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Manganese(II)-Induced Oxidation of Limonene by Dioxygen

Anna Szczepanik · Andrzej Sobkowiak

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Abstract Manganese(II) complexes $\{[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}\}$ in acetonitrile activate dioxygen for the oxidation of limonene to produce mainly carvone, carveol, limonene oxide, and perillaldehyde. The reaction efficiencies after 24 h reaction time are approximately 5-times higher than those obtained for analogous iron(II) complexes. However, the 5 h long induction period is observed for the common conditions of the reaction. The simultaneous presence of the catalyst, dioxygen and the substrate is necessary for the active species to be formed. When *t*-BuOOH is present in the reaction mixture, the induction period does not appear. In contrast, the replacement of *t*-BuOOH by HOOH completely inhibits the reaction. We have proposed a putative mechanism in which a manganese(IV)–hydroperoxo adduct with incorporated substrate is formed during the induction period and it becomes an active catalyst for limonene oxidation.

Keywords Dioxygen activation · Limonene oxidation · Manganese–bipyridine complexes

1 Introduction

Selective oxidation of limonene is one of the most important technological processes because it can lead to production of important compounds, which are used in flavor and fragrance industry [1]. In the recent paper [2] we have reported that coordinately unsaturated $[Fe^{II}(bpy)_2{}^{2+}]_{MeCN}$ complex in acetonitrile activates dioxygen for the oxygenation of limonene. However, the process gives a mixture of products: carvone, carveol, limonene oxide, and perillaldehyde. When $[Fe^{III}(bpy)_2{}^{3+}]_{MeCN}$ was applied as a catalyst, it was reduced by limonene to $[Fe^{II}(bpy)_2{}^{2+}]_{MeCN}$, which appears to be an effective catalyst.

Manganese(III) complexes were used previously to catalyze oxidation/epoxidation of limonene. Mn(III) porphynins both polymer-supported and in homogeneous system, activate HOOH, $PhIO_4$, and NaOCl for selective limonene epoxidation [3–10]. Dioxygen in the presence of an aldehyde (benzaldehyde or pivalaldehyde) was also applied as an oxidant [11–14].

In the previous paper [15] we have found that several manganese(III) complexes in combination with *tert*-butyl hydroperoxide activate dioxygen to oxygenate cyclohexene to its ketone, alcohol, and epoxide. The product profiles depend on the ligand and solvent matrix. With picolinate, 2,2′-bipyridine, and triphenylphosphine oxide as ligands in py/HOAc, the ketone is a dominant product whereas Schiff base complexes produce ketone, alcohol, and epoxide in almost equal yields. However, in acetonitrile ketone is a dominant product for all the complexes used. The effect of several factors (type of oxidant, catalyst, solvent, and time) on the selectivity of limonene oxidation process has been recently reported [16]. Taking into account the above findings and a fact, which was established in our previous works [17–20], that the oxidation state of the catalyst applied varies during the oxidation process depending on reaction conditions we have applied manganese(II)–2,2′-bipiridine complex to catalyze limonene oxidation by dioxygen. Manganese(II) complexes have not yet been used to catalyze dioxygen activation.

A. Szczepanik · A. Sobkowiak (✉)
Faculty of Chemistry, Rzeszów University of Technology,
35-959 Rzeszów, Poland
e-mail: asobkow@prz.rzeszow.pl

2 Experimental

2.1 Equipment

The reaction products were separated and identified with a Hewlett-Packard 4890A Series gas chromatograph equipped with an HP-1 capillary column (cross-linked methylsilicone-gum phase, 30 m × 0.53 mm i.d.).

A three-electrode potentiostat (Princeton Applied Research Model 273A) was used to record the voltammograms. The experiments were conducted in a 15-mL electrochemical cell with provision to control the presence of dioxygen with an argon-purge system. The working electrode was a Bioanalytical Systems glassy-carbon (area 0.09 cm^2) inlay, the auxiliary electrode—a platinum wire, and the reference electrode—an Ag/AgCl wire in an aqueous tetramethylammonium chloride solution that was adjusted to give a potential of 0.00 V versus SCE. The latter was contained in a Pyrex tube with a cracked soft-glass tip, which was placed inside a Luggin capillary [21].

