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Iron Hydroxides—New Catalysts for Oxidation Reactions in Aqueous Solutions

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Abstract—The catalytic decomposition of H_2O_2 in the presence of SiO_2-supported amorphous and colloidal FeO(OH) is studied. An inactive Fe(IV) compound and a Fe(III) peroxo complex are found in the hydroxide composition. Possible mechanisms are discussed for H_2O_2 decomposition involving no free radicals. It is shown that lower hydrocarbons are oxidized in the systems, and their presence changes both the kinetics of H_2O_2 decomposition and the behavior of Fe(IV) and the peroxo complex.

INTRODUCTION

Iron hydroxides are widely used in the laboratory and in chemical technology as adsorbents and collectors of minor amounts of various ions and their compounds. Therefore, the mechanism of FeO(OH) formation and its chemical and physical properties have been studied in more detail compared to the properties of other hydroxides [1]. However, unlike iron oxides, iron hydroxides were not used or studied as catalysts. Iron is one of the most abundant elements in nature and enters the composition of many heme and non-heme enzymes, which have been studied for many years. Iron complexes with different organic ligands are usually used to model various iron-containing monooxygenases, although the syntheses of ligands and complexes are very difficult [2]. Therefore, a search for simple systems capable of catalyzing redox reactions in aqueous solutions is of interest.

This paper describes the catalytic properties of iron hydroxy compounds in the decomposition of H_2O_2 and the oxidation of lower hydrocarbons (methane, ethane, and ethylene) by hydrogen peroxide in aqueous solutions under mild conditions.

EXPERIMENTAL

$Fe(NO_3)_3 \cdot 9H_2O$, $FeCl_3 \cdot 6H_2O$, $Fe_2(SO_4)_3$, $Na_4P_2O_7$, NaH_2PO_4, bipyridyl (bpy), and β-naphthol were without additional purification. A 33% solution of H_2O_2, water-soluble starch for iodometry, and silica gel (KSKG trademark, $S_{sp} = 300$ m^2/g) were used.

$NaFe_3(OH)_6(SO_4)_2$ [3], $Fe_4(OH)_{10}(SO_4)$ [4], amorphous FeO(OH) [5], and α- and γ-FeO(OH) [6, 7] were synthesized by known procedures.

The concentration of H_2O_2 was found by the reaction with Ti(IV) [8], and the concentration of HCOOH was determined by the reaction with chromotropic acid [9].

The kinetics of H_2O_2 decomposition was studied at 298 K by the rate of dioxygen evolution in a volumetric setup or by H_2O_2 consumption. Methane, ethane, and ethylene were oxidized under the conditions of uninterrupted bubbling of the corresponding gas through the reaction mixture or in a volumetric setup after purging the reactor for 20 min. The values of pH of the reaction mixtures were adjusted to a desired level by adding HNO_3 or NaOH.

An I-135 pH-meter (Georgia) and a UV 300 Shimadzu spectrophotometer (Japan) were used.

Catalyst preparation. Water (50 ml) was added to powdered SiO_2 (5 g), and the required pH was achieved by adding a solution of NaOH (1 mol/l) along with rigorous stirring. Then, a solution (3.2 ml) of iron nitrate (0.55 mol/l) and NaOH were added in drops alternately along with stirring and maintaining an unchanged pH value. Iron ions are hydrolyzed at pH > 2. Hence, under the synthesis conditions, they almost completely transformed into the hydroxide chemically fixed on the support surface. The precipitate was filtered, repeatedly washed with water on a filter, and dried first at 383 K and then at 530 K for 40 min. The prepared catalyst contained 2 wt % iron.

Colloidal catalysts stabilized by 0.5% starch were prepared by the procedure described in [10] (procedure 3).

RESULTS AND DISCUSSION

Iron hydroxides can easily be prepared in different modifications. To check for possible differences in the catalytic properties of these samples, we tested the bulk, colloidal, and SiO_2-supported iron hydroxides in H_2O_2 decomposition.

