Removal of organic compounds using UV/H₂O₂ and UV/K₂S₂O₈

Jacek Leszczyński

Bialystok University of Technology, 45E Wiejska Str., 15-351 Białystok, Poland, email: jleszczynski@pb.edu.pl

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ABSTRACT

The paper presents a study aimed at comparing the efficiency of pretreatment of landfill leachate by means of UV photooxidation using hydrogen peroxide (H_2O_2) and persulfate ($S_2O_8^{-2-}$) in a falling film reactor. The leachate for the study originated from a stabilized solid waste landfill, as indicated by biochemical oxygen demand/chemical oxygen demand (BOD/COD) ratio of 0.08. The studies were conducted using variable doses of reagents, hydrogen peroxide, and persulfate, as well as variable irradiation times from 15 to 120 min. The pH, COD, total organic carbon (TOC), color, UV_{254m} absorbance, and concentrations of residual H_2O_2 and $S_2O_8^{2-}$ ions were determined during the study. Obtained results confirm the possibility of removing selected impurities applying UV method in combination with H_2O_2 and persulfate, while higher removal effect, especially with regard to COD and TOC, was achieved by using UV/S₂O₈²⁻ method, especially at a longer exposure time. The maximum COD and TOC removal effect in UV/H₂O₂ photooxidation was 15.6% and 7.3%. Using persulfate, COD removal efficiency was 22.9%, while TOC was 18.7%.

Keywords: Landfill leachate; Hydrogen peroxide; Persulfate; Falling film reactor; Photooxidation

1. Introduction

Accumulating municipal waste in landfills, due to low costs and uncomplicated exploitation, is still a basic way of waste management. The consequence of the solid waste landfill is the production of very concentrated landfill leachate that is generated during penetration of rainwater and melting water through the waste layer [1,2]. Soluble and suspended fractions of waste, both mineral and organic origin, enter the leachate. In addition, waste during storage is subject to physicochemical and biological changes. Some of them lead to the degradation of organic fraction of waste, which results in producing the pollutants that migrate to leachate waters. Landfill effluents should therefore be considered as highly contaminated sewage with a high concentration of toxic chemicals, and accumulation and treatment of leachate are still an important problem for the functioning and operation of municipal landfills [3].

One of the most important factors affecting the composition and properties of leachates is the operating time of the landfill. Landfills operating over 10 years, considered as old, produce stabilized leachate. With the growing age

of the landfill, there is a decrease in the content of organic compounds expressed by BOD and chemical oxygen demand (COD) as well as decrease in the BOD/COD ratio below 0.1, which is associated with a decrease in the proportion of volatile acids and other low-molecular organic compounds, which are easily decomposed by microorganisms [4]. At the same time, percentage of non-biodegradable compounds increases in stabilized leachate, and the concentration of ammonium nitrogen increases. Because leachate may be a direct or indirect threat to the aquatic system, it should be cleaned prior to introduction into the sewage system, primarily to remove organic substances and inorganic macrocomponents [5–7].

Due to the varied composition, practically all known physicochemical and biological processes are used for the purification or partial purification of leachates. Physicochemical processes allow for partial removal of pollutants and can lead to the transformation of pollutants present in leachates by increasing the proportion of biodegradable fractions, allowing them to be further purified by biological methods. Among the physicochemical processes, coagulation, chemical oxidation, and membrane methods are used to

Presented at the 13th Conference on Microcontaminants in Human Environment, 4–6 December 2017, Czestochowa, Poland. 1944-3994/1944-3986 © 2018 Desalination Publications. All rights reserved. purify leachates. Biological processes are mainly designed to treat leachate characterized by higher BOD/COD ratios originating from new landfill sites, which are in the initial stage of exploitation [8–11]. In the coagulation process, iron and aluminum salts are most commonly used: $FeSO_{4'}$ Fe₂(SO₄)_{3'} and Al₂(SO₄)_{3'} FeCl₃ is quite commonly used [12].