The UV–vis spectrophotometric measurements were made with a Hewlett-Packard Model HP-8453 diode array rapid scan spectrophotometer.

2.2 Chemicals and Reagents

The reagents for the investigations and syntheses were as purchased and were used without further purification. The solvent for all of the experiments was acetonitrile (99.93+%, HPLC grade) purchased from Aldrich. High-purity argon gas was used to deaerate the solutions. Tetraethylammonium perchlorate (TEAP, GFS Chemicals) was dried in vacuo over $CaSO_4$ for 24 h prior to use. Manganese(II) perchlorate, $Mn(ClO_4)_2 \cdot 6H_2O$, 99%; manganese(III) acetate dehydrate, $(CH_3COO)_3Mn \cdot 2H_2O$, 97%; and the organic substances: (*R*)-(+)-limonene, 97%; (−)-carveol, mixture of isomers, 97%; (*R*)-(−)-carvone, 98%; (+)-limonene oxide, mixture of *cis* and *trans*, 97%; (*S*)-(−)-perillyl alcohol, 96%; (*S*)-(−)-perillaldehyde, 92%; 2,2′-bipyridine (bpy, 99+%); biphenyl (PhPh, 99+%) as well as hydrogen peroxide (30%, w/w solution in H_2O); *tert*-butyl hydroperoxide (5.0–6.0 M solution in decane) and *tert*-butyl hydroperoxide (70% solution in H_2O) were obtained from Aldrich.

2.3 Methods

The appropriate amounts of metal salt and ligand were combined in acetonitrile followed by the addition of the substrate (1 M) (total volume = 5 mL) in the reaction cell (25 cm^3 vial with cut-out cap and Teflon-faced septum). The solution was saturated with dioxygen (O_2, 1 atm) or air (O_2, 0.2 atm) or high-purity argon gas (O_2, 0 atm). In the case of dioxygen and air the appropriate gas saturated with acetonitrile was passed over the solution to maintain a constant solution concentration of dioxygen. The reactions were allowed to proceed for 24 h with constant stirring at room temperature (23 ± 1 °C). After the experiment samples of the reaction solution were injected into a capillary-column gas chromatograph for analysis. Authentic samples were always used to confirm product identifications and to produce standard curves for quantitative assays of the product species. Biphenyl (10 mM) was used as an internal standard. All experiments were done in triplicate. The presented values of concentration are the mean values of three independent experiments.

3 Results

As it was mentioned in the introduction there are no reports that manganese(II) complexes activate dioxygen for the oxygenation of organic substrates. To our surprise, after 24 h reaction time, we have found that the oxidation of limonene by dioxygen catalyzed by $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ gives carvone, carveol, limonene oxide, perillaldehyde, and perillyl alcohol as products (Scheme 1). The observed product yields were even higher in comparison with the process catalyzed by iron(II)–2,2′-bipiridine complex [2] (Table 1). At least two more products, exhibiting retention times similar to those of carveol isomers, have been obtained in assessed amounts exceeding the yield of perillyl alcohol. However, the similarity between their mass spectra and that of carveol has made their identification impossible (they are probably terpineol isomers). The product profiles after 24-h reaction time indicate that the 0.25 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}/O_2(1\ atm)$/1 M limonene combination is the most reactive—532 product/catalyst turnovers. In the absence of the catalyst as well as the

Scheme 1 Products of limonene oxidation by dioxygen catalyzed by $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$

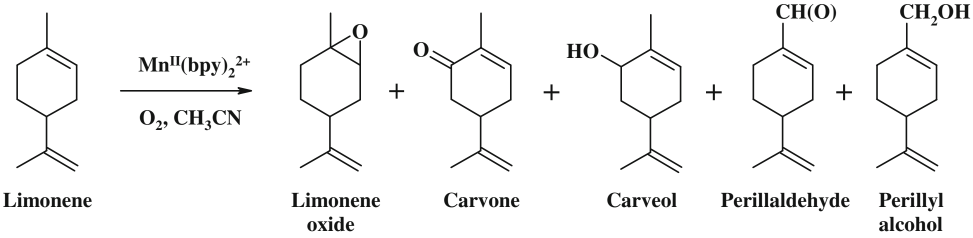


Table 1 Oxidation of limonene by dioxygen catalyzed by manganese complex $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ in acetonitrile

