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Pol. J. Environ. Stud. Vol. 30, No. 4 (2021), 1-7
DOI: *10.15244/pjoes/130909* ONLINE PUBLICATION DATE:

Original Research

Fenton Process Optimization with Landfill Leachate in Janczyce as an Example

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Received: 3 June 2020
Accepted: 26 November 2020

Abstract

One of the advanced oxidation methods is the Fenton's reaction which uses a mixture of iron(II) and hydrogen peroxide to generate hydroxyl radicals. The Fenton process efficiency is influenced by a number of factors including the pH, the Fe^{2+}:H_2O_2 relation or the Fe^{2+} dose. The aim of the study was to optimize the Fenton method in the application for landfill leachate treatment, with the landfill in Janczyce (Świętokrzyskie Voivodeship) as an example. The sequence of determining the most advantageous conditions for the use of Fenton's reagent was as follows: A – the pH effect; B – the oxidation time effect; C – the Fe^{2+}:H_2O_2 relation effect; D – the Fe^{2+} dose effect. The optimum catalyst/oxidant ratio of 0.125 was determined. The optimum process time of 90 minutes was determined. It was demonstrated that the Fenton process can be effectively carried out already at the pH of 4. A reduction by 75% for the TOC and by 89% for the COD, respectively, was obtained.

Keywords: landfill leachate, Fenton's reaction, wastewater treatment, carbon contaminants removal

Introduction

The operation of a landfill and the physical, chemical and biological transformations occurring within it result in the emergence of numerous environmental hazards due to, *inter alia*, emissions of landfill gases, microorganisms or leachates [1]. Leachate is infiltration waters flowing through the landfill cap along with both the waste components washed out and dissolved in them and the products of biochemical reactions occurring in the waste bed [2-5]. The resulting highly concentrated leachate waters require specialized treatment. When selecting a leachate treatment technology, minimum costs of leachate treatment need to be maintained, and e.g. the changing chemical composition and the volume of leachate need to be taken into account. It is also of significance to make maximum use of the existing landfill potential e.g. the existing heat or energy sources [6-8]. Conventional waste treatment or pre-treatment methods are sometimes insufficiently effective. Therefore, advanced oxidation processes (AOP) such as photo-Fenton (UV/H_2O_2+Fe^{2+}), electro-Fenton, ozone (O_3) oxidation, oxidation O_3+UV, O_3+UV+H_2O_2, percarbonate oxidation or the Fenton process (H_2O_2+Fe^{2+}) are increasingly applied. In view of, *inter alia*, the wide range of application, the high non-selectivity of the $^{\cdot}OH$, and the simplicity of running the process, the Fenton process is the most popular

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of the AOP methods, and its main disadvantage is the generation of sediments which need to be properly managed [2, 5, 6, 9-12].

In general, the Fenton process involves a reaction of Fe^{2+} ions with hydrogen peroxide that forms hydroxyl radicals (at the pH value below 5) (Eq. 1). Equation (1) is regarded as the basis of the Fenton's reaction [9, 10]; however, in order to understand the entire mechanism, other reactions should be taken into account. The high oxidation/reduction potential of the formed $^{\bullet}OH$ hydroxyl radicals (the oxidative potential of 2.76÷2.80 V) enables the oxidation of most contaminants to simpler biodegradable forms. In addition to hydroxyl radicals ($^{\bullet}OH$), the whole series of reactions in the Fenton process also produces hydroperoxyl radicals ($^{\bullet}O_2H$) which are also able to eliminate contaminants yet much slower than the $^{\bullet}OH$ radicals [2, 6, 12-15].

$$H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + {}^{\bullet}OH + OH^- \qquad k_1 = 40 \div 80 \text{ L mole}^{-1} \text{ s}^{-1} \tag{1}$$

The efficiency of contaminant elimination due to the application of Fenton's reaction is determined by a number of parameters including the pH value, H_2O_2 and Fe^{2+} doses, the Fe^{2+} to H_2O_2 ratio, and the process temperature and duration.

