INFLUENCE OF SELECTED METAL IONS ON STABILITY OF HYDROGEN PEROXIDE IN PRESENCE OF EDTA

Utjecaj selektiranih metalnih iona na stabilnost vodik peroksida u prisustvu EDTA

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Abstract

Stability of hydrogen peroxide in time was investigated in the presence of various metal ions and a complexing agent ethylenediaminetetraacetic acid (EDTA). Metal ions: Fe(III), Mn(II), Co(II), Cu(II), Ni(II), Zn(II) and Pb(II) were applied at different concentrations with a chelator in a concentration of $1 \cdot 10^{-5}$ mol/dm^3, and the solutions were kept at 4°C between the measurements. Obtained data indicate the existence of pronounced catalytic effect for Fe(III), Mn(II), Co(II) ions on stability of hydrogen peroxide. Cu(II) ions showed significant catalytic influence only when applied at high concentration. Ni(II), Pb(II) and Zn(II) did not show significant catalytic effect on the decay of hydrogen peroxide at applied concentrations.

Key words: hydrogen peroxide, metal ions, EDTA, stability

INTRODUCTION – *Uvod*

Hydrogen peroxide is significant to the gas phase and aqueous chemistry of the atmosphere (GUNZ and HOFFMAN, 1990). There are strong indications that H_2O_2 is involved in the formation of H_2SO_4 and HNO_3 and the corresponding salts from SO_2 and NO_x (CALVERT and STOCKWELL, 1983; CALVERT ET AL 1985). It can be decomposed by various aqueous chemical processes such as reaction with dissolved SO_2, catalyzed destruction by transition metals, oxidation by OH radicals, and photolysis (ZUO and HOIGNE, 1992, 1993; GUNZ and HOFFMANN, 1990; GRAEDEL ET AL 1986; KELLY ET AL. 1985; DENG and ZUO, 1999). If decomposition is not prevented during collection or time elapsed until analysis, the measured values could underestimate the real values. ORTIZ ET AL (2003) found that peroxides incorporated in rainwater decompose with the lifetime between 2-3 h at room temperature, and the decomposition was influenced by the presence of metallic ions. Different approaches in stabilization of H_2O_2 in the

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samples have been used: addition of sodium stannate (SAUER ET AL 1997), stabilization with p-hydroxyphenylacetic acid (KELLY ET AL 1985), acidification and complexation (ORTIZ et al 2000). Storage of the sample at low temperatures is reported by KOK (1980), SAUER et al (1997), DENG and ZUO (1999). Most of the investigations on stability of hydrogen peroxide were carried out at room temperature with less number of investigated ions so that more kinetic measurements at lower temperature are also necessary.

Therefore, the aim of this work was to investigate influence of selected transition metal ions on stability of hydrogen peroxide in presence of a chelator EDTA in synthetic solutions kept at 4°C. Investigations were carried out with solutions containing 200 μg/dm^3 of hydrogen peroxide, EDTA ($1 \cdot 10^{-5}$ mol/dm^3) and metal ions applied at three different concentrations. Co(II), Mn(II), Fe(III), Ni(II), Cu(II), Pb(II), Zn(II) were used and their influence on the stability of the analyte was monitored in time. Chemiluminescence sensors based on the Co(II) catalyzed reaction of luminol with hydrogen peroxide were used for the investigations. This work presents second part of investigations on the influence of transition metal ions on decay of hydrogen peroxide in aqueous solutions stored at 4°C.

MATERIALS AND METHODS – *Materijali i metode*

Solutions and reagents – *Otopine i reagensi*

All reagents were analytical grade, obtained from Fluka and Merck. All chemicals were used as received.

A stock solution of hydrogen peroxide (10,000 mg/ dm^3) was prepared by diluting the necessary amount of standardized 30% hydrogen peroxide to 30 cm^3 with water. Standard solutions of lower concentrations were prepared daily by appropriate dilution of the stock before use. All solutions were stored in the dark at 4°C.

Stock solutions of cobalt chloride (0.1 mol/dm^3), luminol (56.4 mmol/dm^3), and sodium lauryl sulphate were prepared according to TAHIROVIĆ ET. AL (2007).