Substrate concentration (M)	Catalyst concentration (mM)	Oxidant	Carvone	Carveol	Limonene oxide	Perillaldehyde	Perillyl alcohol	TON
1	1	O_2	68.8 (33)	23.3 (11)	84.7 (40)	26.7 (13)	5.7 (3)	209
1	1	Air	7.6 (26)	8.0 (27)	10.7 (37)	2.1 (7)	0.9 (3)	29
1	0.25	O_2	45.5 (34)	20.2 (15)	45.0 (34)	18.7 (14)	3.6 (3)	532
1	0.5	O_2	61.4 (32)	25.4 (13)	79.6 (41)	23.4 (12)	5.1 (3)	390
1	2	O_2	67.6 (36)	21.8 (12)	68.8 (37)	23.1 (12)	4.8 (3)	93
1	3	O_2	67.7 (38)	20.3 (11)	60.4 (34)	24.0 (14)	4.2 (2)	59
1	4	O_2	66.2 (38)	21.3 (12)	62.1 (35)	22.6 (13)	3.7 (2)	44
1	5	O_2	66.9 (39)	21.6 (13)	57.7 (34)	21.5 (13)	3.5 (2)	40
1	6	O_2	Traces	Traces	Traces	0.0	0.0	–
1	10	O_2	0.0	Traces	Traces	0.0	0.0	–
0.5	1	O_2	31.7 (42)	7.6 (10)	21.8 (29)	12.1 (16)	2.1 (3)	75
2	1	O_2	94.1 (35)	47.4 (18)	102.0 (38)	20.7 (8)	6.2 (2)	270

Reaction time 24 h

Product concentrations are in mM. Selectivities toward particular products were count as a concentration of a product divided by a sum of the concentrations of the main products (in %) and are in brackets. By-products were not taken into account

TON—product molecules per catalyst molecule

ligand (only Mn-solvated ions are present in the reaction mixture) no products are formed. The use of air (O_2, 0.2 atm) instead of dioxygen (O_2, 1 atm) causes ca. sevenfold drop in the reaction rate (29 vs. 209 TON for air and dioxygen, respectively), whereas the 1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$/1 M limonene combination under argon atmosphere gives no products after 24 h reaction time.

As it can be seen from the results presented in Table 1 the increase of the substrate concentration causes almost proportional increase of the reaction yield. The catalyst concentration does not exert an effect on the overall yield of the reaction for its concentration in the range of 0.5–5 mM. For the catalyst concentration equal or higher than 6 mM at 1 M substrate concentration, the reaction is completely inhibited (Fig. 1). Similarly, no product formation is observed after 24 h reaction time at 0.5 M substrate concentration with 3 as well as 5 mM of $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$, which indicates that the reaction suppression is caused by the catalyst/substrate ratio and not by the excessively high initial catalyst concentration in the reaction solution.

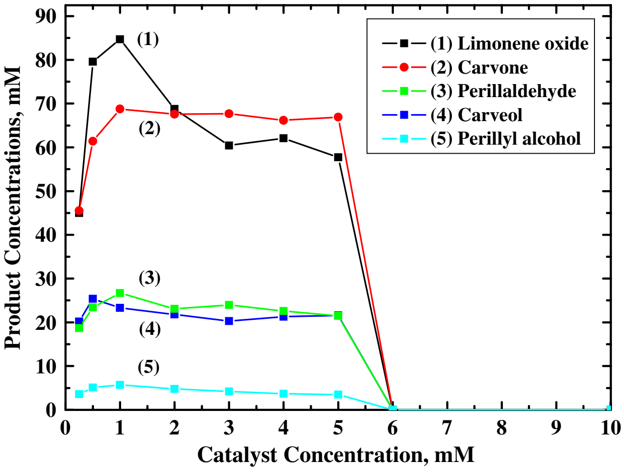


Fig. 1 Effect of catalyst concentration on the product concentrations (**1**) limonene oxide, (**2**) carvone, (**3**) perillaldehyde, (**4**) carveol, and (**5**) perillyl alcohol for the oxidation of 1 M limonene by dioxygen catalyzed by 1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ in acetonitrile. Reaction time 24 h