Catalytic Properties of FeO(OH)/SiO$_2$

When FeO(OH) is hydrolytically precipitated on the silica surface, the catalytic properties of resulting samples depend on the nature of the iron salt anions and the pH of the synthesis. Therefore, we prepared the catalysts at very different pH using iron(III) nitrate, chloride, and sulfate and tested them in the decomposition of H_2O_2. The results are presented in the table, which shows that the catalytic properties of supported FeO(OH) slightly depend on these parameters. Only samples obtained from sulfate exhibited a lower activity when they are synthesized in acidic solutions. This is understandable, recalling that, of the three anions presented above, sulfates form the strongest complexes with iron and hence, a higher pH value is required for their hydrolysis. The catalysts containing 2 and 5 wt % iron had almost the same activity.

Reaction rate under different conditions. The addition of H_2O_2 to an aqueous suspension of the catalyst acidifies the reaction mixture and then, during the decomposition of H_2O_2, alkalizes it. However, these changes are within 0.2–0.3 pH units, which are much smaller than the changes in pH observed for the copper-containing catalysts [11]. To determine the reaction rate constant as a function of pH in the reaction, the mixture was brought to the required value after each addition of H_2O_2. The reaction rate was maximal in a very narrow pH interval of 7.2–7.4 (Fig. 1, curve *1*).

The anamorphoses of the kinetic curves of H_2O_2 consumption in the semilogarithmic coordinates fit the straight line well, indicating the first order of the reaction. The first orders were also found for the concentration of H_2O_2 in the interval from 0.02 to 1.0 mol/l (at a constant weighed sample of the catalyst of 0.5 g and pH 7.2) and for the weighed sample of the catalyst from 0.1 to 0.5 g (at the same pH and H_2O_2 concentration of 0.5 mol/l).

Effects of different additives on the reaction rate. When complex-forming ligands (bpy, phosphate, and pyrophosphate) were added to the reaction mixture containing 0.5 g of the catalyst in 10 ml of H_2O_2 (0.1 mol/l) at pH 7.2), the reaction rate (by contrast to that on $Cu(OH)_2/SiO_2$ [11]) decreased by a factor of three to five, depending on the concentration of the additive, in the 10^{-3}–10^{-2} mol/l interval. Therefore, it was impossible to use buffer solutions in the study of these systems.

The shape of kinetic curves did not change to the *S*-like shape in the presence of β-naphthol ($(2–5) \times 10^{-4}$ mol/l), which is a trap of free radicals in H_2O_2 decomposition [12]. This indicates the absence of a considerable contribution of the radical mechanism of H_2O_2 decomposition in the presence of FeO(OH). Lesina *et al.* [13] came to the same conclusion using *para*-nitrosodimethylaniline as a trap. In this case, the reaction probably occurred via the formation of Fe(IV). To check this assumption, the influence of ascorbic acid additives was used [12]. We found that, in the presence of ascorbic acid ($(1–10) \times 10^{-3}$ mol/l), the reaction rate decreased by three to five times for the reaction mixture containing 0.5 g of the catalyst in 10 ml of H_2O_2 (0.5 mol/l) at pH 7.2. However, this retardation can be due to the formation of inactive iron complexes with the ascorbate ion as in the case of other complex-forming additives described above. Therefore, we studied changes in the concentration of ascorbic acid in the reaction mixtures with different compositions. We sampled aliquots at certain time intervals, centrifuged them, and measured the concentration of ascorbic acid using its absorption maximum at 260 nm. The results are presented in Fig. 2. In an aqueous solution at pH 7, we observed a very slow decrease in the concentration of ascorbic acid, probably due to its oxidation with air (Fig. 2, curve *1*). This process is accelerated when H_2O_2 serves as an oxidant (curve *2*) and occurs more rapidly in the presence of $FeO(OH)/SiO_2$ (curve *3*). At pH 7.5–10.5, the oxidation rate of ascorbic acid remains unchanged rather than decreased as might be expected for the participa-

