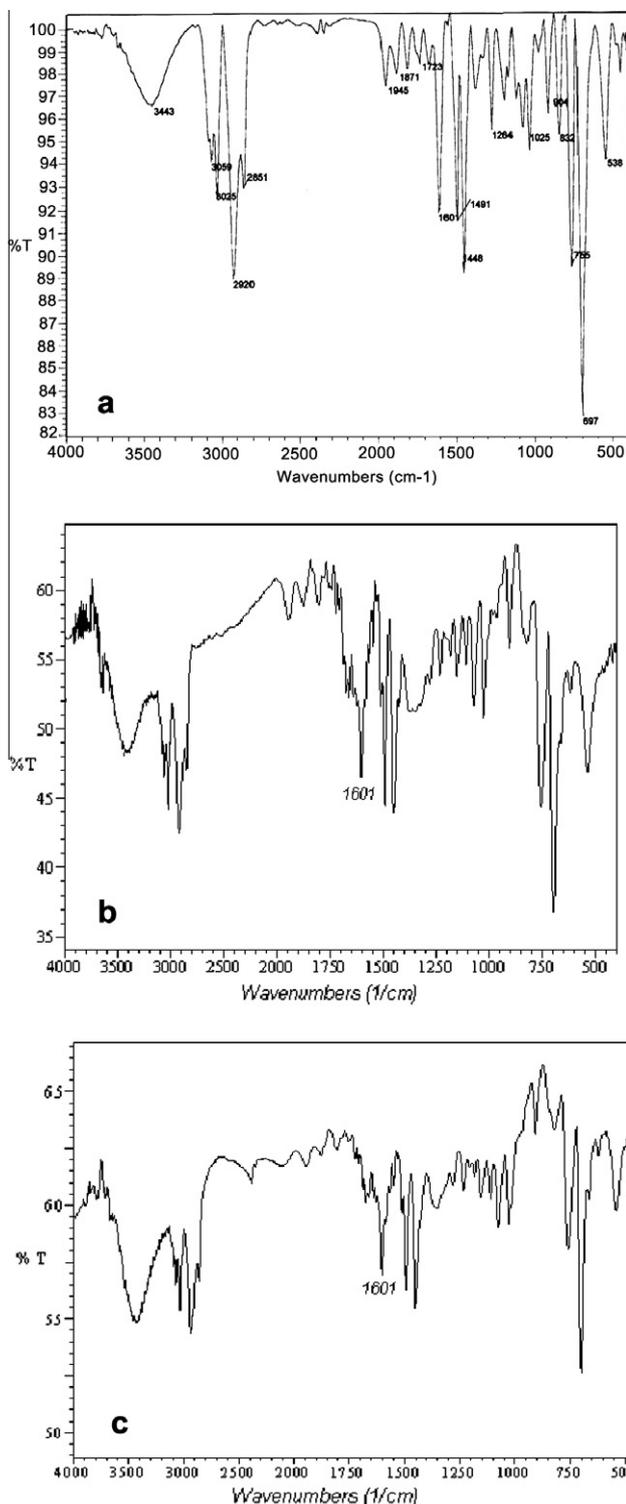


Fig. 1. FTIR spectra of: (a) chloromethylated polystyrene, (b) PSI and (c) [Mn(TPP)Cl@PSI].

with NaIO₄ under MS and US. As shown in Table 1, the highest yield was obtained in the presence of 500 mg (12 μmol) of catalyst. Then, the effect of different solvents was investigated for choosing the reaction media. The results show that a 2:1 mixture of H₂O/CH₃CN is the best solvent for conversion of 2-imidazolines to their corresponding 2-imidazoles (Table 2). Under these conditions a wide range of 2-aryl-2-imidazolines were dehydrogenated to their corresponding imidazoles with NaIO₄ in good to excellent yields