We have noticed that the reaction mixture was gradually changing the color from colorless to brownish. After approximately 4 h there was no further color change of the solution. Therefore, time-dependent product profiles were measured (Fig. 2). These profiles exhibit sigmoidal shape which is characteristic of autocatalytic reactions. About 5 h long induction period appears before rapid increase of the reaction rate. Using air instead of dioxygen strongly lengthens the induction period, which in this case accounts for 9 h. The induction period is also observed for the same system, but after replacement of limonene by cyclohexene. No oxidation products were detected in the case of cyclohexane.

To establish whether the long interaction between the catalyst and dioxygen is necessary to initiate the limonene oxidation process, 1 mM solution of the catalyst was prepared in acetonitrile and was kept under either oxygen or argon atmosphere for 24 h prior to addition of the substrate. There was no change in the color for the samples containing the catalyst kept either under dioxygen or argon,

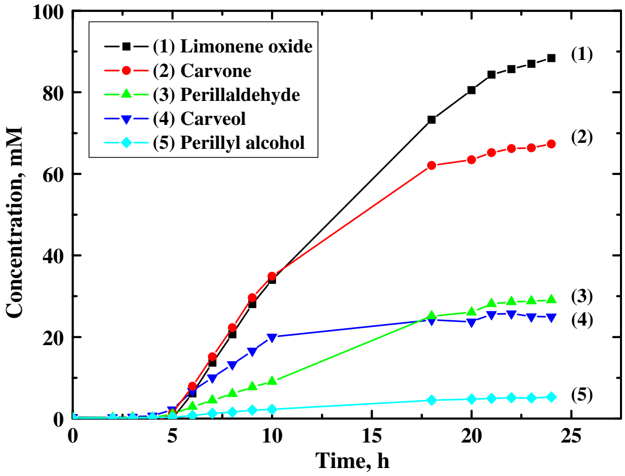


Fig. 2 Dependence of product concentrations (**1**) limonene oxide, (**2**) carvone, (**3**) perillaldehyde, (**4**) carveol, and (**5**) perillyl alcohol on time for the oxidation of 1 M limonene by dioxygen catalyzed by 1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ in acetonitrile

which indicates that the catalyst and dioxygen do not interact in the absence of the substrate. After addition of limonene the reaction was allowed to proceed for the next 24 h and then products were analyzed. The yields of the products were similar to these reported in Table 1 for analogous concentrations of the catalyst and the substrate. It is characteristic that when the mixture of 1 mM of catalyst $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ and 1 M of limonene was kept under argon for 24 h and then dioxygen was introduced to the system no products were observed after 24 h.

In some experiments either 50 mM HOOH or 50 mM *t*-BuOOH was added to the reaction mixture at the beginning of the experiment run, keeping all other variables as previously {1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}/O_2$(1 atm)/1 M limonene}. In the presence of *t*-BuOOH the reaction starts immediately reaching after 24 h the yield of the products only slightly lower than indicated in Table 1 for analogous conditions. The decrease in *t*-BuOOH concentration to 10 mM also eliminates the induction period. The addition of HOOH causes no product formation. Clearly, the interaction of $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ with *t*-BuOOH causes the formation of an active catalyst, whereas hydrogen peroxide deactivates Mn-complex and prevents formation of an active catalyst. The induction period can be also eliminated when $(bpy)_2{}^{2+}Mn^{II}(OAc)$ complex (prepared in situ by mixing 2,2′-bipyridine and $Mn(OAc)_3$ in acetonitrile in 2 to 1 molar ratio) was used as catalyst.