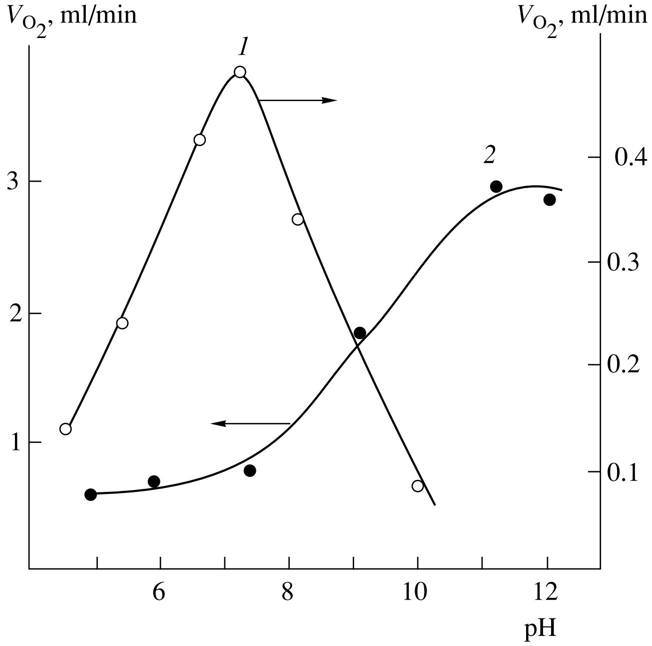


Fig. 1. Initial rate of O_2 evolution as a function of pH in the presence of (*1*) $FeO(OH)/SiO_2$ and (*2*) amorphous FeO(OH): (*1*) catalyst (0.5 g) in 10 ml of H_2O_2 (0.2 mol/l) and (*2*) catalyst (0.1 g) in 10 ml of H_2O_2 (0.1 mol/l).

Initial rate of O_2 evolution (ml/min) for different pH of the synthesis of the $FeO(OH)/SiO_2$ catalysts and compositions of the initial iron salt*

Initial salt	pH of synthesis			
	4	5	6	7
$Fe(NO_3)_3$	1.0	1.1	1.1	0.9
$FeCl_3$	1.0	1.0	1.1	1.1
$Fe_2(SO_4)_3$	0.7	–	0.6	0.9

* The catalyst (0.5 g) in 10 ml of H_2O_2 (0.5 mol/l), pH 7.2.

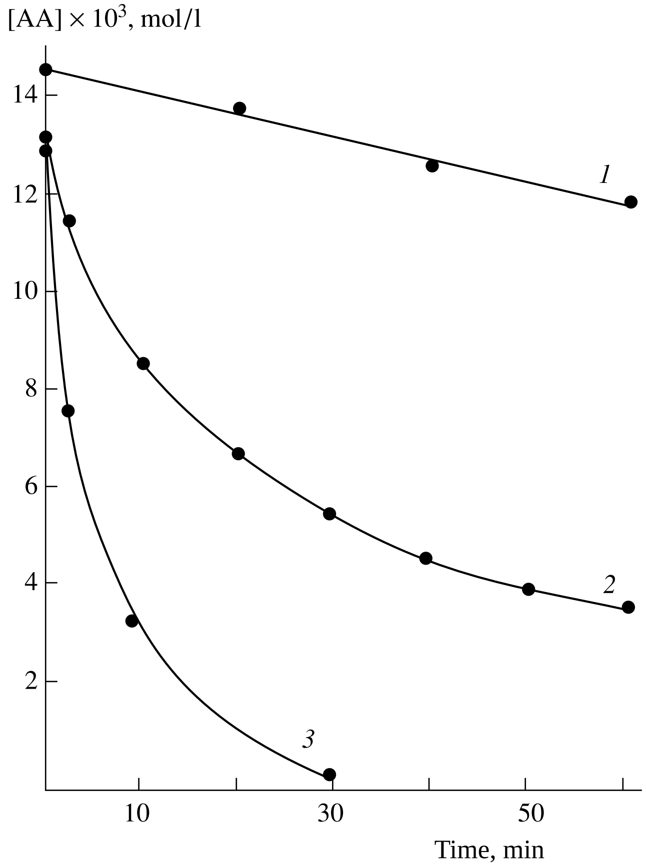


Fig. 2. The concentration of ascorbic acid vs. time under different conditions: (*1*) in an aqueous solution in air; (*2*) the same as *1*, but in the presence of H_2O_2 (0.2 mol/l); and (*3*) the same as *2*, but in the presence of $FeO(OH)/SiO_2$ (0.5 g). In all experiments, pH 7.2 was achieved by the addition of NaOH.

tion of $OH^{\bullet}$ radicals in the process [12]. Thus, these experiments also indicate the nonradical nature of H_2O_2 decomposition.