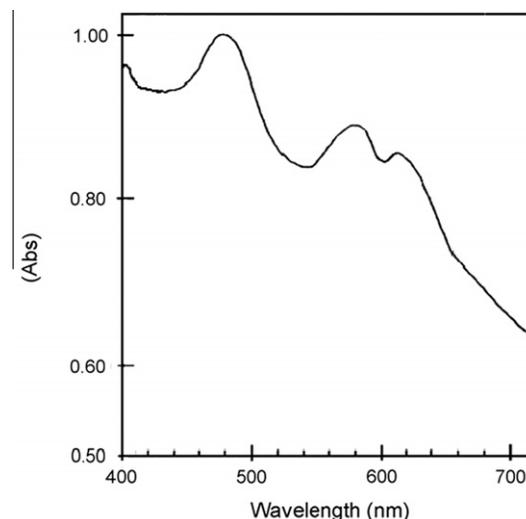


Fig. 2. Diffuse reflectance UV-Vis spectrum of [Mn(TPP)Cl@PSI].

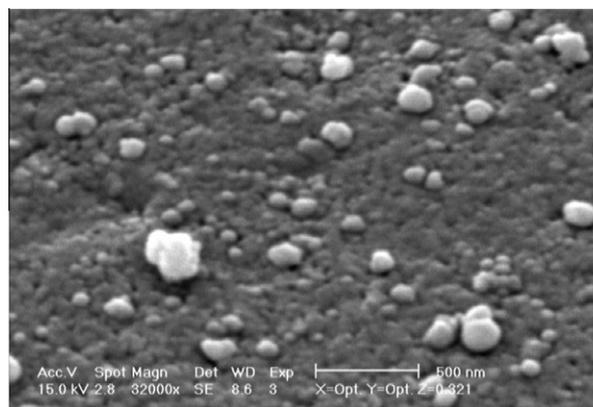


Fig. 3. SEM image of [Mn(TPP)Cl@PSI].

Table 1

Optimization of the catalyst amount in the oxidation of 2-phenyl imidazoline with NaIO₄ under MS and US irradiation.^a

Entry	Catalyst amount (mg)	MS		US	
		Time (h)	Yield (%) ^b	Time (h)	Yield (%) ^b
1	100 (2.4 μmol)	10	45	1	48
2	200 (4.8 μmol)	10	60	1	62
3	300 (7.2 μmol)	10	72	1	73
4	400 (9.6 μmol)	10	77	1	78
5	500 (12.0 μmol)	10	81	1	82
6	600 (14.4 μmol)	10	81	1	82

^a Reaction conditions: 2-imidazoline (1 mmol), catalyst, NaIO₄ (2 mmol), CH₃CN (5 mL), H₂O (10 mL), T = 25 °C for MS and 40 °C for US.

^b Isolated yield.

(entries **1a–h**) under MS at room temperature (Table 3). Some of the reported reagents, such as DMSO [29] and Pd/C [29], were not effective for dehydrogenation of 2-alkylimidazolines. Thus, efficient oxidation of 2-alkylimidazolines to their imidazoles by the [Mn(TPP)Cl@PSI]/NaIO₄ catalytic system is an interesting feature of the presented method (entries **1i–j**). The results show that in the absence of catalyst, only 5–10% of the imidazoles were produced.

In comparison with its homogeneous counterpart [37], [Mn(TPP)Cl@PSI] has a higher catalytic activity in these reactions.

Table 2

The effect of solvent on the dehydrogenation of 2-phenyl imidazolines with NaIO₄ catalyzed by [Mn(TPP)Cl@PSI] under MS and US irradiation.^a

Entry	Solvent	MS		US	
		Time (h)	Yield (%) ^b	Time (h)	Yield (%) ^b
1	CH ₃ CN:H ₂ O (1:2)	10	81	1	82
2	CH ₃ CN:H ₂ O (1:1)	10	70	1	73
3	CH ₃ CN:H ₂ O (2:1)	10	42	1	45
4	CH ₃ COCH ₃ :H ₂ O (1:2)	10	68	1	70
5	CH ₃ OH:H ₂ O (1:2)	10	47	1	50
6	CH ₃ CH ₂ OH:H ₂ O (1:2)	10	51	1	55
7	CHCl ₃ :H ₂ O (1:2)	10	30	1	35
8	CH ₂ Cl ₂ :H ₂ O (1:2)	10	39	1	45
9	CCl ₄ :H ₂ O (1:2)	10	15	1	18

^a Reaction conditions: 2-imidazoline (1 mmol), catalyst (12 μmol), NaIO₄ (2 mmol), solvent. *T* = 25 °C for MS and 40 °C for US.

