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Effect of H_2O_2 sequential dosing in the Fenton process on leachate treatment

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ABSTRACT

Landfill leachate is infiltration water percolating through the landfill body together with waste ingredients that were washed out and dissolved, and also the products of biochemical reactions that occur in stored wastes. Leachate produced in this way requires specialised treatment, in which it is necessary to account for variability in leachate volume and chemical composition. Advanced oxidation processes, in which highly reactive and non-selective •OH free radicals are produced, constitute an alternative to classical pre-treatment options. Tests were conducted on leachate samples collected from the closed landfill in Barcza in the Świętokrzyskie Province, Poland. Fenton reaction was performed for Fe^{2+} to H_2O_2 ratio of 1:10 and pH = 4 (pH was adjusted using H_2SO_4) for six Fe^{2+} catalyst doses (50, 100, 150, 200, 250 and 300 mg/L) at the temperature of 20°C ± 1°C. The entire Fe^{2+} dose was delivered at the beginning of the process. The impact of H_2O_2 sequential dosing was investigated for four different configurations at Fe^{2+} to H_2O_2 ratio of 1:10. A simple mode of linear sequencing and advanced nonlinear dosing mode were used. It was demonstrated that oxidant sequencing while the catalyst dose is kept constant leads to substantial improvement in total organic carbon, chemical oxygen demand and $UVA_{(254)}$ absorbance compared with a conventional Fenton reaction.

Keywords: Fenton reaction; Leachate; Heavy metals; AOP

1. Introduction

Landfill leachate is made up of precipitation water moving through the landfill body. It contains waste ingredients that were washed out and solved, and also products of biochemical reactions that occur in wastes [1,2]. Leachate composition is affected by many factors including the type of wastes stored, waste shredding and compaction, climate, landfill age, and surface sealing [3–6].

Leachate composition reflects biochemical changes that occur in the landfill body. During the first 5 years of the site service life, acid digestion proceeds most intensely producing volatile acids and low molecular weight alcohols. Then, the value of chemical oxygen demand (COD) index ranges 3–60 g/L. As the landfill matures, biochemical reactions become more complex. Low molecular weight fulvic and humic acids are created as products of partial digestion. COD index remains at the level of 100–500 mg/L and BOD_5/COD ratio is lower than 0.3 [5,7]. In addition to organic substances and nitrogenous compounds, leachate may contain approximately 39 potentially hazardous organic substances, including chloroorganic compounds, alkyl compounds, aromatic hydrocarbons, multi-ring aromatic hydrocarbons, and phthalates [8,9]. Consequently, the selection of an appropriate treatment method is a very difficult task that requires conducting preliminary tests on every occasion. It is also necessary to account for the variability in the leachate chemical composition and quantity. Landfill leachate management

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options include the following [5]: leachate discharge to the common sewer system, leachate storage in tanks and periodic delivery to the treatment facility, on-site leachate treatment at a small treatment plant, leachate recirculation into the landfill, and also leachate evaporation using biogas. For leachate treatment, mechanical, chemical, biological methods, and also combinations of those methods are used. Advanced oxidation processes (AOPs) provide an alternative to conventional pre-treatment methods. In AOPs, highly reactive and non-selective •OH free radicals with redox potential of 2.80 V are produced [10–12]. In AOPs, various oxidant (O_3, H_2O_2, O_3/UV, H_2O_2/UV, O_3/H_2O_2/UV) and catalyst (Mn^{2+}, Fe^{2+}, TiO_2) [1,13,14] configurations are applied. The Fenton process is the most promising AOP system [15] and is carried out in four stages [5]:

- change in pH (range 2–4),
- oxidation and coagulation reactions,
- leachate neutralisation (pH 7–8),
- removal of the sludge formed.