Catalytic Properties of Bulk Iron Hydroxides

Earlier, we [10] studied the catalytic oxidation of water to O_2 by the $Ru(bpy)_3^{3+}$ complex in the presence of several iron hydroxy compounds. We found that they can be arranged according to the catalytic effect in the series

$$Fe_4(OH)_{10}(SO_4) > \gamma\text{-}FeO(OH) >$$
$$\text{amorphous } FeO(OH) > NaFe_3(OH)_6(SO_4)_2,\ \alpha\text{-}FeO(OH).$$

Since, according to our data [14], the mechanism of water oxidation includes the intermediate formation of peroxo complexes with catalytically active ions, this suggests possible catalytic activity of the catalysts for water oxidation in other oxidation reactions in aqueous solutions involving O_2 and H_2O_2. This hypothesis was confirmed by the oxidation of 1,1-dimethylhydrazine with air oxygen [15] and lower hydrocarbons with hydrogen peroxide [16]. Therefore, it was of interest to test several iron hydroxo compounds presented above in H_2O_2 decomposition. This activity series remains almost unchanged for the decomposition of H_2O_2, except for amorphous FeO(OH), whose activity is only slightly lower than that of the most active $Fe_4(OH)_{10}(SO_4)$.

The activity of $FeO(OH)/SiO_2$ per one iron atom under the optimum conditions is approximately the same as that of the amorphous one. Bipyridyl and the pyrophosphate ion exert the same inhibiting effect on FeO(OH), and β-naphthol has no effect on it. Still more unexpected was a substantial difference in the reaction rate as a function of pH for these two catalysts (Fig. 1). The reason for this is yet unclear, but the influence of the support is possible.

Oxidation of lower hydrocarbons. Since various iron complexes now attract attention as possible models of the methane monooxygenase enzyme, we tested iron hydroxo compounds in the oxidation of methane, ethane, and ethylene with hydrogen peroxide in aqueous solutions. The kinetic curves of O_2 evolution in air (curve *1*) and hydrocarbons listed above (curves *2–4*) in an 0.1 mol/l solution of H_2O_2 at the initial pH 9.5 in the presence of amorphous FeO(OH) are presented in Fig. 3. As for the oxidation of ascorbic acid, the retardation of H_2O_2 decomposition and the acidification of the solution to pH 8.8–8.9 are observed. After the end of H_2O_2 decomposition in an atmosphere of methane and ethylene, formic acid was detected in the reaction mixture (2×10^{-4} mol/l (0.6% H_2O_2 conversion) and 4×10^{-4} mol/l (1.6% H_2O_2 conversion), respectively. The reaction with ethane exhibited the formation of acetaldehyde in approximately the same amounts. Note that the products of methane oxidation were not observed in the presence of the most active catalyst of H_2O_2 decomposition, $Fe_4(OH)_{10}(SO_4)$.

Catalytic Properties of Colloidal FeO(OH)

Colloidal FeO(OH) stabilized by 0.5% starch is similar to the amorphous hydroxide in H_2O_2 decomposition: the reaction rate monotonically increases with an increase in pH, decreases in the presence of ascorbic acid, and remains unchanged in the presence of β-naphthol. In all cases, the yields of O_2 corresponded to the amount of consumed H_2O_2, indicating the absence of side processes of starch oxidation. The reaction rate decreased when the same colloid was used for a second time. For example, if 0.1 mol/l FeO(OH) is used after the decomposition of 0.2 mol/l H_2O_2 at pH 10.5, under the same conditions, the rate is six times lower. However, when the same weighed sample of amorphous

FeO(OH) was repeatedly used, the reaction rate decreased only 1.2–1.3 times. The catalytic activity of supported, amorphous, and colloidal FeO(OH) per iron ion was approximately the same.

