Fenton Process Optimization with Landfill Leachate in Janczyce as an Example

Joanna Muszyńska, Łukasz Bąk, Jarosław Górski, Katarzyna Góriska, Aleksandra Salata, Jarosław Gawdzik*

Kielce University of Technology, Faculty of Environmental, Geomatic and Energy Engineering, al. Tysiąclecia Państwa Polskiego 7, 25-314 Kielce, Poland

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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 Fe²⁺:H₂O₂ relation or the Fe²⁺ 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 Fe²⁺:H₂O₂ relation effect; D – the Fe²⁺ 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/H₂O₂+Fe²⁺), electro-Fenton, ozone (O₃) oxidation, oxidation O₃+UV, O₃+UV+H₂O₂, percarbonate oxidation or the Fenton process (H₂O₂+Fe²⁺) are increasingly applied. In view of, inter alia, the wide range of application, the high non-selectivity of the OH, and the simplicity of running the process, the Fenton process is the most popular
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\textsuperscript{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 \textsuperscript{•}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 (\textsuperscript{•}OH), the whole
series of reactions in the Fenton process also produces
hydroperoxyl radicals (\textsuperscript{R}O\textsubscript{2}H) which are also able to
eliminate contaminants yet much slower than the \textsuperscript{•}OH
radicals [2, 6, 12-15].

\[
\text{H}_2\text{O}_2 + \text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{OH} + \text{OH}^- \quad k_1 = 40 \div 80 \text{ L mole}^{-1} \text{s}^{-1}
\]  

The efficiency of contaminant elimination due to
the application of Fenton’s reaction is determined by
a number of parameters including the pH value, \text{H}_2\text{O}_2
and \text{Fe}^{2+} doses, the \text{Fe}^{2+} to \text{H}_2\text{O}_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÷3.
Higher pH values contribute to a decrease in reaction
efficiency due to the formation of insoluble Fe(OH)\textsubscript{3}. On
the other hand, at a very low pH value, the \{Fe(\text{H}_2\text{O}_2)\}\textsuperscript{2+}
complexes are formed, which react with \text{H}_2\text{O}_2 more
slowly than the others. In addition, hydrogen peroxide
in the presence of a high H\textsuperscript{+} ion concentration forms a
stable \{H\textsubscript{2}O\textsubscript{2}\}\textsuperscript{-} 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 \text{H}_2\text{O}_2 dose results in better effects
of the contaminant oxidation process, but too high
a dose of \text{H}_2\text{O}_2 in relation to the substrate may cause
the \textsuperscript{•}OH radical binding by \text{H}_2\text{O}_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 \text{H}_2\text{O}_2
content in the treated wastewater. Where the residual
\text{H}_2\text{O}_2 content is found, the COD value needs to be
corrected using the following formula [19]:

\[
\text{COD} = \text{COD}_m - d \cdot f \quad \text{mg/L}
\]  

\text{COD}_m – COD value obtained from titration, mg/L
d – residual \text{H}_2\text{O}_2 concentration in the sample, mg/L
f – correction factor of 0.25 (for the range of
20÷1000 mg \text{H}_2\text{O}_2/L).

Therefore, it is each time necessary to conduct
testing on a laboratory scale to determine the optimum
doses and \text{H}_2\text{O}_2/\text{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÷40°C [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 \text{Fe}^{2+} dose of 12 g/L, a \text{H}_2\text{O}_2/\text{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 \text{H}_2\text{O}_2 dose
of 0.5 M and a \text{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 \text{H}_2\text{O}_2 to \text{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 \text{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 \text{H}_2\text{O}_2 and \text{Fe}^{2+},
the \text{H}_2\text{O}_2/\text{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
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$_4$·7H$_2$O (used as a 5% solution and converted into a Fe$^{3+}$ dose), 30% H$_2$O$_2$, H$_2$SO$_4$ (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$_2$O$_2$ 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$_2$O$_2$ of 1:3 and 1:8; a Fe$^{2+}$ dose of 1000 mg/L and 1500 mg/L;
- **C** – the Fe$^{2+}$:H$_2$O$_2$ relation effect was analysed for the weight relations between Fe$^{2+}$ and H$_2$O$_2$ 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$_2$O$_2$ of 1:8;

The tests were carried out at 20°C±1°C, the pH was corrected using concentrated H$_2$SO$_4$ and a 10% KOH solution. Following the pH correction, a Fe$^{2+}$ dose was added at once to H$_2$O$_2$ 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 neutralized with 10% KOH (a 10% solution). The highest COD value after time t was obtained for the test carried out at the pH of 2 (Fig. 1). Here, the effect of H$_2$O$_2$ cation excess on the effect of HO$^-$ radical scavenging is clearly noticeable. For a higher H$_2$O$_2$ oxonium ion concentration, these ions can successfully compete with organic matter for precedence in the reaction with hydroxyl radicals:

$$H_3O^+ + HO^- + e^- \rightarrow 2H_2O$$  \hspace{1cm} (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$_2$SO$_4$ 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$^-$ radicals. In view of the above, the subsequent tests were conducted at the pH of 4.
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)/H₂O₂ 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, 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 Fe²⁺ catalyst doses in terms of the leachate COD and TOC elimination (Figs 3 and 4).

The greatest increase in efficiency was noted when the Fe²⁺/H₂O₂ 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 Fe²⁺ ions is required. As demonstrated by Barbusiński in the studies [10, 15], too high a concentration of Fe²⁺ ions in relation to H₂O₂ may, however, decrease the hydroxyl radical concentration in the reaction as below:

\[
\text{Fe}^{2+} + \text{HO}^- \rightarrow \text{Fe}^{3+} + \text{OH}^- \quad (4)
\]

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’ radical...
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|
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 H₂O₂/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 O₂/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/H₂O₂ 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 demonstrated. It was confirmed 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.

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Conflict of Interest

The authors declare no conflict of interest.

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