For the Fenton process, the optimum pH range at which the best treatment effects can be obtained is 2÷5. Higher pH values contribute to a decrease in reaction efficiency due to the formation of insoluble $Fe(OH)_3$. On the other hand, at a very low pH value, the $[Fe(H_2O)_6]^{2+}$ complexes are formed, which react with H_2O_2 more slowly than the others. In addition, hydrogen peroxide in the presence of a high H^+ ion concentration forms a stable $[H_3O_2]^+$ ion; hydrogen peroxide is less stable and decreases reactivity with iron ions [10,16]. A study by Zhang et al. [16] demonstrated that for landfill leachate, the best effect in COD and TOC values decrease was obtained at the pH of 2÷3.

Hydrogen peroxide and iron(II) ions are the main chemical reagents used in the conventional Fenton's reaction, therefore their doses and mutual relation are significant parameters affecting the process efficiency. An increase in the H_2O_2 dose results in better effects of the contaminant oxidation process, but too high a dose of H_2O_2 in relation to the substrate may cause the $^{\bullet}OH$ radical binding by H_2O_2. Hydrogen peroxide is harmful to many organisms, which is of significance if the Fenton process is carried out prior to biological treatment [12, 17, 18]. The unused portion of hydrogen peroxide results in an overvaluation of the COD being determined (in a reaction with strong oxidants e.g. potassium dichromate used to determine the COD, hydrogen peroxide acts as a reducing agent). In such a case, it is necessary to check on the residual H_2O_2 content in the treated wastewater. Where the residual H_2O_2 content is found, the COD value needs to be corrected using the following formula [19]:

$$COD = COD_M - d \cdot f \text{ mg/L} \tag{2}$$

COD_M – COD value obtained from titration, mg/L
d – residual H_2O_2 concentration in the sample, mg/L
f – correction factor of 0.25 (for the range of 20÷1000 mg H_2O_2/L).

Therefore, it is each time necessary to conduct testing on a laboratory scale to determine the optimum doses and H_2O_2/Fe^{2+} relations [10]. A high molar ratio above the stoichiometric reduction may be associated with the formation of a number of side reactions which affect the Fenton's reaction efficiency [16].

It is recommended that the process temperature should fall within the range of 20÷40ºC [10, 19, 20].

According to a study conducted by E. R. B. Dantas et al. [21], the optimum conditions for running the Fenton process for the treatment of landfill leachates are: an Fe^{2+} dose of 12 g/L, a H_2O_2/Fe^{2+} molar ratio of 9, the pH of 2.0, oxidation duration of 30 min, flocculation duration of 10 min, and sedimentation duration of 15 min. At such parameters, the following values were decreased: COD by approx. 87%, BOD by approx. 70%, and the TC (residual true colour) by over 90%, at initial values of the COD of 17 988 mg/L, BOD of 6012 mg/L, and TC of 4900 uH. As regards the study conducted by S.T. Gowd and S. Yashaswini [22], the optimum parameters of running the Fenton process was the reaction duration of 60 min, the pH of 3.0, a H_2O_2 dose of 0.5 M and a Fe^{2+} dose of 1000 mg/L. At the following initial wastewater parameters: BOD of 2400 mg/L, COD of 13 500 mg/L and chlorides of 3000 mg/L, a reduction in the parameter values by 48% (BOD), 67% (COD) and 40% (chlorides) was obtained. On the other hand, H. Zhang et al. [16] obtained the optimum process values at the oxidation time of 30 min, the pH of 2.5, the molar ratio of H_2O_2 do Fe(II) of 1.5, at the following parameter initial values in landfill leachate: the pH of 6.65÷6.69, COD of 8298÷8894 mg/L, TOC of 2040÷2207 mg/L, and the alkalinity as $CaCO_3$ of 3500÷4600 mg/L.

The above results clearly show that it is each time necessary to conduct testing on a laboratory scale to determine the optimum doses of H_2O_2 and Fe^{2+}, the H_2O_2/Fe^{2+} relations, the pH and the process duration.