Of chemical oxidation processes, ozone and deep-oxidation methods, in which highly reactive HO[•] radicals are generated, are used in the oxidation process. These radicals act non-selectively, react quickly with many organic compounds, including difficult-to-remove refractive compounds. In advanced oxidation methods, ozone is commonly used with hydrogen peroxide, UV radiation, and their combinations: O_3/UV , H_2O_2/UV , and $O_3/H_2O_2/UV$. Catalysts, for example, TiO₂ and H_2O_2/Fe^{2+} (Fenton reaction), TiO₂/UV and TiO₂/H₂O₂/UV can also be applied [2,13].

Hydrogen peroxide is one of the most efficient oxidants capable of generating free radicals [13–15]. In the UV/H_2O_2 process, HO[•] radicals are produced according to the following reaction:

$$H_2O_2 + h\nu (UV) \rightarrow 2HO^{\bullet}$$
(1)

where one molecule of hydrogen peroxide due to light absorption generates two radicals HO[•].

In recent years, more attention has been paid to oxidation methods using persulfates ($S_2O_8^{2-}$) activated by high temperature, UV radiation, microwaves, and metals. As a result, SO_4^{--} free radicals are generated, the potential of which is $E_0 = 2.60 \text{ V}$ [16]. Photolysis of $S_2O_8^{2-}$ ion leads to the formation of SO_4^{--} according to reaction:

$$S_2O_8^{2-} + hv (UV) \to 2SO_4^{--}$$
 (2)

The SO₄ \cdot radicals are highly reactive oxidants that can initiate oxidation and mineralization of organic compounds and ammonium nitrogen. As with hydroxyl radicals, sulfate radicals can react with organic substances by electron transfer or by other mechanisms [17,18].

The paper presents a study aimed at comparing the efficiency of landfill leachate pretreatment using photooxidation in the system UV/H₂O₂ and UV/S₂O₈²⁻ in a falling film reactor.

2. Materials and methods

2.1. Materials

Potassium persulfate ($K_2S_2O_8$) and 30% H_2O_2 purchased from Sigma-Aldrich (Poland) were used as the sources of persulfate ($S_2O_8^{2-}$) and H_2O_2 . Sodium thiosulfate ($Na_2S_2O_3 \bullet 5H_2O$) was used as a quenching agent to stop the reaction in the samples. Samples of landfill leachate were taken from municipal landfill in north-eastern part of Poland.

2.2. Experimental

During the study, two various processes were applied: oxidation by UV/H_2O_2 and $UV/S_2O_8^{2-}$ process. The scheme of the experimental setup is shown in Fig. 1. The experiments were carried out in a borosilicate glass photoreactor. Ultraviolet system was composed of a falling film photoreactor, a sample

Fig. 1. Schematic diagram of the falling film photoreactor. M, rotating impeller and T, temperature sensor.

container, a cooling system, and a circulation pump. 120-W low-pressure UV lamp with a wavelength of 254 nm was used to generate UV light. Dimensions of the reactor were 100 mm in diameter and 900 mm in length. The UV lamp's glass tube is made of quartz glass. The water is fed from the tank to the upper end of the reactor by the pump. From here, the water flows downward as a thin film on the inside of the reactor wall. The recirculation of leachate was pumped from a water reservoir through reactor with 3.0 L/min of flow rate. For each experiment, 4.0 L landfill leachate was irradiated at temperature (29°C).

The effect of H_2O_2 and $S_2O_8^{2-}$ on the removal of COD, total organic carbon (TOC), color, and UV absorbance under UV irradiation was studied over a hydrogen peroxide and persulfate concentration range of 7–28 mM and reaction time from 15 to 120 min. After reaction periods of 15, 30, 60, 90, and 120 min; pH, COD, TOC, color, UV absorbance, and H_2O_2 and $S_2O_8^{2-}$ concentrations were determined. During the experimental run, samples were withdrawn at specified time intervals, quenched immediately with $Na_2S_2O_3 \bullet 5H_2O$, and filtered. The filtrate was subsequently used for further analysis by UV absorbance and TOC. All experiments were repeated three times, and the standard deviations of all measured data were below 5%.