An advantage of the colloidal catalyst is the possibility of spectrophotometric monitoring of its state during the reaction with H_2O_2. The spectrum of colloidal FeO(OH) does not contain any pronounced maximum (Fig. 4, curve *1*). New maxima do not appear after the addition of H_2O_2, but the absorption at 230–300 and 400–450 nm is enhanced depending on the concentration of H_2O_2 (Fig. 4, curves *2* and *3*). When spectra were recorded against the background of the starting colloid containing no H_2O_2, new bands at 240 and 420 nm were well seen. After the end of H_2O_2 decomposition, the absorption at 240 nm disappears and that at 420 nm remains for many hours. At pH 7, the intensity of both bands is lower than that in an alkaline medium.

A very high molar absorption coefficient of the UV band ($\varepsilon_{240} > 10^4$ mol^{-1} l cm^{-1}) suggests that this is a charge-transfer band in the complex containing the peroxide ion coordinated to the iron. At the same time, the molar absorption coefficient $\varepsilon_{420} \sim 10^2$ mol^{-1} l cm^{-1} indicates that the absorbing species is not a peroxo complex. This was proven by the following experiment. A decrease in both the absorbance D_{420} and H_2O_2 concentration (using the reaction with Ti(IV)) in time was monitored in a colloidal solution containing FeO(OH) (0.01 mol/l) and H_2O_2 (0.5 mol/l) after the decomposition of the latter (~90%) at pH 10–11. The results are presented in Fig. 5 and show that D_{420} decreases only by one-third at the instant when almost all H_2O_2 is consumed. After the complete disappearance of this band, the spectrum of the colloid returns to the initial state. According to [17], the band at 420 nm can be assigned to the absorption of Fe(IV). This assumption was also confirmed by the low oxidation potential of Fe(III)/Fe(II) hydroxides (0.27 V [18]), which becomes negative at pH > 6.

When the decomposition of H_2O_2 by the colloidal catalyst was carried out in a methane atmosphere, the behavior of absorption bands at 240 and 420 nm changed strongly (Fig. 6). The rate of peroxide disappearance (D_{240}) in methane is much higher than that in air, and Fe(IV) accumulates for a longer time than in air. These changes become more pronounced in the presence of ethylene. In hydrocarbons, the colloidal solution is additionally acidified (pH changes from 11.6 to 10.6–10.8), which is probably due to the formation of formic acid, whereas under the same conditions in air, pH remains unchanged. Unfortunately, we failed to determine the concentration of HCOOH in this reaction mixture by the reaction with chromotropic acid because of the impeding influence of iron ions and starch in the analysis.