Experimental

The leachate samples were collected and stored, and their chemical composition was studied in accordance with the existing standards PN-ISO 5667-10:1997. Leachate samples were collected from a landfill with an area of 3.64 ha in Janczyce (Świętokrzyskie voivodeship), which is a part of the Municipal Waste Disposal Plant in Janczyce. The Waste Disposal Plant in Janczyce has been operating since 2003, and provides services to approx. 150 000 inhabitants. On the landfill, waste after mechanical processing of mixed municipal waste, sorting remnants and other non-recyclable waste

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are deposited. According to estimates, over 44 thousand Mg of waste are neutralized on the landfill on an annual basis, and the operation of the landfill generated from approx. 3600 m^3 of leachate per year (2012) to over 10000 m^3 leachate per year (2015).

Laboratory tests for the landfill leachate pre-treatment were carried out on leachate samples with a volume of 0.6 L (the sample volume was selected in such a way that all the parameters being determined could be analysed). In the tests, the following analytical grade reagents were used: $FeSO_4 \cdot 7H_2O$ (used as a 5% solution and converted into a Fe^{2+} dose), 30% H_2O_2, H_2SO_4 (concentration of 98%), KOH (a 10% solution). The sequence of determining the most advantageous conditions for the use of Fenton's reagent was as follows:

- A – the pH effect was analyzed for the pH of 2.0÷5.0 (an interval of 1.0); duration of 120 min; weight relations between Fe^{2+} and H_2O_2 of 1:3 and 1:8; a Fe^{2+} dose of 1000 mg/L and 1500 mg/L;
- B – the oxidation time effect was analysed for the duration of 10 min, 30 min, 60 min, 90 min, 120 min, 150 min, 180 min, 210 min; weight relations between Fe^{2+} and H_2O_2 of 1:3 and 1:8; a Fe^{2+} dose of 1000 mg/L and 1500 mg/L;
- C – the Fe^{2+}:H_2O_2 relation effect was analysed for the weight relations between Fe^{2+} and H_2O_2 of 1:2, 1:3, 1:4, 1:5, 1:6, 1:7, 1:8, 1:9, 1:10; a Fe^{2+} dose of 100 mg/L, 500 mg/L, 1000 mg/L and 1500 mg/L; oxidation time of 90 min; the initial pH of 4.0;
- D – the Fe^{2+} dose effect was analysed for a Fe^{2+} dose ranging from 1000 to 1800 mg/L (an interval every 50 mg/L); pH of 4.0; duration of 90 min; a weight relation between Fe^{2+} and H_2O_2 of 1:8;

The tests were carried out at 20ºC±1ºC, the pH was corrected using concentrated H_2SO_4 and a 10% KOH solution. Following the pH correction, a Fe^{2+} dose was added at once to H_2O_2 to the leachate sample, and after the pre-defined oxidation time (the mixing rate during the reaction of 50 revolutions/min) in a Conbest JLT6 flocculator, the samples were neutralised with 10% KOH to the pH of approx. 7.5. Then, the leachate was mixed for 30 min at a rate of 10 revolutions/min, and afterwards sedimented for 30 min. After this stage, the effect of particular process elements on the efficiency of contaminant removal from landfill leachate was analyzed.

Both prior to and following the treatment process, the contents of the following were determined: COD, TOC, selected elements including heavy metals (Al, Ca, Cd, Cu, Cr, Fe, Mg, Na, Ni, K, Pb, Zn), and their pH, specific conductance, total amount of dissolved substances, salinity, temperature and the redox potential were measured. All analyses were conducted in line with the existing standards:

- chemical oxygen demand was determined by the Spectroquant photometric method – determination by cuvette tests using a MERCK Spectroquant Nova 60 spectrophotometer after pre-heating in a MERCK TR 320 thermoreactor (120 min at 148ºC); depending on the method, the limit of quantification was 10÷10000 mg/L, a method analogous to PN-ISO 15705:2005;
- total organic carbon was determined by the UV oxidation with peroxydisulphate method using a StarTOC System apparatus according to PN-EN 1484:1999; depending on the method, the limit of quantification was 0.050÷1000 mg/L;
- the pH was determined by the potentiometric method using a pH meter according to PN-EN ISO 10523:2012;
- total dissolved solids, temperature and specific conductance were determined using a Mettler Toledo SevenMulti pH meter/conductometer;
- Al, Ca, Cd, Cu, Cr, Fe, Mg, Na, Ni, K, Pb and Zn were determined using a Parkin Elmer ICP inductively coupled plasma optical emission spectrometer according to PN-EN ISO 11885:2009 after previous aqua regia digestion according to PN-EN ISO 15587-1:2005; the limit of quantification ranged from 0.00001 to 0.0001 mg/L.

Results and Discussion

The study results obtained following the analysis of leachate are presented in Figs 1-6 and in Table 1. Raw leachate COD was (2789±12) mg O_2/L, the TOC was (789±1) mg C/L, while the pH was 6.794. Initially, the pH effect on the efficiency of landfill leachate remediation using the Fenton's reagent at the Fe(II)/H_2O_2 relation was of 0.125 and 0.333, respectively. The tests were carried out at the pH ranging from 2 to 5, in increments of 1. The effects of COD decrease for doses of Fe(II) of, respectively, 1000 mg/L and 1500 mg/L are presented in Fig. 1. The process duration was arbitrarily adopted to be 120 min. The highest COD values after time t was obtained for the test carried out at the pH of 2 (Fig. 1). Here, the effect of H_3O^+ cation excess on the effect of $HO^\bullet$ radical scavenging is clearly noticeable. For a higher H_3O^+ oxonium ion concentration, these ions can successfully compete with organic matter for precedence in the reaction with hydroxyl radicals:

$$H_3O^+ + HO^\bullet + e^- \rightarrow 2H_2O \qquad (3)$$

Increasing the pH by 1 to a value of 3 has a marked effect on the COD of treated leachate. However, similar or even better results may be obtained when the reaction is carried out at the pH of 4. Since the pH of raw leachate is 6.794, a decrease in the pH to 3 with H_2SO_4 appears to be disputable. On the other hand, too low a portion of acid at which the pH of 5 was obtained will not be optimum (Fig. 1). In this case, theoretical premises indicate the decreasing probability of the formation of the required number of $HO^\bullet$ radicals. In view of the above, the subsequent tests were conducted at the pH of 4.

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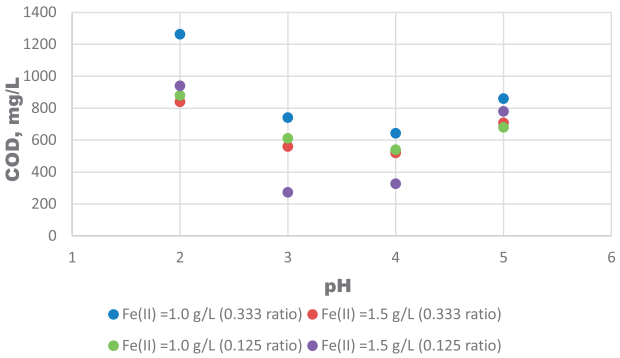

Fig. 1. Effect of the solution pH on the COD of the treated effluent.

After the pH value determination, the effect of process duration on the efficiency of landfill leachate decontamination was measured. Relations and doses similar to those at the first stage were adopted; however, the oxidation reaction was carried out within a time interval from 10 to 210 min. The test results are presented below in Fig. 2.

The tests into the effects of the duration of landfill leachate decontamination were carried out at the adopted catalyst dose of 1000 and 1500 mg/L, the catalyst/oxidant relation of 0.125 and 0.333, and at the reference pH. It was demonstrated (Fig. 2) that for the selected catalyst dose combinations, the required reaction time should not be shorter than 90 minutes. The application of shorter time does not ensure the required decontamination efficiency measured by the COD loss (Fig. 2). Extending the reaction time to 120 minutes does not significantly changes the process efficiency.