2.3. Analytical methods

A multi-function WTW inoLab Multi 9310 IDS instrument was used to analyze pH, conductivity, and



temperature. BOD₅ was measured with the OxiTop kit. TOC measurements were performed using Analytik Jena TOC multi NC3100 instrument. The true color was measured by Platinum-Cobalt (PtCo color unit) standard method using Hach DR/4000 Spectrophotometer. Ion Chromatography Thermo Scientific ICS 5000+ was used to determine the concentrations of inorganic ions. The optical absorption spectra (200-800 nm) and ultraviolet absorbance at 254 nm (UV $_{254}$ absorbance) were measured with a spectrophotometer Merck Pharo 300. The UV_{254} measures the concentration of some organics such as humic substances and various aromatic compounds. $\rm H_2O_2$ and $\rm S_2O_8^{\ 2-}$ were determined by iodometric method [19,20]. Other analyzes were performed according to standard methods [21]. All samples were filtered through a 0.45 µm membrane prior to analysis. To report the COD concentration of the treated samples, interferences caused by residual H₂O₂ were avoided [22].

The removal efficiency of COD, TOC, color, and UV absorbance was calculated using the following equation:

$$R = \frac{\left(C_i - C_f\right) \bullet 100}{C_i} \%$$
(3)

where C_i and C_f refer to the pollutant concentration in the landfill leachate before and after the reaction, respectively.

3. Results and discussion

3.1. Leachate characteristics

The characteristics of the landfill leachate used in this study are presented in Table 1. The main parameters of samples were pH = 8.1, COD = 816.4 mg/L, TOC = 204.1 mg/L, 5-d biological oxygen demand (BOD₅) = 62 mg/L, ammonia nitrogen (NH₄⁺–N) = 405.12 mg/L, and color = 302 PtCo unit. Comparatively low BOD/COD ratios (<0.08) imply that this leachate is mature [23].

Table 1

Landfill leachate characteristics

Parameter	Value	Parameter	Value
pН	8.1	Li+	0.26
Conductivity (mS/cm)	9.83	Na ⁺	1,143.46
Color (PtCo units)	302	NH4+-N	405.12
Turbidity (NTU)	1.39	Mg^{2+}	207.81
COD (mg/L)	816.4	K^{+}	752.86
BOD (mg/L)	62	Ca ²⁺	361.57
TOC (mg/L)	204.1	F-	4.28
Dissolved organic carbon	190.4	Cl-	2,057.98
(DOC; mg/L)			
Suspended solid (SS; mg/L)	20	Br-	0.65
Total Kjeldahl nitrogen	497.2	NO ³⁻	0.29
(TKN; mg/L)			
Alkalinity (mg/L as CaCO ₃)	3,100	NO ₂ -	0.78
UV ₂₅₄ absorbance (cm ⁻¹)	0.153	SO ₄ ²⁻	88.60

3.2. Effect of COD and TOC removal

The effect of H_2O_2 and $S_2O_8^{2-}$ on the removal of COD, TOC, color, and UV absorbance under UV irradiation was studied over a hydrogen peroxide and persulfate concentration range of 7-28 mM. Initial tests were carried out only using UV radiation without the addition of H_2O_2 and $S_2O_8^{2-}$, as well as only oxidants without UV radiation. The experiment was conducted at the same oxidizer doses and reaction times as in the UV radiation process. Studies have shown very low COD removal efficiency both in the case of only UV radiation and in the case of the use of non-radiated oxidants alone. In both cases, the COD removal efficiency did not exceed 3% and was within the limits of experimental error. In the case of color and UV₂₅₄ absorbance, the use of hydrogen peroxide gave slightly better result, namely removal of 5.4% and 4.1%, respectively, at the highest H₂O₂ dose. Low photolysis efficiency may be associated with low sample permeability, which is related to the high color of landfill leachate [24]. Using only very high concentrations of hydrogen peroxide (232.7 mM) and very long reaction time of 600 min, Shu et al. [25] achieved color and COD removal efficiency, respectively, 52.3% and 59.6% with an initial COD value of 4,030 mg/L and PtCo color units of 12.275.