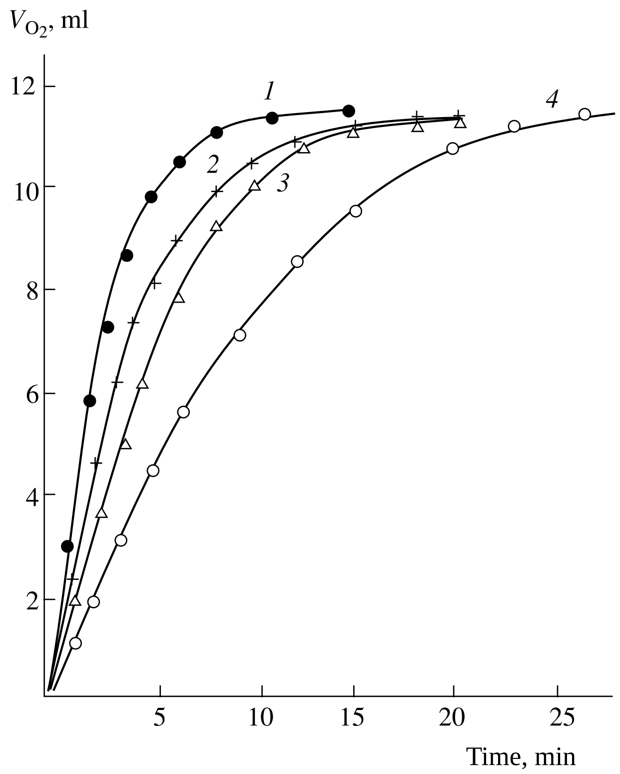


Fig. 3. Kinetics of H_2O_2 decomposition in (*1*) air, (*2*) methane, (*3*) ethane, and (*4*) ethylene. Amorphous FeO(OH) (0.1 g) in 10 ml of 0.1 mol/l H_2O_2, pH 9.5.

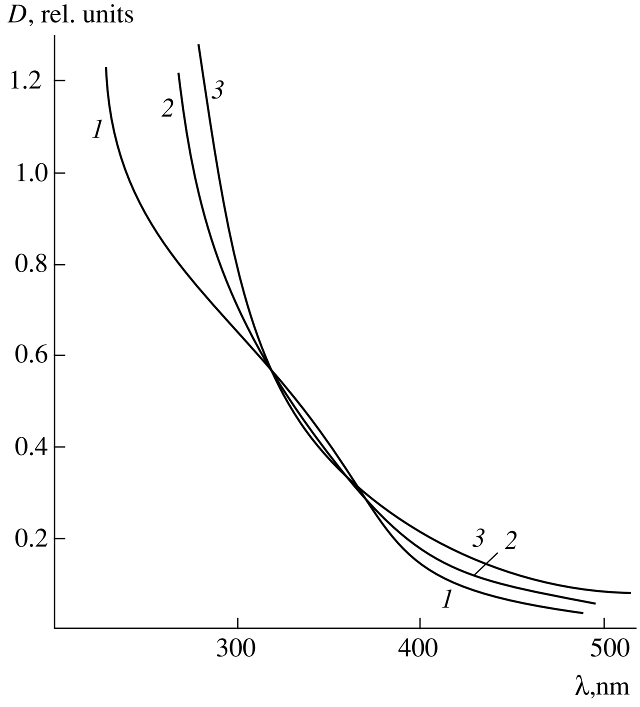


Fig. 4. Absorption spectra of colloidal FeO(OH) (*1*) in the absence and presence of (*2*) 0.1 and (*3*) 0.2 mol/l H_2O_2. FeO(OH) (10^{-3} mol/l), 0.5% starch, pH 10, 0.2-cm cell.

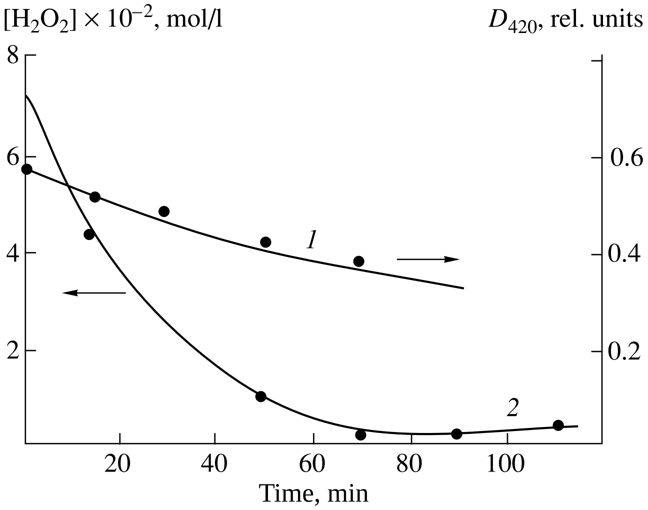


Fig. 5. Time change in (*1*) the absorbance at 420 nm and (*2*) H_2O_2 concentration after the ~90% decomposition of 0.5 mol/l H_2O_2 in an 0.01 mol/l colloidal solution of FeO(OH) stabilized by 0.5% starch. pH 10.5, 0.5-cm cell.