Stock solutions of metal ions were prepared in concentrations of 1000 mg/dm^3. Working solutions were prepared daily before use. Solutions were kept in the refrigerator at 4°C between the measurements.
0.1 mol/dm^3 solution of ethylenediaminetetraacetic acid (EDTA) was prepared by dissolving 1.8615 g of Na$_2$H$_2$Y x 2H$_2$O in water. The solution was kept at 4°C. Distilled, deionized water (Milli-Q, USA) was used for preparation of all solutions.

Analytical method – *Analitička metoda*

A method based on the chemiluminescence reaction of hydrogen peroxide with luminol under alkaline conditions in the presence of Co(II) ions as a catalyst was

used. Disposable low-cost chemical sensors designed for single use were constructed according to TAHIROVIĆ ET AL (2007). The membranes were prepared by dispensing 10 cm^3 of polymer solution containing hydroxyethyl cellulose (1.5 m/v), cobalt chloride (1 $mmol/dm^3$) and sodium lauryl sulphate (400 mg/dm^3) on a microscope cover glass. Luminol and sodium phosphate are mixed to the analyte solution with a metal ion and EDTA prior to analysis. Measurements were done at 25^oC.

Chemiluminescence determinations of H_2O_2 were carried out by the home-made luminometer with a photodiode detector. The device was connected to a personal computer with an in house-written software. 10 μL of sample volume was dispensed onto the polymer membrane with a micropipette placed directly on the photodiode. The peak area of the measured signal for 100 points was used for the evaluation of the results. The two channels with highest sensitivity were used and the signals were monitored in dependence of time. Results are expressed as concentration decrease (%) versus time.

Investigation of stability of hydrogen peroxide - *Ispitivanje stabilnosti vodik peroksida*

Experiments were carried out with solutions (200 cm^3) containing hydrogen peroxide (200 $μg/dm^3$) and appropriate concentrations of the investigated metal ions (200 $μg/dm^3$, 1000 $μg/dm^3$ and 2000 $μg/dm^3$). EDTA was added before the addition of the analyte in a concentration of $1 \cdot 10^{-5}$ mol/dm^3. At the same time, blank solutions containing corresponding concentration of investigated ions, and EDTA were included. Standard solutions of H_2O_2 with EDTA were also used to compare with the obtained data. All samples were kept at 4^oC between the measurements.

Test solutions were prepared by adding luminol (1.4 $mmol/dm^3$) and sodium phosphate (53 $mmol/dm^3$) to 10 mL of the previously prepared solutions of the analyte with metal ions and EDTA. A home-made portable luminometer (Institute for chemistry, Karl-Franzens University, Graz, Austria) was used for the measurements of chemiluminescence.

RESULTS – *Rezultati*

Stability of aqueous solutions of hydrogen peroxide in concentration of 200 $μg/dm^3$ with EDTA was investigated. Solutions were prepared according to above mentioned procedures and kept in refrigerator at 4^oC. All solutions contained 200 $μg/dm^3$ of H_2O_2. Influence of Fe(III), Mn(II), Cu(II), Co(II), Ni(II), Zn(II) and Pb(II) ions was investigated in concentrations of 200 $μg/dm^3$, 1000 $μg/dm^3$, and 2000 $μg/dm^3$. EDTA was added in the solutions in a concentration of $1 \cdot 10^{-5}$ mol/dm^3. Obtained results are presented in Figure 1.
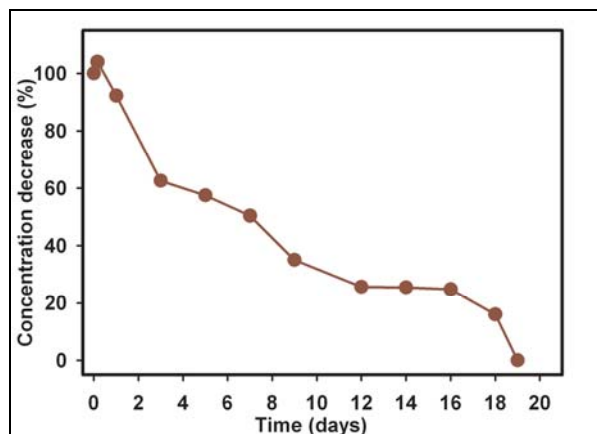
Figure 1. Decomposition of hydrogen peroxide (200 μg/dm^3) in the presence of EDTA (1 ·10^{-5} mol/dm^3).
Slika 1. Razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1 ·10^{-5} mol/dm^3).

