Joint Use of Advanced Oxidation Process (AOP) and Advanced Reduction Process (ARP) for Alleviation of Leachate Organic Matter (LOM)

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Abstract

Landfilling is the dominant municipal solid waste (MSW) management option in the United States and many other countries. Leachate management and treatment represent an essential component in sustainable management of landfills. However, leachate organic matter (LOM) present in landfill leachate challenges existing treatment practices. The long-term goal of this study is to develop innovative treatment technologies for mitigation of LOM impacts for supporting sustainability in waste management and wastewater treatment. The overall objective of this thesis research aimed to investigate technical performance of the joint use of advanced oxidation process (AOP) and advanced reduction process (ARP) for treatment of mature landfill leachate with a focus on alleviation of LOM. In this study, the Fenton treatment and UV/sulfite process were selected as AOP and ARP, respectively, for treatment of a typical mature leachate.

Laboratory-scale batch tests were performed to evaluate the integrated ARP-AOP design for removal of LOM. LOM was substantially alleviated in the Fenton treatment in terms of chemical oxygen demand (COD), dissolved oxygen carbon (DOC), and UV$_{254}$ absorbance. At the optimal operational conditions, i.e., pH 5.0, [H$_2$O$_2$]:[Fe$^{2+}$] = 2:1, and DOD =1, COD and UV$_{254}$ absorbance were removed by 78% and 75%, respectively. The Fenton-treated leachate was further treated in the ARP system. ARP did not significantly mineralize the recalcitrant LOM, but the LOM degradation was evidenced from the alleviation of UV$_{254}$ absorbance. The reduction in UV$_{254}$ absorbance was not obviously affected by pH (8.5 -10.5) in this study, but enhanced with the increasing sulfite dose. The maximum UV$_{254}$ absorbance achieved in the ARP system (pH 9.5) was 53% in comparison to that after the Fenton treatment. Although further increase in the sulfite dose did not markedly translate into the improvement in the overall UV$_{254}$ absorbance, LOM characterization tests suggested, as the sulfite dose increased, more high
molecular weight (MW) compounds were degraded to low MW molecules, while the UV\textsubscript{254} absorbance contributed from hydrophobic LOM declined.

This study represented the first study on application of ARPs for treatment of landfill leachate. Meanwhile, a new treatment train in the combination of AOP and ARP was proposed and tested for alleviation of LOM. This study clearly demonstrates that the propose LOM treatment method is a promising approach to addressing LOM impacts for supporting sustainable management of landfill leachate.
MONTCLAIR STATE UNIVERSITY

Joint Use of Advanced Oxidation Process (AOP) and Advanced Reduction Process (ARP) for Alleviation of Leachate Organic Matter (LOM)

By
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1. INTRODUCTION AND PROBLEM STATEMENT

1.1 Background

Municipal solid waste (MSW) is one of the most serious environmental problems in the world (Li, Zhou, & Hua, 2010). Among the different MSW management options, landfilling has consistently remained the dominant one in the United States and many other countries (Abbas, Jingsong, Ping, Ya, & Al-Rekabi, 2009). Until 2019, in the United States, approximately 243 million tons of MSW was generated, of which 52.1% was disposed in municipal landfills (EPA, 2019). Sustainable MSW management requires attentions to over 13,000 closed, inactive, or active landfills in the country. However, the landfill management is greatly challenged by different negative environmental impacts. One of them is the continuous production of a large quantity of landfill leachate from these municipal landfills.

Landfill leachate is a highly polluted wastewater derived from landfills due to moisture oversaturation primarily as a consequence of precipitation (Li et al., 2010; Renou, Givaudan, Poulain, Dirassouyan, & Moulin, 2008). It is a high strength organic and inorganic wastewater, exhibiting acute and chronic toxicity, with a variety of organic wastes and inorganic species (N Calace, Liberatori, Petronio, & Pietroletti, 2001; Nicoletta Calace & Petronio, 1997; T. H. Christensen, Cossu, & Stegmann, 1992; Huo et al., 2008; Kjeldsen et al., 2002). Recently, leachate management has become an increasingly important component in integrated and sustainable solid waste management as a result of gradually tightened regulations and significant associated expenses. For example, leachate management was reported to have a typical capital costs ranging between $750 K and $14 M, and accounts for 20-33% of operating costs in landfills (No.1 single landfill operating expense) (Torrens, 2013; Walker, 2013).
1.2 Pollutants in Landfill Leachate

Municipal landfills are well-engineered sites for disposing of MSW on land for mitigation of hazards to public health and safety (G Tchobanoglous et al., 2014). The first landfill investigation began in the 1930s. Leachate is generated from a landfill when the moisture content of MSW exceeds its field capacity, which is the quantity of water held against the pull of gravity (George Tchobanoglous & Kreith, 2002). In the design of the early municipal landfills, leachate was not particularly concerned. Therefore, leachate generated could easily escape from landfills and pollute soil, groundwater, and even surface water. For example, 45 of 146 Superfund sites in New Jersey, United States are associated with the pollution due to landfill leachate (Ezyske & Deng, 2012).