The application of longer durations appears to be pointless, and may contribute to the worsening of remediation effects, particularly in economic terms. In the third step, the effect of the Fe(II)/H_2O_2 relation on the COD and TOC of treated leachate (retentates). The study was carried out at nine different catalyst/oxidant relations ranging from 0.1 to 0.5, and the test results are presented in Figs 3 and 4 in order to select the optimum value. The test was carried out for catalyst doses of 500,

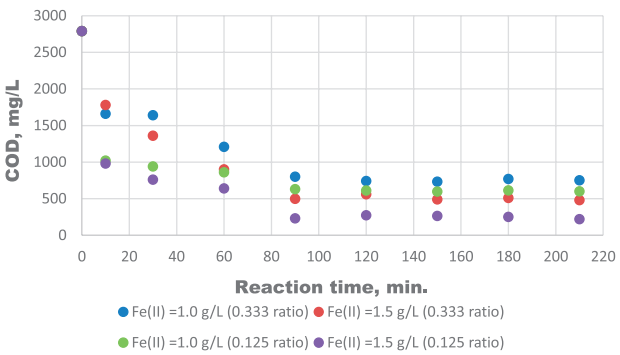

Fig. 2. The effect of the oxidation process duration on the COD of treated leachate.

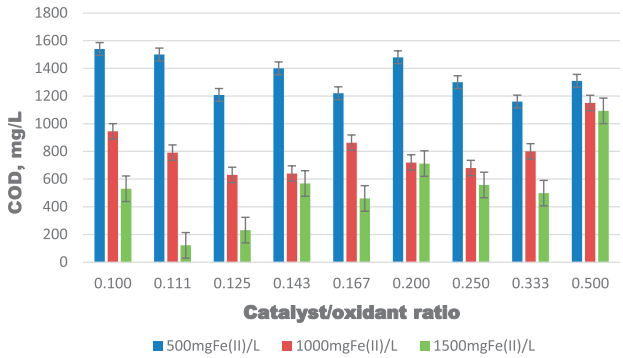

Fig. 3. The effect of Fe^{2+}/H_2O_2 weight ratio value on the treated leachate COD.

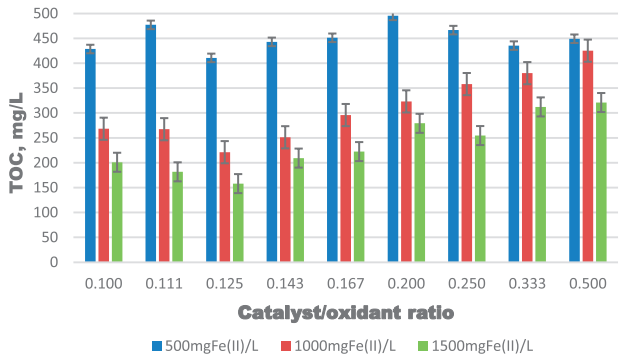

Fig. 4. The effect of Fe^{2+}/H_2O_2 weight ratio value on the treated leachate TOC.

1000 and 1500 mg Fe(II)/L at the optimum process duration and pH. Based on the results obtained at this stage of the study, it can be concluded that maintaining the catalyst/oxidant relation ranging from 0.111 to 0.125 yields the best results for the adopted Fe^{2+} catalyst doses in terms of the leachate COD and TOC elimination (Figs 3 and 4).