Concentration of the analyte was diminished by 20% after 1 day and by 50% after 7 days. Complete disintegration of the analyte requires about 19 days.
The effect of Mn(II) ions on the decomposition of H_2O_2 in the solutions containing EDTA (1 ·10^{-5} mol/dm^3) is displayed in Figure 2. H_2O_2 in solutions with 200 μg/dm^3 of Mn(II) is decomposed completely after 11 days; with 1000 μg/dm^3 of Mn(II) ions after 8 days, and with 2000 μg/dm^3 Mn(II) after 7 days.

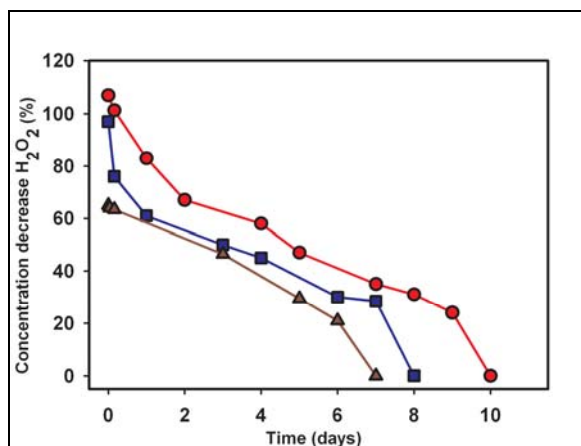Figure 2. Influence of Mn(II) ions on decay of hydrogen peroxide (200 μg/dm^3) in presence of EDTA (1·10^{-5} mol/dm^3);(●) 200 μg/dm^3 Mn(II); (■) 1000 μg/dm^3 Mn(II); (▲) 2000 μg/dm^3 Mn(II).
Slika 2. Utjecaj Mn (II) iona na razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3) ; (●) 200 μg/dm^3 Mn(II); (■) 1000 μg/dm^3 Mn(II); (▲) 2000 μg/dm^3 Mn(II).

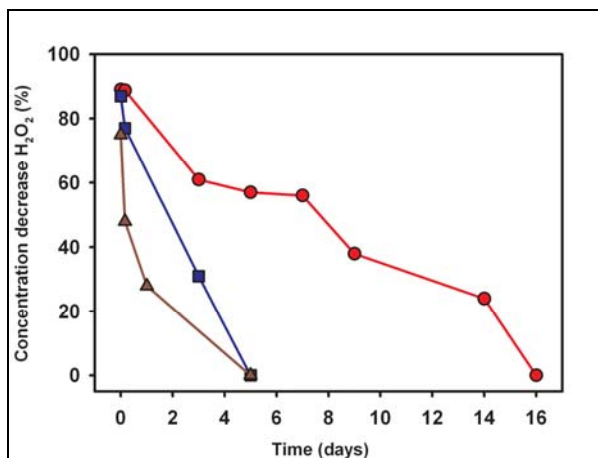The decay of H_2O_2 in the presence of Fe(III) ions with EDTA is shown in Figure 3.

Figure 3. Influence of Fe(III) ions on decay of hydrogen peroxide (200 μg/dm^3) in presence of EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Fe(III); (■) 1000 μg/dm^3 Fe(III), (▲) 2000 μg/dm^3 Fe(III).
Slika 3. Utjecaj Fe(III) iona na razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Fe(III); (■) 1000 μg/dm^3 Fe(III), (▲) 2000 μg/dm^3 Fe(III).

The decomposition of the analyte in the presence of 200 μg/dm^3 Fe(III) was very similar to hydrogen peroxide and EDTA alone, H$_2$O$_2$ will be disintegrated in 16 days. With higher concentration of Fe(III) ions (1000 and 2000 μg/dm^3) fast decomposition occurs and accomplished within 5 days.
Influence of Co(II) ions at different concentrations in the presence of EDTA was also investigated and the results are shown in Figure 4.
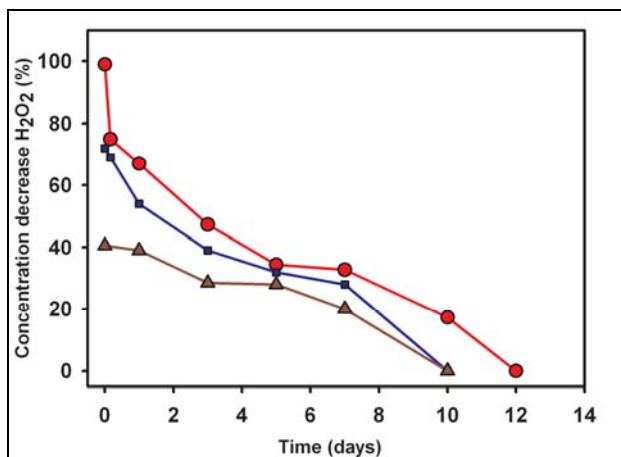