Traditional pollutants typically present in municipal landfill leachates include leachate organic matter (LOM), ammonia, and toxic inorganics (e.g., As, Cr, and Pb) (Kjeldsen et al., 2002). Recent attention has been paid to emerging MSW-derived micropollutants such as per- and polyfluoroalkyl substances (PFAS), pharmaceutical and personal care products (PPCPs), and bromated flame retardants (Eggen, Moeder, & Arukwe, 2010; Gallen et al., 2016; Lang, Allred, Field, Levis, & Barlaz, 2017; Masoner et al., 2020; Osako, Kim, & Sakai, 2004; Sui et al., 2017).

Properties of LOM depends on many factors, one of which is landfilling age. Because LOM in a young leachate (typically < 1-2 years) is most biodegradable, the organic compounds can be readily treated by cost-effective biological wastewater treatment (Yang Deng, 2009). In contrast, LOM in a mature one (typically > 5 years) is biologically refractory with the ratio of the biological oxygen demand to the chemical oxygen demand (BOD$_5$/COD) below 0.3. Treatment of the persistent compounds remains a challenge. The LOM is typically characterized by a high fraction of humic-like and high molecular weight (MW) compounds (Kjeldsen et al., 2002; Zhao,
Jung, Trzopek, Torrens, & Deng, 2018). Besides traditional methods to quantify LOM, advanced analytical techniques have been used for characterization of LOM, such as size exclusion chromatography (SEC), liquid chromatography with on-line organic carbon detection (LC-OCD), nuclear magnetic resonance (NMR), and Fourier transform infrared (FTIR) spectroscopy. These techniques help to better understand the functional and structural characterization of LOM as well as the molecular weight distribution of the LOM sample (Her, Amy, Chung, Yoon, & Yoon, 2008; Lankes, Ludemann, & Frimmel, 2008; Parsi, Hartog, Górecki, & Poerschmann, 2007; C. Tang, Kwon, & Leckie, 2007; Zheng, Khan, & Croue, 2014; Zhou & Meng, 2015).

1.3 Management of Landfill Leachate

In the modern landfill design, special designs for leachate collection, storage, and management ought to be considered. The collection system includes liners at the landfill bottom to prevent leachate infiltration, the slope to drain to collection sumps, collection pipes, and pumps. Once collected, leachate can be temporarily stored in aboveground or underground holding tanks. Finally, leachate needs to be managed for minimizing the risks in ecological and human health.

Different leachate management schemes have been explored and practiced, such as spray irrigation on adjacent grassland (Gray, Pollard, Spence, Smith, & Gronow, 2005; Menser, Winant, & Bennett, 1983), recirculation within the landfill (Benson, Barlaz, Lane, & Rawe, 2007; W. Huang et al., 2016; Reinhart & Basel Al-Yousfi, 1996), evaporation using landfill gas as fuel (Benyoucef, Makan, El Ghmari, & Ouatmane, 2016; Birchler, Milke, Marks, & Luthy, 1994; Hendrych, Hejralová, Kroužek, Špaček, & Sobek, 2019), and treatment (Renou et al., 2008; Wiszniewski, Robert, Surmacz-Gorska, Miksch, & Weber, 2006). Of note, different schemes have their respective advantages and limitations in terms of technical viability and
reliability, operational ease, space requirement, economical analyses, and environmental and social impacts, which were previously discussed elsewhere (Y. Deng, 2006). Among the above schemes, leachate treatment has been widely applied for alleviation of various leachate pollutants of concern prior to discharge to natural water bodies.