In the classical Fenton process, $FeSO_4{\cdot}7H_2O$ providing a source of iron (II) ions and 30% hydrogen peroxide is applied [10]. The Fenton process involves Fe^{2+} ions reaction with hydrogen peroxide (perhydrol) which generates hydroxyl radicals (at the pH value of 2–4) [11,15–18]:

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + \bullet OH + OH^- \tag{1}$$

Then, Fe^{3+} ions undergo further reactions, resulting in the formation of Fe^{2+} ions [15,17,18]:

$$H_2O_2 + Fe^{3+} \rightarrow Fe^{2+} + HO_2\bullet + H^+ \tag{2}$$

Excess of H_2O_2 ions may bind •OH radicals, reducing the oxidation effect of organic substances [16]:

$$H_2O_2 + \bullet OH \rightarrow H_2O + HO_2\bullet \tag{3}$$

The radicals $HO_2\bullet$ formed in Reactions (2) and (3) are much less reactive than the radicals •OH and have a very small share in the direct oxidation of organic compounds. Fenton reaction is much a more efficient means of removing organic compounds from leachate than other processes, including coagulation and active carbon adsorption [16]. Additionally, it is possible to perform this process in the installations used for coagulation, after they have been modified [1]. Compared with other advanced oxidation processes, this reaction provides a low-cost option for leachate treatment [12].

Photocatalytic Fenton reaction (UV-Fenton) is a classical Fenton reaction assisted by UV radiation (H_2O_2/Fe^{2+}/UV). As regards UV-Fenton reaction, UV radiation is used to generate additional •OH radicals and regenerate Fe^{2+} ions [15].

$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + \bullet OH \tag{4}$$

$$Fe(OH)_2^{+} + h\nu \rightarrow Fe(OH)^{+} + \bullet OH \tag{5}$$

The major drawback of UV-Fenton reaction is high energy costs, and also problems related to the device operation, that is, cleaning of UV lamps.

Electro-Fenton method (EF) is another modification of the classical Fenton reaction, in which electrochemical processes are employed. The process involves the formation of hydrogen peroxide due to the reduction of oxygen, delivered in the form of molecular oxygen or air, at the graphite cathode made of vitreous carbon, or carbonate-Teflon cathode. The major advantage offered by the Electro-Fenton method is that it is the possible to control •OH generation, H_2O_2 is not transported and •OH radicals do not lose activity [19,20]. The aim of study was to assess the effect of H_2O_2 sequential dosing in the Fenton process on the reduction of organic pollutants in landfill leachate.

2. Methodology of investigations

Investigations into the effect of H_2O_2 sequential dosing in the Fenton process on the reduction of pollutants in leachate were conducted at the Faculty of Environmental, Geomatics and Energy Engineering. Laboratory-scale tests were carried out at the Faculty laboratory. While collecting leachate samples, storing them and examining their chemical composition, binding standards, namely PN-ISO 5667-10:1997, were followed [21]. Leachate samples were collected from the closed MSW landfill in Barcza in the Świętokrzyskie Province, Poland. The former Barcza Zachodnia quarry lower excavation site was converted into landfill. Beginning from 1972, the landfill area of 2.2 ha received solid municipal wastes from the city of Kielce. The site closure in 1985 resulted from the fact that the landfill did not conform to construction regulations. Annually, the average quantity of leachate generated is 23,943 m^3. It is collected in two tanks, and then transported to the treatment plant.

Laboratory tests on landfill leachate pre-treatment were conducted using leachate sample volumes of 0.6 L. The sample volume was selected in such a way so that all determined parameters could be analysed. In the laboratory tests, analytically pure reagents were employed: $FeSO_4{\cdot}7H_2O$ (used as a solution at 5% concentration and calculated per Fe^{2+} dose), and H_2O_2 at 30% concentration.

In partially optimised Fenton process (at Fe^{2+} to H_2O_2 ratio of 1:10), the impact of H_2O_2 sequential dosing was examined. The tests were conducted for the process initial pH of 4 (adjusted using concentrated H_2SO_4), for different Fe^{2+} catalyst doses at the temperature of 20°C ± 1°C. After the pre-set oxidation time (120 min, stirring speed during the reaction was 50 rpm) in the Conbest JLT6 reactor, the samples were neutralised with 5% KOH to pH = 8.0. Then, the leachate was stirred at 10 rpm for 30 min. After that, $Fe(OH)_3$ was sedimented for 30 min. The whole Fe^{2+} dose and H_2O_2 were delivered at the process beginning, first for the reference sample (A series). Then, the experiment was repeated and again, the entire Fe^{2+} dose was applied at the beginning of the process. H_2O_2 dose was 10 times higher than that of Fe^{2+}; however, the H_2O_2 dosing procedure was carried out differently:

- H_2O_2 dose added in four equal portions every 30 min (e.g., on start: 100 Fe^{2+}mg/L + 250 mgH_2O_2/L; next for t = 30 min + 250 mgH_2O_2; for t = 60 min + 250 mgH_2O_2; and the last for t = 90 min + 250 mgH_2O_2) (B series);
- H_2O_2 dose added in four equal portions under different timing scenarios, namely immediately after the process

onset, after 15 min, after 45 min and after 105 min (time elapsed from the process beginning) (C series);

- H_2O_2 dose added in four equal portions under different timing scenarios, namely immediately after the process onset, after 15 min, after 45 min and after 105 min (time elapsed from the process beginning). H_2O_2 dose in the first portion added as an entire dose for Fe^{2+} to H_2O_2 ratio of 1:7 (D series);
- alternatively for Fe^{2+} to H_2O_2 ratio of 1:5, the other three portions constituted 1/3 of the remaining hydrogen peroxide (E series).

The stage above was followed by leachate examination. In leachate, prior to and after the treatment process, the following were determined: COD, total organic carbon (TOC), and selected elements including heavy metals (Cd, Cu, Cr, Fe, Ni, Pb, and Zn). The following quantities were measured: pH, electrolytic conductivity, salinity, absorbance at the 254 nm wavelength, colour, temperature, potential. All analyses performed were in compliance with the binding standards. COD was determined with the Spectroquant photometric test. Cuvette tests measurements were performed using MERCK Nova 60 spectrophotometer. Prior to the tests, the samples were heated in MERCK TR 320 thermoreactor (120 min at temperature of 148°C). Depending on the method, the determinability level varied from 10 to 10,000 mg/L. TOC was determined with persulphate–UV oxidation method using StarTOC System, it was performed in accordance with PN-EN 1484:1999 standard. Depending on the method, the determinability level was 0.05–1.0 mg/L. pH was determined with the potentiometric method using pH meter [22]. It was done in accordance with PN-EN ISO 10523:2012 standard [23]. Colour was determined with spectrophotometric method using UV/VIS spectrophotometer in accordance with PN-EN ISO 7887:2012 standard [24]. Temperature and electrolytic conductivity were determined using pH meter/SevenMulti (Mettler, Toledo) conductivity meter. Cd, Cu, Cr, Fe, Ni, Pb, Zn contents were determined using PerkinElmer Optima 8000 Inductively Coupled Plasma-Optic Emission Spectrometer (ICP-OES, Kielce). The examination was conducted according to PN-EN ISO 11885:2009 standard, it was preceded by mineralisation with aqua regia in accordance with PN-EN ISO 15587-1:2005 [25,26]. The determinability level varied 0.00001–0.0001 mg/L. Absorbance at the 254 nm were determined according to PN-C-04572:1984 [27].

The response time was chosen arbitrarily using literature data [19] as well as the experience gained and published by the authors during the research on the process of decontamination of landfill leachate. The effects of temperature and reaction time on the effluent treatment effects are shown in Fig. 1. The analysis of the effects of the reaction time and temperature showed that in all cases the largest changes in COD values took place in the first 90 min. After 120 min, stable COD values were observed. At the same time, it can be noted that the effects of the decrease in COD values improved as the reaction temperature increased.

3. Results and discussion

The results were obtained for five measurement series. Only data that met the Dixon test criterion was presented. The observations were presented graphically as the degree of reduction of COD, TOC, and $UVA_{(254)}$ for the mean value. Despite the use of three different indicators to evaluate the content of organic compounds, there is a statistically significant (Student's *t*-test of differences in par) advantage of the method of sequencing the oxidant over the classic Fenton method. The results obtained from leachate analysis are presented in Figs. 2–5 and Table 1. In raw sewage, COD value was (840 ± 33) mg/L. During the studies in the reference

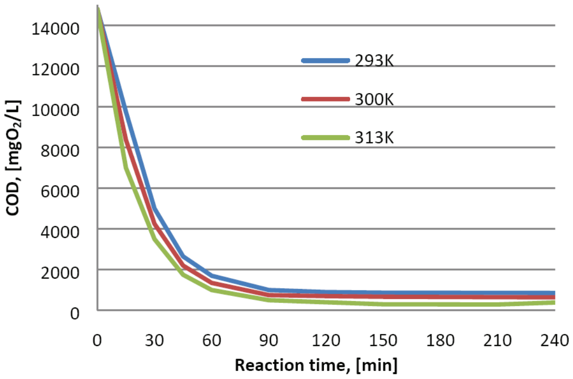

Fig. 1. Kinetics of COD changes in landfill leachate at different temperatures [19].