Figure 4. Influence of Co(II) ions on decay of hydrogen peroxide (200 μg/dm^3) in the presence of EDTA (1·10^{-5} mol/dm^3); 200 μg/dm^3 (●) Co(II); (■) 1000 μg/dm^3 Co(II), (▲) 2000 μg/dm^3 Co(II).
Slika 4. Utjecaj Co(II) iona na razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Co(II); (■) 1000 μg/dm^3 Co(II), (▲) 2000 μg/dm^3 Co(II).

With increasing concentrations of Co(II) ions the signals of the analyte decrease more rapidly. H_2O_2 in solutions containing 200 μg/dm^3 Co(II) ions was completely degraded after 12 days, and with 1000 μg/dm^3 and 2000 μg/dm^3 Co(II) after 10 days. The decrease of the signal for the analyte at two higher concentrations is significant.

Figure 5. shows the effect of Ni(II) ions on the decomposition of hydrogen peroxide in the presence of EDTA.
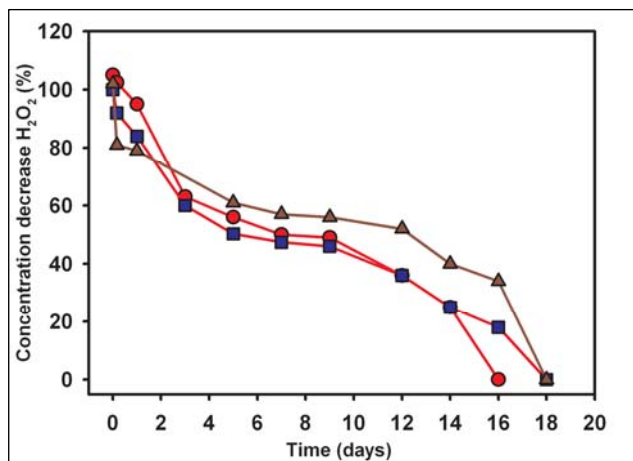

Figure 5. Influence of Ni(II) ions on the decomposition of hydrogen peroxide (200 μg/dm^3) in the presence of EDTA (1·10^{-5} mol/dm^3).; (●) 200 μg/dm^3 Ni(II); (■) 1000 μg/dm^3 Ni(II), (▲) 2000 μg/dm^3 Ni(II).
Slika 5. Utjecaj Ni(II) iona na razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Ni(II); (■) 1000 μg/dm^3 Ni(II), (▲) 2000 μg/dm^3 Ni(II).

All three curves are very similar although the signals of 200 μg/dm^3 and 1000 μg/dm^3 of the ions resemble even more. The total decomposition is accomplished after some 18 days for Ni (II) concentrations of 200 μg/ dm^3 and 1000 μg/dm^3, and after 16 days for the concentration of 2000 μg/dm^3.

The influence of Zn(II) and EDTA on the disintegration of H_2O_2 is presented in Figure 6. For all three concentrations the graphs are practically identical and very similar to the graph obtained for the solution of H_2O_2 with EDTA only. The total decomposition of H_2O_2 is accomplished after 16 days.

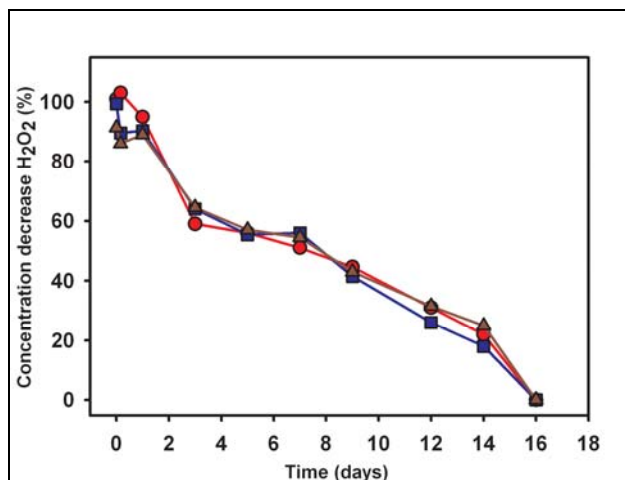Figure 6. Influence of Zn(II) ions on decay of hydrogen peroxide (200 μg/dm^3) in the presence of EDTA (1·10^{-5} mol/dm^3) ; (●) 200 μg/dm^3 Zn(II); (■) 1000 μg/dm^3 Zn(II), (▲) 2000 μg/dm^3 Zn(II).
Slika 6. Utjecaj Zn(II) iona na razlaganje vodik peroksida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3) ; (●) 200 μg/dm^3 Zn(II); (■) 1000 μg/dm^3 Zn(II), (▲) 2000 μg/dm^3 Zn(II).