To exclude the role of limonene hydroperoxide in the initiation process, 2 mM solution of $(bpy)_2{}^{2+}Mn^{II}$, and 2 M solution of limonene, both in acetonitrile, have been kept separately under oxygen atmosphere overnight. After mixing the same volumes of the solutions, the induction period was also observed.

Cyclic-voltammetric measurements of the investigated reaction mixture after 24 h exhibit a broad plateau in the first cathodic scan at the potential region from +0.2 to −0.4 V (vs. SCE) and a shoulder at +1.2 V in the first anodic scan. These phenomena are not observed in the voltammograms registered immediately after mixing the reagents (Fig. 3). The addition of 50 mM of *t*-BuOOH to 3 mM solution of the catalyst in the presence of 1 M limonene causes the appearance of the analogous shoulders on CV-curves whereas the addition of hydrogen peroxide at the same experimental condition does not (Fig. 4). The sharp cathodic peak (curve c) observed in the last case is due to concerted reduction of manganese and dioxygen from catalytic decomposition of hydrogen peroxide [22].

This observation was confirmed by UV–vis measurements. As it is shown in Fig. 5 the addition of 5 mM *t*-BuOOH to 0.1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ in acetonitrile causes the increase of the absorption band at $\lambda = 240$ nm and the decrease of the band at $\lambda = 295$ nm. Two isosbestic points are visible in the spectra. These phenomena do not occur when *t*-BuOOH is replaced by HOOH at the same experimental conditions. It is characteristic that in the presence of limonene and dioxygen the same decrease of the band at $\lambda = 295$ nm occurs within the reaction

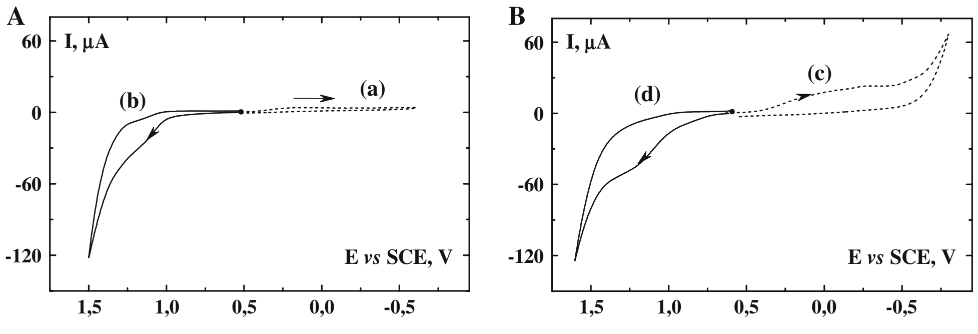


Fig. 3 Cyclic-voltammograms in acetonitrile [0.1 M $(Et_4\,N)ClO_4$] for (**A**) the mixture of 3 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ and 1 M limonene immediately after mixing—(**a**) cathodic scan was recorded first, (**b**) anodic scan was recorded first; (**B**) the same as (**A**) after 24 h under O_2 atmosphere—(**c**) cathodic scan was recorded first, (**d**) anodic scan was recorded first. Scan rate, 0.1 V s^{-1}, GCE (0.09 cm^2); SCE versus NHE, +0.242 V