^b Isolated yield.

The TOFs for this heterogeneous system are higher than those for the homogeneous system. This may be due to the isolation of catalytic active sites (Table 3).

The exact mechanism of the reaction is not clear at present. However, a plausible explanation is that the catalyst is first transformed to the corresponding oxo compound **1** by the oxidant. The oxo compound reacts with imidazoline to afford **2**, which in turn is 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 a hydrogen shift (Scheme 3). It seems that the stabilization of **3** by resonance and also by the electron-withdrawing character of the R group is a good indication for the proposed mechanism. These explanations are also supported by the results in Table 4, in which imidazolines bearing electron-withdrawing groups (entries 5–7) are more reactive in these transformations.

Table 3

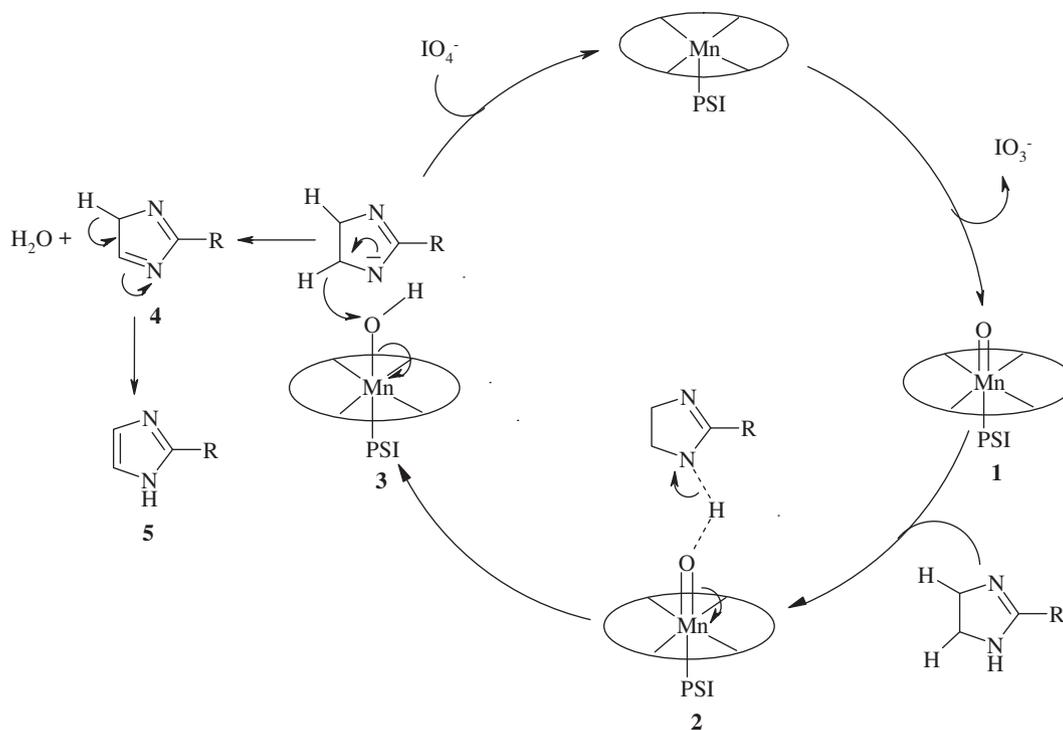
Oxidation of 2-imidazolines with NaIO₄ catalyzed by [Mn(TPP)Cl@PSI] under MS and US irradiation.^a

Row	Imidazoline	Imidazole	MS			US			Homogeneous Mn(TPP)Cl [37] TOF (h ⁻¹)
			Time (h)	Yield (%) ^{b,c}	TOF (h ⁻¹)	Time (h)	Yield (%) ^b	TOF (h ⁻¹)	
1			10	81 (79)	6.75	1	82	68.33	3.36
2			10	72 (71)	6.00	1	80	66.67	2.92
3			10	78 (71)	6.50	1	79	65.83	3.08
4			10	73 (65)	6.08	1	74	61.67	3.00
5			10	92 (84)	7.67	1	94	78.33	3.68
6			10	90 (85)	7.50	1	92	76.67	3.44
7			10	82 (78)	6.83	1	85	70.83	3.24
8			10	70 (68)	5.83	1	78	65.00	2.88
9			10	88 (81)	7.33	1	91	75.83	3.80
10			10	90 (84)	7.50	1	92	76.67	3.68