Figure 7. Influence of Pb(II) ions on decay of hydrogen peroxide (200 μg/dm^3) in the presence of EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Pb(II); (■) 1000 μg/dm^3 Pb(II), (▲) 2000 μg/dm^3 Pb(II).
Slika 7. Utjecaj Pb(II) iona na razlaganje vodik peroxida (200 μg/dm^3) u prisustvu EDTA (1·10^{-5} mol/dm^3); (●) 200 μg/dm^3 Pb(II); (■) 1000 μg/dm^3 Pb(II), (▲) 2000 μg/dm^3 Pb(II).

Similar results were obtained with Pb(II) in the presence of EDTA, with an even more dilating effect on the decomposition of H_2O_2 than Zn(II) (Figure 7).

The signals for all three concentrations of Pb(II) ions were decreasing identically up to 15 days. After that there was a small difference between the lowest concentration of Pb(II) and the remaining two higher concentrations.

The effect of Cu(II) ions in the presence of EDTA was investigated only with two concentrations, 200 and 1000 $\mu g/dm^3$, since 2000 $\mu g/dm^3$ of Cu(II) ions suppressed the signal completely after addition to the solution of hydrogen peroxide even in presence of EDTA. Decomposition of the analyte with two investigated concentrations was finished in 19 and 14 days respectively. The results are shown in Figure 8.

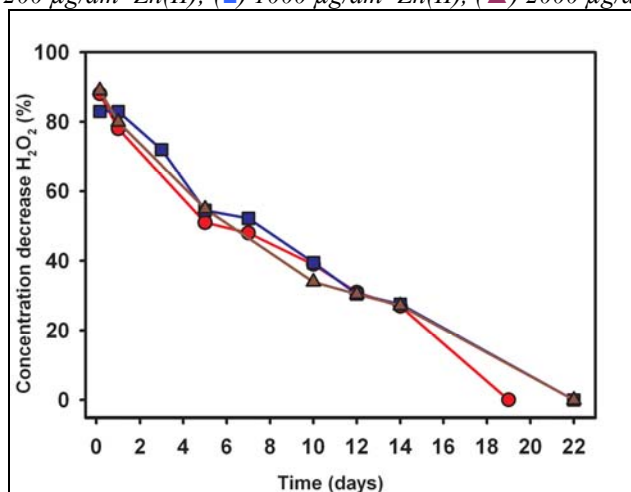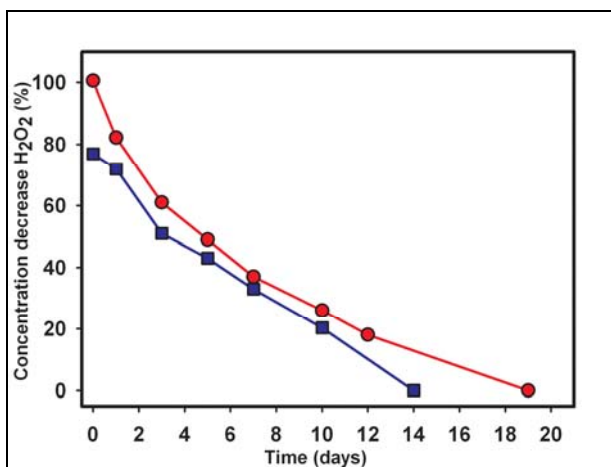Figure 8. Influence of Cu(II) ions on decay of hydrogen peroxide (200 $\mu g/dm^3$) in presence of EDTA ($1 \cdot 10^{-5}$ mol/dm^3); (●) 200 $\mu g/dm^3$ Cu(II); (■) 1000 $\mu g/dm^3$ Cu(II).
Slika 8. Utjecaj Cu(II) iona na razlaganje vodik peroksida (200 μg/dm³) u prisustvu EDTA (1·10⁻⁵ mol/dm³); (●) 200 μg/dm³ Cu(II); (■) 1000 μg/dm³ Cu(II).