Municipal landfill leachate can be treated at two different scenarios. The first one is co-treatment of leachate with municipal wastewater, in which leachate, with or without on-site pretreatment, is discharged to an adjacent municipal wastewater treatment plant (WWTP). This off-site option is a common and preferred leachate management practice in the United States, due to the lowest treatment cost and the least management complexity as compared to other strategies. Even though the co-treatment is carefully operated, this approach may bring various negative impacts, particularly for small WWTPs, such as sludge bulking, elevation of effluent COD, equipment corrosion, erratic chlorine residual, and inhibition of biological oxidation at WWTPs (Ahnert & Ehrig, 1992; Berry & Lin, 1997; Englehardt et al., 2006). Moreover, the benefits of this option have diminished because substantial UV quenching substances (UVQS) in leachate can enter WWTPs and significantly interfere with UV disinfection performance.

The second scheme is on-site treatment of landfill leachate with physical, chemical, and/or biological treatment methods, before the treated leachate is discharged to receiving water bodies or WWTPs. Earliest investigations on the leachate treatment started in the 1970. Thereafter a broad range of treatment methods have been studied and even been applied in practice (Boyle & Ham, 1974; Chian & DeWalle, 1976; Ho, Boyle, & Ham, 1974). Because persistent LOM in mature landfill leachate is little biodegradable, different physicochemical methods have been applied to mitigate the recalcitrant compounds.
Physical and chemical treatment of landfill leachate was first reported in the 1970s (Ho et al., 1974). The treatment approach is expected to serve as a full leachate treatment or a pre-/post-treatment step of biological treatment. In most cases, physical and chemical treatment aims to remove LOM or promote its biodegradability for ensuing bio-treatment. Membrane filtration and chemical oxidation are commonly used physical land chemical treatment options (Yang Deng, 2007c).

1.3.1 Membrane filtration

There are different types of membrane technologies for separating impurities from water based on the size, such as microfiltration, ultrafiltration, nanofiltration (NF), and reverse osmosis (RO), which have proven for removing various pollutants from wastewater (Hube et al., 2020). The major limitation of the membrane processes is membrane fouling that causes the accumulation of particulate or dissolved chemical or microbial species on the top of or inside the membrane material (I.-S. Chang & Kim, 2005). Membrane fouling can reduce water flux and require cleaning or replacement of membrane units, thereby increasing the costs (I. S. Chang, Clech, Jefferson, & Judd, 2002). Among the different membranes, NF and RO are effective for removing dissolved organic matter. NF can remove dissolved organic matter with MW of 150-2000 Da (Yang Deng, 2007b), while RO can remove almost all the organic matter and inorganic substances except some dissolved gas and water. However, NF and RO both suffer from a high operational cost due to the requirement in high pressure, besides membrane fouling.

1.3.2 Chemical oxidation

As previously mentioned, considerable LOM and its persistence make it impossible to achieve the removal of organic matter by biological methods only. Instead, chemical oxidation
provides a potential approach to the recalcitrant organic matter. Leachate treatment with chemical oxidation has been investigated since 1970s. Chemical oxidation can substantially alter chemical structure and decompose complex molecules into simple and less harmful compounds (Yang Deng, 2007b; Steensen, 1997). A variety of chemical oxidants have been applied, such as chlorine, permanganate, ferrate(VI), ozone, and advanced oxidation processes (AOPs) (Yang Deng, 2007c). However, the most chemical oxidants except hydroxyl radicals (•OH) or sulfate radicals cannot provide a viable removal of LOM due to their selective oxidizing capability, which limits the treatment performance.

1.3.2.1 Advanced oxidation processes

AOPs have been recognized as one of the most effective treatment methods to degrade LOM (Wu, Wu, Ma, & Chang, 2004). During the AOP, sufficient highly oxidative hydroxyl radicals (•OH) are generated to degrade target compounds. Hydroxyl radical has an extremely high reduction potential (E°) of 2.80V (Parsons, 2005a), which is just lower than that of fluorine (Table 1) (Parsons, 2005b). To generate •OH, different methods have been used, such as UVV-based AOPs (e.g., UV/TiO₂), ozone-based AOPs (e.g., O₃/H₂O₂) and catalyst-based AOPs (e.g., the Fenton process). Detailed reviews on the AOP chemistry and its application to water treatment are available elsewhere (Yang Deng & Zhao, 2015; C. Huang, Dong, & Tang, 1993). Among the different AOPs, the Fenton process has been intensively studied for treatment of various wastewaters, such as landfill leachate.
Table 1 Comparison of reduction potentials of different oxidants