The greatest increase in efficiency was noted when the Fe^{2+}/H_2O_2 ratio was increased from 0.100 to 0.111. This is due to the fact that in order to obtain high efficiency of the Fenton process, an appropriately high concentration of Fe^{2+} ions is required. As demonstrated by Barbusiński in the studies [10, 15], too high a concentration of Fe^{2+} ions in relation to H_2O_2 may, however, decrease the hydroxyl radical concentration in the reaction as below:

$$Fe^{2+} + HO^{\bullet} \rightarrow Fe^{3+} + OH^{-} \quad (4)$$

This effect becomes noticeable when the value of 0.125 is exceeded, particularly for the relation of 0.200. On the other hand, at a too low catalyst/oxidant quotient (e.g. 0.100), the iron(II) dose may be insufficient to form the required number of hydroxyl radicals, as presented using the general Fenton's reaction equation (1). It is worth mentioning that the optimum $HO^{\bullet}$ radical

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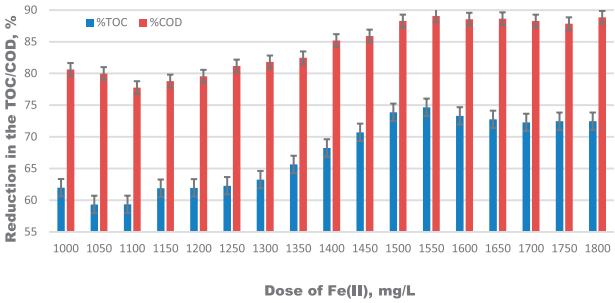

Fig. 5. Percentage reduction in the TOC and COD (α = 0.05; df = 33).

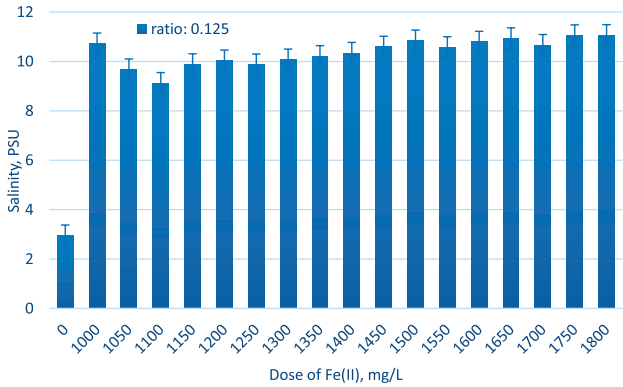

Fig. 6. Dependence of the solution salinity on the Fe(II) dose function (α = 0.05; df = 16).

concentration is the main factor affecting the efficiency of the landfill leachate decontamination process. Given that the best results in the TOC elimination were obtained at the weight ratio of 0.125, this particular relation was adopted for the subsequent stages of the study (Fig. 4).

Then, the susceptibility of landfill leachate to oxidation by the Fenton's method using hydrogen peroxide at the set pH of 4.0, the catalyst/oxidant

Table 1. Heavy metal content (mg/L) of leachate samples before and after the Fenton process.

Sample	HMs	Cd	Cu	Cr	Ni	Pb	Fe	Zn
Raw	Mean	0.000	0.139	0.094	0.067	0.011	24.49	0.767
	Min	0.000	0.102	0.089	0.066	0.006	24.11	0.691
	Max	0.002	0.144	0.099	0.068	0.015	24.53	0.771
	Median	0.000	0.138	0.094	0.066	0.009	24.51	0.766
1000	Mean	0.000	0.000	0.020	0.069	0.001	0.356	0.723
	Min	0.000	0.000	0.016	0.049	0.000	0.351	0.693
	Max	0.000	0.000	0.027	0.081	0.007	0.357	0.762
	Median	0.000	0.000	0.020	0.071	0.000	0.355	0.720
1150	Mean	0.000	0.039	0.068	0.126	0.002	8.129	0.735
	Min	0.000	0.033	0.030	0.071	0.000	8.111	0.706
	Max	0.000	0.052	0.102	0.161	0.007	8.133	0.762
	Median	0.000	0.037	0.066	0.129	0.001	8.131	0.737
1300	Mean	0.002	0.203	0.040	0.055	0.060	12.23	0.871
	Min	0.001	0.069	0.025	0.043	0.051	12.12	0.870
	Max	0.003	0.369	0.072	0.063	0.064	12.30	0.873
	Median	0.002	0.185	0.034	0.059	0.060	12.23	0.872
1550	Mean	0.002	0.018	0.030	0.038	0.035	15.26	0.381
	Min	0.000	0.000	0.019	0.035	0.009	15.17	0.379
	Max	0.004	0.072	0.038	0.041	0.048	15.31	0.383
	Median	0.000	0.000	0.029	0.039	0.038	15.26	0.381
1800 mgFe/L	Mean	0.001	0.199	0.064	0.056	0.064	7.982	0.231
	Min	0.000	0.180	0.023	0.036	0.058	7.911	0.229
	Max	0.002	0.250	0.246	0.091	0.070	8.002	0.232
	Median	0.001	0.191	0.030	0.041	0.064	7.980	0.231