Mechanism of Catalytic Decomposition of H_2O_2

We may assume that the reaction under study begins with the complex formation between H_2O_2 and iron ions in the hydroxide composition. This step in the simplified form can be written as

$$\begin{gathered}\mathrm{Fe(III)\ldots Fe(III) + HOOH}\\ \rightleftharpoons \mathrm{Fe(III)[OOH]^{-}\ldots Fe(III) + H^{+}}.\end{gathered} \tag{I}$$

The absorption band with a maximum at 240 nm can be attributed to peroxide with the terminal structure because the known bridged iron peroxides have an intense absorption band at 500–600 nm [19]. Equilibrium (I) is confirmed by the acidification of the reaction mixture immediately after the addition of H_2O_2 and an increase in D_{240} with an increase in the concentration of H_2O_2 and pH.

Then the second H_2O_2 molecule can coordinate to the same active site, and intramolecular transfer of two electrons can occur to form O_2. However, this disagrees with the first order with respect to H_2O_2 found for the reaction.

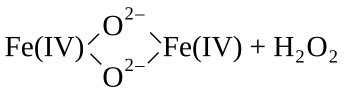
Another variant is intramolecular one-electron transfer in the formed peroxide to form Fe(IV) and the $OH^{\bullet}$ radical, but the experiments with radical traps and ascorbic acid indicate the absence of this pathway. This is also indicated by 100% yields of O_2 in the colloids, because the stabilizer of these solutions (starch) can also be oxidized with radical species. Therefore, we assume that the following step can be the structural rearrangement of the starting terminal peroxide, for example, into the bridged peroxide similarly to that observed for copper hydroxide catalysts [11]

$$\begin{gathered}\mathrm{Fe(III)[OOH]^{-}\ldots Fe(III)}\\ \rightleftharpoons \mathrm{Fe(III)[O_2]^{2-}Fe(III) + H^{+}}.\end{gathered} \tag{II}$$

Two adjacent Fe(III) ions can be oxidized to Fe(IV) in the peroxide formed

$$\begin{gathered}\mathrm{Fe(III)[O_2]^{2-}Fe(III)}\\ \longrightarrow \mathrm{Fe(IV){=}O^{2-}\ldots Fe(IV){=}O^{2-}}.\end{gathered} \tag{III}$$

The Fe(IV) ions in the hydroperoxide are relatively stable (they are retained for many hours) and poorly reactive as indicated by a decrease in the rate of H_2O_2 decomposition when the same Fe(IV)-containing catalyst is repeatedly used. When the molar absorption coefficient ε_{420} 500 $mol^{-1}\ l\ cm^{-1}$ is used for Fe(IV) [17], the colloidal catalyst contains 30–50% Fe(IV) at the end of H_2O_2 decomposition (depending on pH and the H_2O_2 concentration). Therefore, the activity loss can reasonably be related to the presence of this ion in the catalyst. However, we cannot exclude that this inactive form is preceded by another form; a more active dimeric Fe(IV) intermediate that acts as a two-electron oxidant for the second H_2O_2 molecule



$$\begin{gathered}\mathrm{Fe(IV)}\begin{smallmatrix}\diagup\,\mathrm{O^{2-}}\,\diagdown\\ \diagdown\,\mathrm{O^{2-}}\,\diagup\end{smallmatrix}\mathrm{Fe(IV) + H_2O_2}\\ \longrightarrow \mathrm{Fe(III)\ldots Fe(III) + O_2 + 2OH^{-}}.\end{gathered} \tag{IV}$$

Note that the mechanism of methane monooxygenase functioning in one of these steps assumes the transformation of the bridged peroxide with the μ-1,2 structure into either μ-1,1 [19] or μ-$\eta^2:\eta^2$ [20] and then into the methane-oxidizing dimer of Fe(IV) [21].