The effect of COD and TOC removal after UV/H₂O₂ and UV/S₂O₈²⁻ depending on the oxidant dose and exposure time is shown in Figs. 2 and 3. It was found that the initial concentrations of H₂O₂ and S₂O₈²⁻ are important parameters for the degradation of examined parameters in the process of



Fig. 2. Effect of initial concentration of $\rm H_2O_2$ and $\rm S_2O_8^{2-}$ on the COD removal under UV irradiation.



Fig. 3. Effect of initial concentration of $\rm H_2O_2$ and $\rm S_2O_8^{2-}$ on the TOC removal under UV irradiation.

photooxidation. Studies have shown that the highest removal efficiencies of COD (22.9%) and TOC (18.7%) were obtained with 28 mM persulfate after 120 min of exposure.

Much higher COD removal efficiency was obtained by Hassan et al. [26] using the persulfate at the dose of 1.5 g/L and 0.5-h exposure time for leachate after biochemical treatment; the COD removal at 30°C and 40°C was 79% and 81%, respectively.

In the case of $H_2O_{2'}$ the removal of COD and TOC at a hydrogen peroxide dose of 28 mM and a 120-min exposure time, was slightly lower, corresponding to COD (15.6%) and TOC (7.3%), respectively. In each case, the removal efficiency of COD and TOC increased with the radiation time regardless of hydrogen peroxide and persulfates dose.

The highest increase in COD removal efficiency in the UV/ H_2O_2 process was observed at a hydrogen peroxide dose of 28 mM; in the case of persulfate, the efficiency was increased successively with the dose. Somewhat different was the removal of TOC. When using persulfate, the efficiency clearly increased at higher doses in the range of 14–28 mM. However, with both hydrogen peroxide and persulfates, a decrease in the TOC removal efficiency was observed with the exposure time.

Similarly, for the lowest dose of H_2O_2 (7 mM), there was no constant increase in the COD removal efficiency throughout the whole reaction. After 60 min of exposure, the COD removal efficiency decreased markedly. This phenomenon was not observed in the UV/S₂O₈²⁻ process with respect to COD, and also at higher H_2O_2 doses applied. In each case, the presence of peroxides was detected in the leachate after the UV/H₂O₂ process and therefore this phenomenon cannot be explained by the degradation and depletion of the reactants. On the other hand, lower H_2O_2 concentrations could generate fewer HO[•] radicals, which in consequence caused lower efficiency of COD removal. Residual hydrogen peroxide and persulfate concentrations in UV/H₂O₂ and UV/S₂O₈²⁻ oxidation system are illustrated in Fig. 4.

Because of the low H_2O_2 molar absorption of about 200–300 nm, the UV/ H_2O_2 process requires high doses of hydrogen peroxide and a longer exposure time to UV radiation. In addition, high concentrations of HCO_3^- in the basic range (pH 8–9) act as free radical scavengers. High concentrations of H_2O_2 can act a similar way, which slows down the rate of oxidation [27,28].

Relatively low COD and TOC removal effect may also be associated with slightly alkaline reaction of leachates. Research by Pieczykolan et al. [29] revealed that the highest increase in COD removal efficiency was observed in hydrogen peroxide doses in the range of 3–5 g/L at initial pH 2, 4, and 5. The lowest efficiency was reached at initial pH 8.5. The maximum COD removal efficiency in this case was only 19.5% at 5 g H₂O₂/L and 0.5 h reaction time.