DISCUSSION – *Diskusija*

It is know that the decomposition of hydrogen peroxide and its reaction with inorganic are catalyzed by transition metal ions or their complexes in both heterogeneous and homogeneous systems (HABER and WEISS, 1934; GLASNER, 1959; KREMER, 1985).

Two mechanisms were suggested for these reactions. The first is a radical chain mechanism for transition metal ions which have two available oxidation states (Cu, Fe, Co, Mn). The second mechanism is that hydrogen peroxide reacts with metal species to form an intermediate complex, M^{n+}-H_2O_2 which can decompose into O_2 and the original metal species (AUGUSTI ET AL 1998).

These investigations deal with the stability of hydrogen peroxide solutions containing different amounts of interfering metal ions. The solutions were kept at 4°C

and the signals were monitored in dependence of time. All previously checked ions were investigated in the presence of a complexing agent, EDTA ($1 \cdot 10^{-5}$ mol/L) and solutions contained 200 µg/ dm^3 of H_2O_2. Each ion was investigated in concentrations of 200 µg/dm^3, 1000 µg/dm^3 and 2000 µg/dm^3. For each test solution, a blank solution was prepared containing corresponding concentration of investigated ion, and complexing agent for experiments where EDTA was included. They were kept under the same conditions as the test solutions.

H_2O_2 added to ultra pure water with EDTA is extremely stable with total decomposition time of 19 days. Also addition of EDTA does not prevent the peroxide degradation in the solution (Figure 1).

Generally, we can notice that in presence of EDTA following ions catalyse the decay of hydrogen peroxide at higher concentrations: Fe(III)>Mn(II)>Co(II). The influence of Co (II) is pronounced at the beginning just after the addition of the ions, whereas the influences of Fe(III) and Mn(II) ions are more pronounced in the course of decaying. The data show that the influence by manganese can be reduced significantly but can not be removed completely with the addition of EDTA (Figure 2).

When comparing these data with the decomposition of hydrogen peroxide in the presence of Fe(III) without a complexing agent it can be seen that the decomposition is generally faster when EDTA was present (TAHIROVIĆ, 2006). With 1000 µg/dm^3 of Fe(III) during the first two days the decomposition of hydrogen peroxide is slower, but after that it was increased. Much faster disintegration was observed in solutions with 2000 µg/dm^3 of Fe(III) ions (Figure 3). Thus, EDTA seems to be an oxidizable substrate, which might favour the decay of H_2O_2 in case that there is still sufficient catalyst present to promote of the analyte. Some investigators found that iron complexes of EDTA (1:1) induce the catalytic reduction of hydrogen peroxide (ENGELMANN ET AL 2003). It was stated that the addition of EDTA may enhance the oxidative processes if the ligand-to-metal-ratio is low, less than one whereas the rates of progressive oxidative processes may decrease if the ratio, along with the relative amount of hydrogen peroxide, is high (excess ligand). This is full in agreement with our observations.

When EDTA is present in solutions containing Co(II) and H_2O_2, a stable complex is formed which delays the catalytic activity of Co(II) ions to significantly. As there is some amount of uncomplexed Co(II) ions due to a limited concentration of EDTA in the solution, it is expected that higher concentrations of Co(II) ions will exert a more pronounced catalytic effect on the analyte. As a consequence, it is important to notice that the decrease of the signal of hydrogen peroxide after addition of the Co(II) ions at concentrations of 1000 µg/dm^3 and 2000 µg/dm^3 to the solutions of hydrogen peroxide is still significant, despite the presence of EDTA (Figure 4).

Ni(II) at all investigated concentrations did not show any influence on the decomposition of hydrogen peroxide. There is not much significant difference between the decomposition of hydrogen peroxide in presence of various Ni(II) concentrations during the first ten days (Figure 5).