The nonradical mechanism of H_2O_2 decomposition in the presence of FeO(OH) suggests that the oxidation of hydrocarbons in the systems under study also can occur without radical participation. A strong change in the behavior of the absorption bands at 240 and 420 nm in the presence of hydrocarbons indicates that they interact with the intermediates of H_2O_2 decomposition, which can either be an iron peroxide or an active form of the Fe(IV) dimer. In our opinion, the formation of the $CH_3^{\bullet}$ or $C_2H_5^{\bullet}$ radicals under such mild conditions in the absence of other radical species is improbable. Therefore, we believe that hydrocarbon oxidation in the

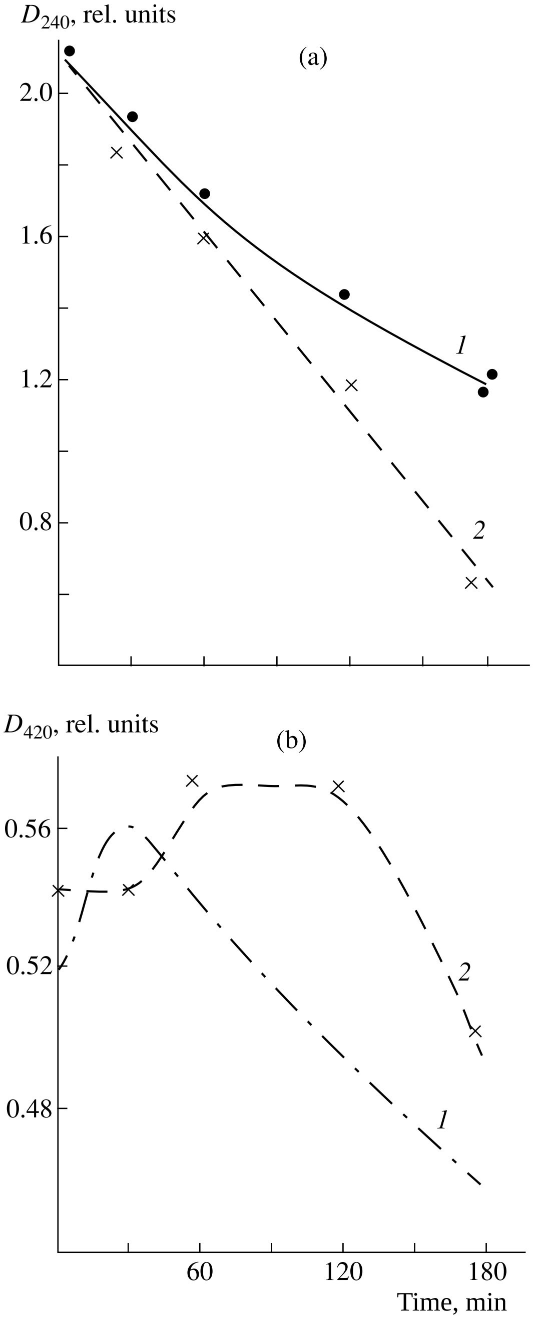


Fig. 6. Optical densities vs. time at (a) 240 and (b) 420 nm of 10^{-3} mol/l colloidal solutions of FeO(OH) in 0.1 mol/l H_2O_2 in (*1*) air and (*2*) methane. Cells (a) 0.2 and (b) 2 cm thick, colloidal FeO(OH) without H_2O_2 as the background, 290 K.

systems under study occurs via a nonradical mechanism.

Although details of the mechanism of H_2O_2 decomposition and hydrocarbon oxidation in the presence of FeO(OH) are yet unclear, the assumed schemes are of great interest because the mechanisms of this type are widely discussed for iron-containing monooxygenase [21].

CONCLUSIONS

Thus, hydroxy iron compounds as catalysts provide new, very interesting information even for the well-studied reaction of H_2O_2 decomposition. The possibility for the activation of saturated hydrocarbons under mild conditions in aqueous solutions is of interest. These objects have great advantages: facile synthesis, variety of modifications, and the absence of organic ligands, which usually impede a mechanistic study of the reaction due to side oxidation processes. Moreover, under mild conditions, they readily form intermediates, which were postulated for heme and non-heme monooxygenases: iron in high oxidation states and its peroxides. Even though these intermediates are not exact analogs of intermediates in the prosthetic centers of enzymes, their study is of great interest because they were found directly in the catalytic process occurring via a molecular mechanism in an aqueous solution rather than on individual model complexes.

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