3.3. Effect of color and UV absorbance removal

Color removal and UV_{254} absorbance efficiencies are shown in Figs. 5 and 6. In the leachates after photooxidation process at the lowest H2O2 dose and persulfate 7 mM and 15 min of reaction, the color removal was 9.4% and UV $_{254}$ absorbance was 2.3%. Higher doses of hydrogen peroxide increased the effectiveness of these indicators removal, and at 28 mM, removal of color and UV $_{\rm 254}$ absorbance was respectively 23.1% and 7.1%. By analyzing the changes in color removal efficiency shown in Fig. 5, it can be seen that with a reaction time of 15 and 30 min, the hydrogen peroxide at 28 mM dose was more effective than the persulfate. In addition, lower H₂O₂ doses during the tested reaction time were more effective than S₂O₈²⁻. However, similarly as in the case of TOC and COD, lower effectiveness of hydrogen peroxide with the exposure time was observed even at the highest dose of 28 mM. Some slowdown of the color removal efficiency in the UV/S₂O₈²⁻ process observed at the exposure time in the range of 15-30 min, further increased along with increased radiation duration. In this case, there was no reduction in removal efficiency with reaction time as with the UV/H₂O₂ process. At a 120-min photooxidation time and 7 mM persulfate dose, the color removal effect was similar to that of a 28 mM hydrogen peroxide dose. Maximum color removal in the UV/S₂O₈²⁻ at 28 mM was 63.3%. The UV₂₅₄ removal effects shown in Fig. 6 prove a similar trend as for the color removal. Also in this case, with the time of exposure, the effectiveness of H₂O₂ was significantly reduced as opposed to the UV/S₂O₈²⁻ process. However, compared with the color removal, for the UV_{254} absorbance, the persulfate effect in the 14-28 mM dose range was significantly more effective than that of hydrogen peroxide. Achieving greater color removal efficiency would require much higher doses of hydrogen peroxide. Studies performed by Shu et al. [25] revealed that the higher hydrogen peroxide dosage promoted the more significant decolorization such as 15.7%, 25.9%, 27.6%, and 70.5% of decolorization achieving by 52.8, 116.4, 174.5, and 232.7 mM of hydrogen peroxide dosage in 150.0 min [25].



Fig. 4. Residual H_2O_2 and persulfate concentration during UV/H₂O₂ and UV/S₂O₈²⁻ oxidation.



Fig. 5. Effect of initial concentration of H_2O_2 and $S_2O_8^{2-}$ on the color removal under UV irradiation.



Fig. 6. Effect of initial concentration of $\rm H_2O_2$ and $\rm S_2O_8^{2-}$ on the UV $_{254}$ removal under UV irradiation.

4. Conclusions

- This study reports the comparison of advanced oxidation processes, UV/H₂O₂ and UV/S₂O₈²⁻, for treatment of landfill leachates using falling thin film reactor.
- Examination of physicochemical parameters of raw leachates indicated that the test material came from a stabilized solid waste landfill.
- As leading parameters illustrating efficiency of leachate pretreatment with the H₂O₂ and persulfate doses increase along with radiation time, the color, TOC, COD, and UV₂₅₄ absorbance was considered.
- Analysis of the UV/H₂O₂ UV/S₂O₈²⁻ results shows that the conditions, under which the deep oxidation process is carried out, affect its progress and effectiveness. It has been observed that the quality of leachate treatment is influenced by both the exposure time and the dose of reagents.
- Obtained results confirm the possibility of removal of selected pollution indicators by means of photooxidation in combination with H_2O_2 as well as with $S_2O_8^{2-}$; however, higher removal effect, especially with respect to TOC and COD, was achieved by using the UV/ $S_2O_8^{2-}$, especially at longer exposure time. It has also been shown that the effectiveness of UV/ H_2O_2 process was significantly influenced by the H_2O_2 dose rather than the radiation duration.
- Maximum effect of color, $UV_{254'}$ COD, and TOC removal in the case of photooxidation with hydrogen peroxide at 28 mM dose and radiation time of 120 min was, respectively, 39.2%, 17.8%, 15.6%, and 7.3%. Applying persulfate, higher effect of tested parameters removal was reached: maximum effects were as follows: 63.3%, 52.6%, 22.9%, and 18.7%.

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