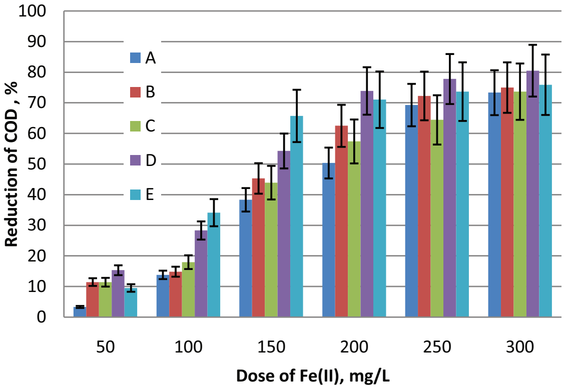

Fig. 2. Percentage reduction in COD ($\alpha = 0.05$; d$f = 5$).

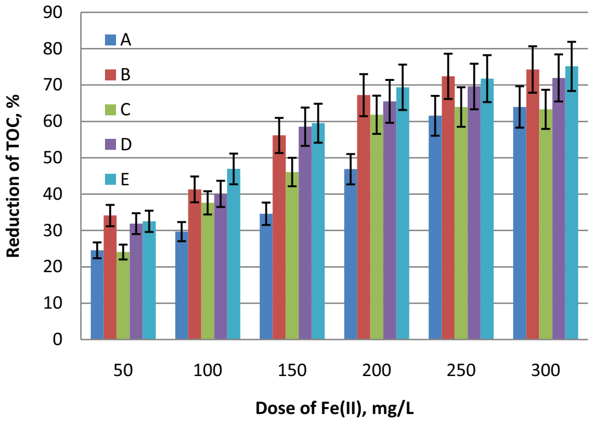

Fig. 3. Percentage reduction in TOC ($\alpha = 0.05$; d$f = 5$).

series iron doses in the range of 50–300 mg/L were applied at constant parameters pH = 4.0, reaction time t = 120 min. and Fe^{2+}/H_2O_2 = 1:10. It was observed that with the increase of Fe(II) dose gradual decrease of COD values of leachate was obtained. The highest increase in the effectiveness of COD removal was observed during an increase in the dose of this reagent from 200 to 250 mg/L (Fig. 1(A) series). At that time, the COD of the effluents decreased from 420 to 260 mg/L. Maximum reduction of COD parameter was equal only 73% for the Fe(II) catalyst dose of as much as 300 mg/L. For lower catalyst doses, COD removal results were proportionally worse. Additionally, a decrease in the ordinate of the curve

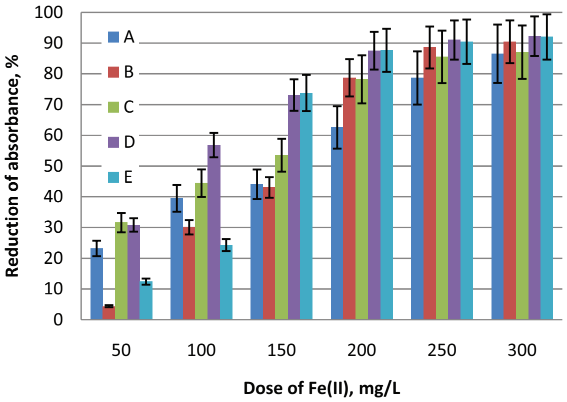

Fig. 4. Percentage reduction in $UVA_{(254)}$ absorbance (α = 0.05; df = 5).

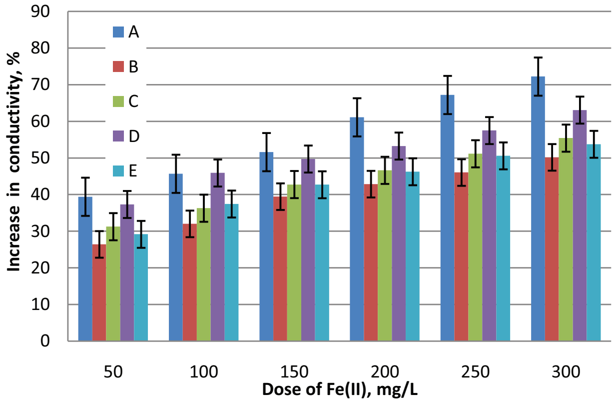

Fig. 5. Percentage increase in conductivity (α = 0.05; df = 5).