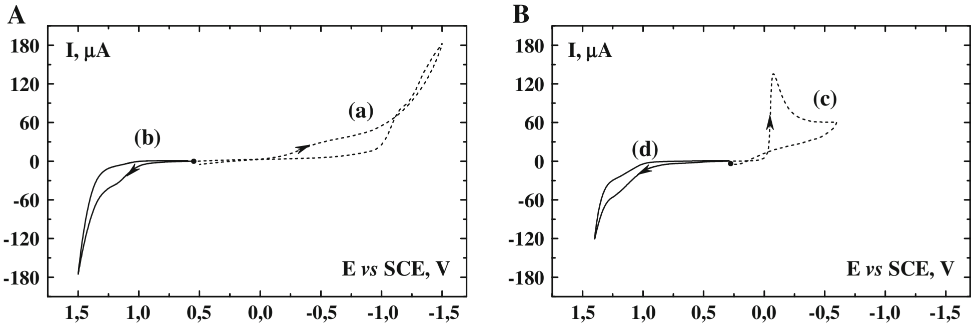


Fig. 4 Cyclic-voltammograms in acetonitrile [0.1 M $(Et_4N)ClO_4$] for (**A**) the mixture of 3 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$, 1 M limonene and 50 mM *t*-BuOOH immediately after mixing—(**a**) cathodic scan was recorded first—(**b**) anodic scan was recorded first; (**B**) the same as (**A**), but 50 mM HOOH was added instead of *t*-BuOOH—(**c**) cathodic scan was recorded first, (**d**) anodic scan was recorded first. Scan rate, 0.1 V s^{-1}, GCE (0.09 cm^2); SCE versus NHE, +0.242 V

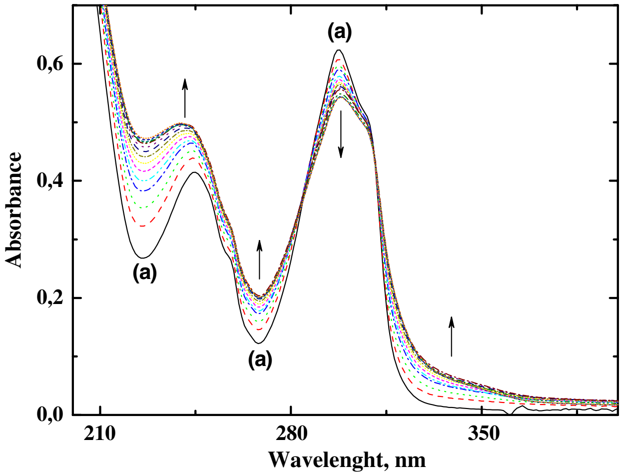


Fig. 5 UV–vis spectra of 0.1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ in acetonitrile (curve a) and after addition of 5 mM *t*-BuOOH—each spectrum was registered every 2 min up to 30 min

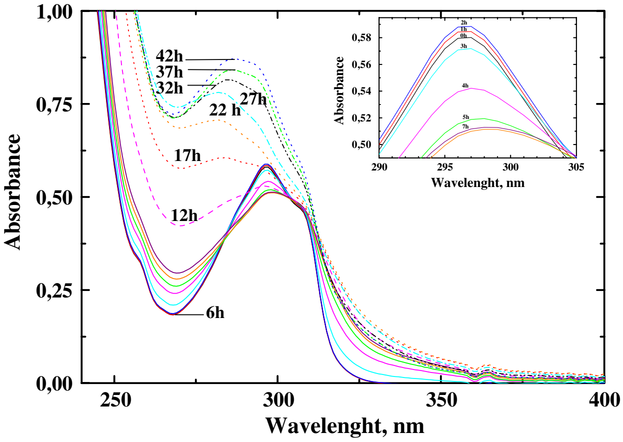


Fig. 6 UV–vis spectra on time for the mixture of 0.1 mM $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ and 0.1 M limonene under dioxygen atmosphere in acetonitrile. Inset—the spectra taken during the initial period of the oxidation reaction