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relation of 0.125, and for the duration of 90 minutes was checked. The effects of the treated leachate COD and TOC values decrease for the catalyst doses ranging from 1000 to 1800 mg/L were noted, and the test results are presented in Fig. 5. With an increase in iron(II) dose within the range from 1000 to 1250 mg Fe(II)/L, no improvement in the effects of the COD and TOC value decrease was noted. Only after exceeding the value of 1300 mg Fe(II)/L, an improvement in the effects of remediation, measured particularly by the TOC indicator, was noted. The greatest changes were observed for doses ranging from 1350 to 1500 mg/L. On the other hand, a high rate of changes in the efficiency of the COD and TOC removal was noted for doses ranging from 1450 to 1500 mg Fe(II)/L. For this reason, the optimum catalyst dose of 1550 mg/L was selected (Fig. 5). At the adopted catalyst/oxidant relation of 0.125, the complementary oxidant dose is 12400 mg H_2O_2/L.

For the optimum conditions Fenton process conditions, the TOC value of (199±1) mg C/L and the COD value of (305±5) mg O_2/L were obtained in the treated leachate. A 75% level of the landfill leachate TOC elimination and the COD elimination by over 89% were obtained. The disadvantage of the system is a significant increase in salinity, which is illustrated in Figure 6. What is interesting, for the adopted catalyst doses the effect of the catalyst dose on the treated wastewater salinity level did not become noticeable. On the other hand, a properly run Fenton process may contribute to a slight decrease in heavy metal concentrations (Table 1). In view of the above, it appears that the Fenton's method is a promising technique of organic contaminant elimination from landfill leachate, and may be an effective supplement and/or support for other techniques, including the membrane methods.

Conclusions

The conducted study demonstrated that this method for the formation of hydroxyl radicals enabled an effective decrease in the organic contaminant content of landfill leachate measured by the summary indicators as the TOC or COD. The Fenton process was run at the Fe(II) catalyst/H_2O_2 oxidant weight ratio ranging from 0.100 to 0.500 at the Fe(II) dose ranging from 1000 to 1800 mg/L, in an increment of 50 mg/L within the pH ranging from 2 to 5. In raw and treated leachate, the COD, TOC, salinity and selected heavy metal contents were determined. It was demonstrated that with an increase in the Fe(II) dose, an improvement occurs in the efficiency of landfill leachate remediation, measured by the loss of the TOC and COD – however, only to a limited extent and to a certain degree. Moreover, a clear increase in the retentate salinity was indicated. The optimum catalyst/oxidant ratio of 0.125 was determined. The optimum process time of 90 minutes was determined. It was demonstrated that the Fenton process can be effectively carried out already at the pH of 4. A reduction by 75% for the TOC and by 89% for the COD, respectively, was obtained. This confirmed the Fenton's method efficiency in the application for the landfill leachate pre-treatment.

Acknowledgements

The project was supported by the program of Minister of Science and Higher Education under the name' Regional Initiative of Excellence in 2019-2022, project number 025/RID/2018/19, financing amount of PLN 12,000,000.

The purchase of instruments (UV/VIS Lambda 25 spectrometer, StarTECH System, Optima 8000 ICP-OES) was funded from the MOLAB program – "Development of the research capacity of specialist laboratories of public universities in the Świętokrzyski Region" funded under the Innovative Economy Operational Programme.

Conflict of Interest

The authors declare no conflict of interest.

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