For all three concentrations of Zn(II) ions the graphs are practically identical and very similar to the signals obtained for the solution of H_2O_2 with EDTA only (Figure 6). Similar results were obtained with Pb(II) in the presence of EDTA, with an even more dilating effect on the decomposition of H_2O_2 than Zn(II) (Figure 7). Also these data here again underline the fact that Pb(II) could exert a slightly stabilizing effect on hydrogen peroxide. The reason could be very similar to Zn(II), also considering the fact that Pb(IV) is not a convenient partner for a suitable redox couple to act as a catalyst for the decomposition of H_2O_2, because of its high oxidation potential. The exact reason for the slightly stabilizing effect of Zn(II) is not yet clear, but probably some comlexation of the analyte may occur, where the complex seems to be more stable than the free H_2O_2. The lack of catalytic activity of Zn^{2+} lies in its electronic configuration, which favors only +2 as a proper oxidation state without the possibility to form an electrochemically active redox couple in solution.

From the obtained results it can be seen that the addition of EDTA suppresses the catalytic effect of Cu(II) on the decomposition with both investigated concentrations probably due to the change in the redox potential of Cu(II) ions as a result of ligation with EDTA (Figure 8).

Generally, we may conclude that EDTA will extend the stability of H_2O_2 in the presence of transition metal ions. It must be also mentioned that lower concentrations of H_2O_2 are much more sensitive to interferences than higher concentrations, even in the presence of complexing agent.

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SAŽETAK

U radu su prikazani rezultati ispitivanja stabilnosti vodik peroksida u prisustvu različitih iona prelaznih metala i kompleksirajućeg sredstva, etilendiamintetraacetatne kiseline (EDTA). U ispitivanjima su korišteni ioni Fe(III), Mn(II), Co(II), Cu(II), Ni(II), Pb(II) i Zn(II) a primjenjivani su pojedinačno. Ioni su aplicirani u koncentracijama od 200, 1000 i 2000 µg/dm^3 uz dodatak EDTA ($1 \cdot 10^{-5}$ mol/dm^3) i vodik peroksida (200 µg/dm^3). Za svaku ispitivanu otopinu korištena je odgovarajuća slijepa proba, koja je sadržala sve komponente izuzev analita. Otopine su čuvane na 4°C između mjerenja i praćena je promjena koncentracije analita u vremenu. Sva mjerenja su vršena na 25°C. Rezultati su izraženi kao procentualna promjena koncentracije vodik peroksida u vremenu.

Za praćenje signala analita korišten je prenosivi luminometar, konstruisan u laboratoriji Instituta za kemiju (Karl-Franzens Univerzitet). Detekcija analita je zasnovana na reakciji vodik peroksida i luminola u baznoj sredini, kataliziranoj Co(II) ionima u prisusutvu tenzida, natrij lauril sulfata. Mjerenja su vršena upotrebom kemiluminiscentnih senzora na bazi prethodno opisane reakcije (TAHIROVIĆ ET AL 2007).

Kompletna dezintegracija vodik peroksida završena je nakon 19 dana. Koncentracija analita bila je umanjena za 20% nakon 1 dana i za 50% nakon 7 dana. Najveći utjecaj na stabilnost analita pokazali su ioni Fe(III), Mn(II) i Co(II). Ioni Fe(III) u koncentraciji 200 µg/dm^3 nisu pokazali značajniji utjecaj na raspad analita, međutim dodani u koncentracijama od 1000 i 2000 µg/dm^3 pokazali su izrazit katalitički utjecaj pri čemu je dezintegracija analita završena za 5 dana.

Ioni mangana pokazali su katalitički efekt izražen u vremenu te je razlaganje analita uz dodatak 200, 1000 i 2000 µg/dm^3 Mn(II) iona bilo završeno za 10, 8 i 7 dana. Utjecaj Co(II) iona bio je više izražen na početku reakcije, te je analit kompletno razložen za 10 dana uz dodatak 1000 i 2000 µg/dm^3 Co(II) iona.

Cu(II) ioni nisu pokazali izražen katalitički efekt u koncentracijama od 200 i 1000 µg/dm^3, ali u koncentraciji od 2000 µg/dm^3 potpuno je potisnut signal analita. Na svim ispitivanim koncentracijama, Ni(II) ioni nisu pokazali jači utjecaj na raspad vodik peroksida. Kompletan raspad analita uočen je nakon 18 dana za 200 i 1000 µg/dm^3 Ni(II) iona i nakon 16 dana u prisustvu 2000 µg/dm^3 Ni(II) iona.

U prisustvu iona Zn(II) i Pb(II) ne dolazi do ubrzavanja raspada vodik peroksida. Na svim ispitivanim koncentracijama iona raspad vodik peroksida se