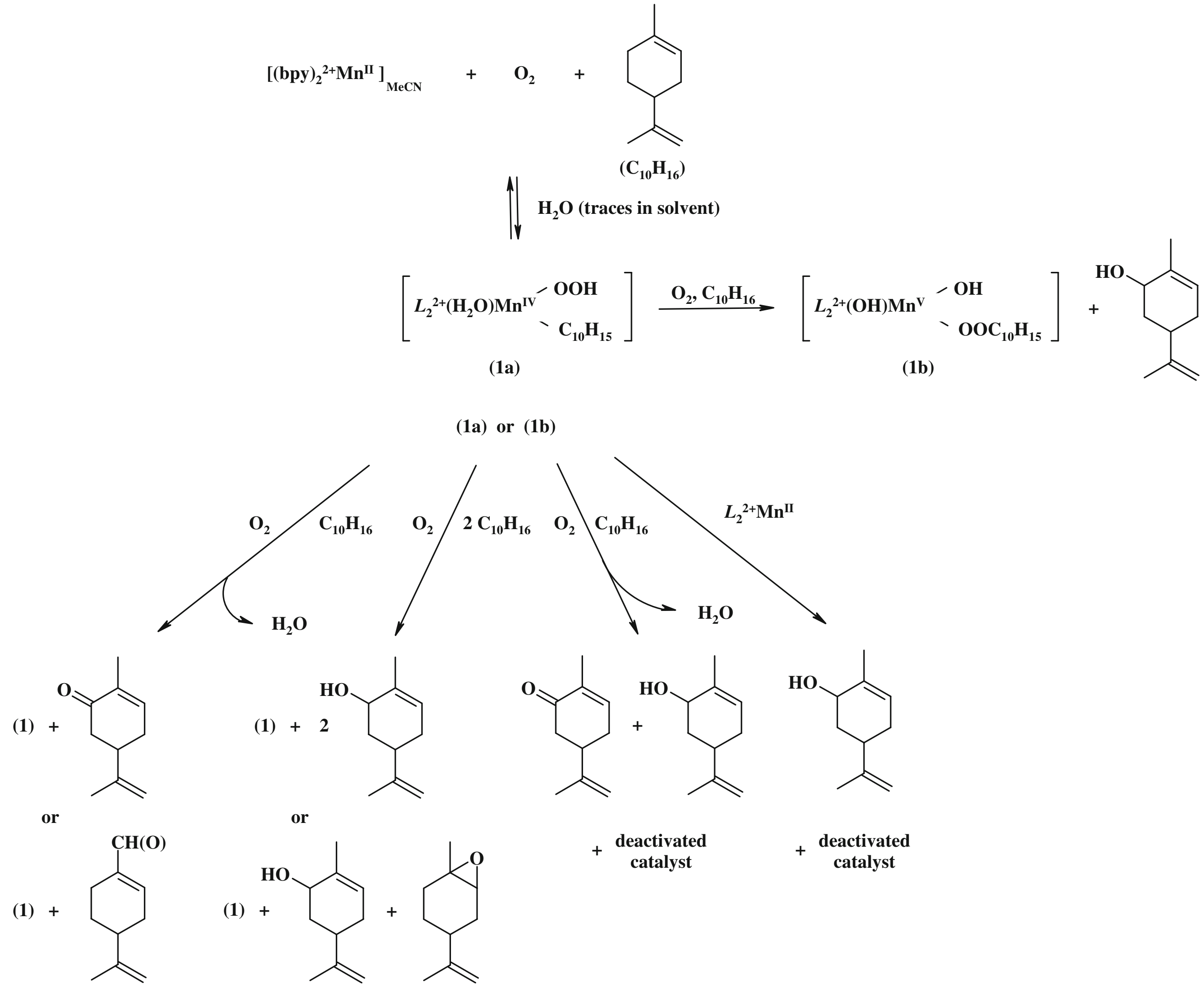
induction time (approximately 6 h—Fig. 6, inset). After that time a broad band at approximately 290 nm is growing with further reaction time.

4 Discussion

The presented results indicate that the combination of $[Mn^{II}(bpy)_2{}^{2+}]_{MeCN}$ and dioxygen causes the oxidation of limonene to give four major products: limonene oxide, carvone, carveol and perillaldehyde (Scheme 1 and Table 1). Much smaller amounts of perillyl alcohol were also detected during the oxidation process. The product profiles presented in Fig. 2 show that the 5 h long induction period is observed during the reaction course. After approximately 24 h plateaus are started to form. This differs much from the results of the oxidation of limonene by dioxygen catalyzed by iron analogue $[Fe^{II}(bpy)_2{}^{2+}]_{MeCN}$ of the catalyst applied in the present paper. In the case no induction period was present and the yields of the products formed increased over the same period of time [2]. The analysis of the product amounts after 24 h reaction time show that the reaction rate is roughly first order versus both dioxygen and the substrate concentration and does not depend on the catalyst concentration within its activity range (0.25–5 mM for 1 M substrate). However, it is interesting to notice that for higher catalyst concentrations, or to be more precise, for higher catalyst to substrate concentration ratio, the catalyst becomes inactive. Moreover, when the combination of the catalyst and the substrate is kept under argon atmosphere, the catalyst is deactivated.