Table 1
Content of mg/L of heavy metals in leachate samples before and after the Fenton process

Sample	HMs	Cd	Cu	Cr	Ni	Pb	Fe	Zn
Raw	Mean	0.000	0.000	0.046	0.091	0.009	3.686	0.166
	Min	0.000	0.000	0.016	0.049	0.005	1.921	0.092
	Max	0.002	0.001	0.091	0.181	0.021	3.901	0.181
	Median	0.000	0.000	0.051	0.092	0.010	3.611	0.165
A	Mean	0.000	0.000	0.020	0.069	0.001	1.585	0.123
	Min	0.000	0.000	0.016	0.049	0.000	0.985	0.093
	Max	0.000	0.000	0.027	0.081	0.007	3.573	0.162
	Median	0.000	0.000	0.020	0.071	0.000	1.291	0.120
B	Mean	0.000	0.039	0.068	0.126	0.002	1.629	0.135
	Min	0.000	0.033	0.030	0.071	0.000	0.939	0.106
	Max	0.000	0.052	0.102	0.161	0.007	2.485	0.162
	Median	0.000	0.037	0.066	0.129	0.001	1.418	0.137
C	Mean	0.002	0.203	0.040	0.055	0.060	1.684	0.283
	Min	0.001	0.069	0.025	0.043	0.051	0.460	0.222
	Max	0.003	0.369	0.072	0.063	0.064	3.842	0.376
	Median	0.002	0.185	0.034	0.059	0.060	0.996	0.254
D	Mean	0.012	0.018	0.030	0.038	0.035	1.424	0.474
	Min	0.000	0.000	0.019	0.035	0.009	0.527	0.312
	Max	0.044	0.072	0.038	0.041	0.048	2.311	0.574
	Median	0.000	0.000	0.029	0.039	0.038	1.399	0.503
E	Mean	0.001	0.199	0.064	0.056	0.064	3.029	0.333
	Min	0.000	0.180	0.023	0.036	0.058	0.604	0.229
	Max	0.002	0.250	0.246	0.091	0.070	8.387	0.615
	Median	0.001	0.191	0.030	0.041	0.064	0.816	0.272

was approximately a linear function of the catalyst dose reduction (Fig. 1(A) series). For the catalyst dose of 50 mg/L, COD reduction was merely 3.3%.

The application of H_2O_2 oxidant sequencing substantially enhanced COD reduction efficiency. The dose division into four equal portions applied in the time sequence of: 0, 30, 60, and 90 min improved the efficiency in COD removal in the Fenton process already for the catalyst dose of 50 mg/L. COD removal increased to 11% (Fig. 2(B) series). Similar results were obtained for non-linear sequencing (C series), they improved clearly for the first ($t = 0$ min) dominant dose of the oxidant of 350 mg/L, whereas the remaining doses of 50 mg/L were delivered at the 15th, 45th, and finally 105th minute of the experiment (D series). In this case, for the catalyst content of 50 mg/L, COD reduction was higher than 15%. The lowest reduction values were found for E series, in addition, the initial oxidant dose was 250 mg/L, which corresponded to the catalyst to oxidant ratio of 1:5. For the lowest catalyst doses applied, namely 50 mg/L, 9.5% COD reduction was obtained.

While looking at the increase curve for E series, it can be seen that the maximum dynamics of increase are found there for the dosing range of 50–150 mg/L Fe(II). The comparison shows the best result with respect to COD reduction, namely 66%, was obtained for the dose of Fe(II) = 150 mg/L. Increase trend in the reduction of the content of organic compounds in treated leachate is especially visible in Figs. 2 and 4. The effect is particularly well visible in the middle catalyst dose range. The sequential dosage of H_2O_2 allows for effective restoration of the concentration of •OH radicals consumed during Fenton's reaction to the mineralisation of organic compounds. On the other hand, a lower concentration of hydrogen peroxide throughout the process is beneficial by reducing the •OH radical scavenger, as demonstrated by Reaction (3).