^a Reaction conditions: 2-imidazoline (1 mmol), catalyst (12 μmol), NaIO₄ (2 mmol), CH₃CN (5 mL), H₂O (10 mL). *T* = 25 °C for MS, 80 °C for reflux conditions and 40 °C for US.

^b Isolated yield.

^c Yields in the parenthesis refer to the reflux conditions after 7 h.



Scheme 3. The proposed mechanism for oxidation of imidazolines with sodium periodate catalyzed by $[\text{Mn}(\text{TPP})\text{Cl}@\text{PSI}]$.

Table 4

The results of $[\text{Mn}(\text{TPP})\text{Cl}@\text{PSI}]$ catalyst recovery and the manganese leached in the oxidation of 2-phenyl imidazoline with sodium periodate under magnetic stirring and ultrasonic irradiation.

Run	Yield (%) ^a		Mn leached (%) ^b	
	MS	US	MS	US
1	81	82	1.5	2
2	79	78	1.0	1.4
3	79	76	0	0.6
4	76	75	0	0
5	75	74	0	0

^a GLC yield based on starting imidazoline.

^b Determined by atomic absorption spectroscopy.

The main effect of ultrasonic irradiation in liquids is the cavitation phenomenon, which involves numerous tiny gas bubbles called cavitation bubbles. The collapse of the bubbles generates high temperature and pressure at the center of the bubbles. These local effects produce a variety of radicals and highly active intermediates, which initiate other secondary chemical reactions in the bulk liquid. These radicals can react with contaminants to form by-products.

The most successful applications of ultrasound have been found in the field of heterogeneous chemistry involving solids and metals. This is due to the mechanical impact of ultrasound on solid surfaces. The mechanical effects of ultrasound offer the opportunity to overcome the following types of problem associated with conventional solid/metal reactions: the break-up of the surface structure allows penetration of reactants and/or release of materials from the surface, degradation of large solid particles occurs due to shear forces induced by shock waves, microstreaming leads to a reduction of particle size and an increase of the surface area, and accelerated motion of suspended particles leads to better mass transfer [66].

When the same reactions were carried out under US irradiation, the reaction times were reduced to 1 h (Table 3). It seems that the effect of ultrasonic irradiation on the catalytic activity enhance-

ment may be due to the break-up of the agglomerates during the sonication process. A part of the ultrasonic irradiation effect is due to this phenomenon. To stress this point, the catalytic activity of a sonicated sample catalyst was studied in the oxidation of 2-phenyl imidazoline under mechanical stirring. It was found that the reaction time reduced from 10 to 6 h for the complete dehydrogenation of 2-phenyl imidazoline 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 of hot spots are other reasons for catalytic enhancement by ultrasonic irradiation. On the other hand, when the oxidation reactions were carried out under reflux conditions, the reaction times decreased, which confirms the effect of producing the hot spots on the enhancement of catalytic activity (Table 3).

3.4. Catalyst reusability

The stability of the supported catalyst was monitored using multiple sequential dehydrogenations of 2-phenyl imidazoline with sodium periodate under agitation with magnetic stirring or under ultrasonic irradiation. For each of the repeated reactions, the catalyst was recovered, washed exhaustively with water, methanol, acetonitrile and diethyl ether, and then dried before being used with fresh 2-phenyl imidazolines and sodium periodate. In both cases, the catalyst was consecutively reused five times without any detectable catalyst leaching or any significant loss of its activity (Table 4). Also, the catalytic behavior of the separated liquid was tested by addition of fresh 2-phenyl imidazoline and NaIO_4 to the filtrates after each run. Execution of the oxidation reaction under the same reaction conditions, as for the catalyst, showed that the obtained results were the same as the blank experiments.

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