From the set of experiments described in the previous section clearly follows that the combination of the catalyst, dioxygen and the substrate is necessary to enable formation of an active species, which carries on the oxidation process. The formation of the species is observed in cyclic-voltammetric curves (Fig. 3) and in UV–vis spectra (Fig. 6). The same species is also formed immediately after mixing the catalyst, the substrate and *t*-BuOOH. Similar phenomena were observed in CV-curves (Fig. 4A) and UV–vis spectra (Fig. 5), and the oxidation process, when initiated with *t*-BuOOH, occur in the presence of dioxygen without an induction period. This makes the system analogous to that described in our previous paper [15], in which the activation of dioxygen by manganese(III) complexes/*t*-BuOOH for the oxygenation of cyclohexene was described. Based on the results presented in this paper as well as taking into account that the *L*Mn–O–O–H moiety is postulated as responsible for the oxidation/epoxidation of olefins [23–27] a tentative mechanism can be proposed for the reactions occurred in the discussed system (Scheme 2).

Thus, the combination of the catalyst, dioxygen and the substrate gives the reactive species (**1a**), which becomes a catalyst for dioxygen activation. The structure of the proposed species is in agreement with that used in the mechanism of iron(II, III)-catalyzed activation of dioxygen for N-dealkylation of N-substituted anilines [20] and the oxidation of limonene [2]. However, the existence of manganese–carbon bond is very rare [28, 29]. Therefore, the modification of the reactive species regarding the results published previously [15, 30] has been proposed. The species (**1b**) catalyzes also the activation of dioxygen and explains the products formation in a similar way as species (**1a**). Note that the reactive intermediates are within square brackets because their formulations are hypothetical. From the presented results it follows that the formation of adequate concentrations of the species requires a long period of time (an induction period). The reactive species can be also easily decomposed to inactive form of manganese complexes. The reaction environment favors the formation of different mixed valence oxo bridged complexes [29–32]. Clearly, the system is very sensitive to the catalyst/dioxygen/substrate ratio.



Scheme 2 Tentative mechanism of $[\mathrm{Mn^{II}(bpy)_2}^{2+}]_{\mathrm{MeCN}}$-induced activation of dioxygen for the oxidation of limonene

From the simple kinetic considerations follows that for the reaction presented in Scheme 2 the rate of products' formation should be first order with respect to dioxygen, substrate and also the catalyst concentration. The discussion on the catalyst behavior presented above can explain that it cannot obey the kinetic law. The observed independence of the reaction rate from the catalyst concentration in the limited range and then complete loss of its catalytic activity can be attributed to the instability of the catalyst.

The use of hydrogen peroxide as an oxidant and also as an activator (in similar way like *t*-BuOOH) was unsuccessful. Taking into account the presented results it is obvious that HOOH causes the destruction of the catalyst and evolution of dioxygen (vide Fig. 4B). Assuming that the reaction suppression effect may occur due to water introduction to the investigated system (note that water solution of HOOH and decane solution of *t*-BuOOH were used), some experiments using aqueous *t*-BuOOH solution were performed. The results obtained proved to be the same as for *t*-BuOOH in decane solution. Moreover, some trials with addition of water itself were carried out. Their results confirmed that such amounts of water, which were introduced to the system together with HOOH do not influence reaction yield. Five-times higher water concentration in the reaction solution causes a decrease in the reaction yield, which in this case accounts for approximately 85% of the reaction performed saving typical conditions.

The presented results indicate that Mn(II)-catalyzed activation of dioxygen for oxidation of limonene is similar to that catalyzed by Fe(II) [2]. The product yields after 24 h and consequently TONs in the current system are approximately 5-times higher. However, the system is also not selective but produces four major products, which are of interest for flavor and fragrance industry.

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