However, TOC removal proceeds in a much more linear manner for E series (Fig. 3). This indicator is particularly helpful as it indicates a lower influence of residual hydrogen peroxide on the determination results. A certain non-linearity is observed for the reference sample (A series), where a clear improvement in TOC reduction is found only above the dose of 150 m/L Fe(II). Even then, however, the results obtained (64%) are much lower than those attainable by means of H_2O_2 dose sequencing, namely 72% for D series, 74% for B series, and 75% for E series (Fig. 3).

Only for the experiment on C series, at the highest catalyst dose of 300 mg/L and oxidant dose divided into four equal portions applied immediately after the process start, after 15 min, after 45 min and after 105 min, the lowest degree of TOC reduction, equal to 63%, was obtained. However, similar results were found for 200, and also 250 mg/L Fe(II) dose, which shows an improvement compared with the reference sample (Fig. 3).

Sequential dosing of oxidant in the Fenton process did not adversely affect an increase in electrolytic conductivity of the solutions after the Fenton process, compared with the reference method (Fig. 5). Generally, a slight improvement in this respect can be observed; however, it is difficult to attribute the results to any particular factors.

It was not possible to confirm the effect of sequential dosing on the enhanced removal of heavy metals from landfill leachate (Table 1). In many cases, the best results in this respect were obtained for the reference A series.

Table 2
Value of the t_{exp} parameter for Student's t-test of differences in par $\{\alpha = 0.05, t_{crit..} = 2.02\}$

Couple/indicator:	COD	TOC	UV_{254}	$\varkappa$
{A vs. B}	3.1	6.35	**0.02**	9,42
{A vs. C}	**1.71**	2.23	3.72	7,70
{A vs. D}	**5.64**	**4.50**	**4.22**	**2,89**
{A vs. E}	3.18	5.49	**1.03**	7,31

The effect mentioned above is particularly clearly seen for Cd, Cu, Cr, and also Zn and Pb. As regards Ni, however, the best results were obtained for B, C, D, and E series. Taking into account the above, slight deterioration in the treated wastewater quality should be noted with respect to the removal of heavy metals in Fenton process modified by means of sequential dosing of hydrogen peroxide.

Table 2 shows the test results in pairs for the data shown in Figs. 2–5. A general tendency is shown that the results obtained in the B, C, D, and E series are significantly better than those obtained with the conventional Fenton method (A series). This effect is especially visible for TOC studies. It is worth mentioning that the results obtained for the D series are exceptional, because the improvement of all the examined indices determining the total content of organic compounds in landfill leachate was achieved ($t_{exp} >> t_{crit}$).

4. Summary

Advanced oxidation methods, in which highly reactive and non-selective •OH free radicals with redox potential of 2.80 V are produced, provide an alternative to classical treatment methods. The Fenton process is the most widely used AOP. The Fenton reaction shows a number of advantages: it is highly effective for the removal of organic compounds, it is relatively inexpensive compared with other AOPs; additionally, it does not require the use of advanced equipment. The paper reports an attempt at modifying the Fenton process by sequencing the oxidant dose. Leachate samples were collected from the closed MSW landfill in Barcza (the Swietokrzyskie Province, Poland). The entire Fe^{2+} dose and H_2O_2 were added at the process beginning to obtain results for the reference sample. In raw and treated leachate, the following were determined: COD, TOC, $UVA_{(254)}$ absorbance, and the content of selected elements including metals (Al, Ca, Cd, Cu, Cr, Fe, Mg, Na, Ni, K, Pb, and Zn). The following quantities were measured: pH, electrolytic conductivity, absorbance at the 254 nm wavelength, colour, temperature, and potential. Then, the experiment was repeated for the entire catalyst dose added at the process start, while H_2O_2 dose was applied on sequential basis. A simple mode of linear sequencing and advanced nonlinear dosing were used. Despite the use of three different indicators to evaluate the content of organic compounds, it was possible to demonstrate statistically significant advantage of the oxidative sequencing method over the classical Fenton method. It was demonstrated that oxidant sequencing at a constant catalyst dose results in substantial improvement in elimination of TOC in treated leachate samples compared with the results obtained for the reference sample. Significant increase in electrolytic conductivity or improvement in heavy metal removal was not observed. It seems, however, that the improvement in the effectiveness of remediation in the Fenton process, obtained by sequencing the oxidant, may be insufficient in relation to potential problems related to the necessity of precise selection of doses in the set time intervals.

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