<table>
<thead>
<tr>
<th>Type of oxidizing agent</th>
<th>Oxidation potential ( (E^0) ) V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fluorine</td>
<td>3.06</td>
</tr>
<tr>
<td>Hydroxyl radical</td>
<td>2.8</td>
</tr>
<tr>
<td>Oxygen</td>
<td>2.42</td>
</tr>
<tr>
<td>Ozone</td>
<td>2.08</td>
</tr>
<tr>
<td>Hypochlorite</td>
<td>1.49</td>
</tr>
<tr>
<td>Hydrogen peroxide</td>
<td>1.78</td>
</tr>
<tr>
<td>Chlorine</td>
<td>1.36</td>
</tr>
<tr>
<td>Chlorine dioxide</td>
<td>1.27</td>
</tr>
<tr>
<td>Oxygen</td>
<td>1.23</td>
</tr>
</tbody>
</table>

1.3.2.2 The Fenton treatment of landfill leachate

The Fenton reaction was first reported by H. J. H. Fenton in 1894, in which tartaric acid was degraded by hydrogen peroxide \( (\text{H}_2\text{O}_2) \) in the presence of ferrous ions \( (\text{Fe}^{2+}) \) at an acidic condition (Fenton, 1894). However, application of the Fenton process to treatment of landfill leachate began in the 1990s (Gau & Chang, 1996; Kim, Geissen, & Vogelpohl, 1997; J. Yoon, Cho, Cho, & Kim, 1998). Afterwards, numerous studies have been published to report traditional and modified Fenton processes for alleviation of LOM. Mechanisms and performance of the Fenton treatment of landfill leachate were comprehensively reviewed elsewhere (Yang Deng & Englehardt, 2006; Singh & Tang, 2013; Umar, Aziz, & Yusoff, 2010). Below is a brief introduction to the traditional Fenton chemistry and its application to leachate treatment.

In a traditional Fenton treatment process, \( \text{H}_2\text{O}_2 \) is activated by \( \text{Fe}^{2+} \) to generate highly reactive \( \cdot \text{OH} \) for oxidative degradation of target pollutants (Eq. (1-1)). Meanwhile, other reactions simultaneously occur as shown in Eq. (1-2) – (1-7). \( \text{Fe}^{3+} \) produced from Eq.(1-1) can be reduced to \( \text{Fe}^{2+} \) by hydrogen peroxide (Eq.(1-2)) and the \( \text{Fe}^{2+} \) can produce more hydroxyl radicals through Eq.(1-1). However, at a neutral or alkaline condition, the most \( \text{Fe}^{3+} \) would
precipitate to form iron sludge, and it escapes the system. Eq. (1-3) and (1-4) suggest that H\textsubscript{2}O\textsubscript{2} and Fe\textsuperscript{3+} can scavenge the produced hydroxide radicals, respectively.

\[
\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \cdot \text{OH} + \text{OH}^- \tag{1-1}
\]

\[
\text{Fe}^{3+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{2+} + \text{HO}'_2 + \text{H}^+ \tag{1-2}
\]

\[
\cdot \text{OH} + \text{H}_2\text{O}_2 \rightarrow \text{HO}'_2 + \text{H}_2\text{O} \tag{1-3}
\]

\[
\cdot \text{OH} + \text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{OH}^- \tag{1-4}
\]

\[
\text{Fe}^{3+} + \text{HO}'_2 \rightarrow \text{Fe}^{2+} + \text{O}_2\text{H}^+ \tag{1-5}
\]

\[
\text{Fe}^{2+} + \text{HO}'_2 + \text{H}^+ \rightarrow \text{Fe}^{3+} + \text{H}_2\text{O}_2 \tag{1-6}
\]

\[
2\text{HO}'_2 \rightarrow \text{H}_2\text{O}_2 + \text{O}_2 \tag{1-7}
\]

Performance of the Fenton treatment of landfill leachate relies heavily upon leachate pH, the Fenton reagents’ doses, temperature, and dissolved oxygen (DO) (Yang Deng & Englehardt, 2006). Generally, an acidic pH favors the LOM removal, an optimal [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] exists for the Fenton treatment, and higher Fenton reagents’ doses enhance the treatment.

1.4 Advanced Reduction Process

Advanced reduction processes (ARPs) have recently emerged as an innovative water and wastewater treatment technologies, which are defined as a chemical degradation process producing sufficient and highly reductive radicals for destruction of contaminants in water (Cui, Gao, & Deng, 2020a). Although the term was first proposed in the late 1990s for unintentional degradation of recalcitrant oxidized organic compounds by reducing radical species (e.g., hydrated electrons (e\textsubscript{aq}))) in a project to investigate electron beam irradiation of polluted
groundwater (Gehringer & Eschweiler, 1999), the technologies were recently used as purposeful water treatment option more than one decade later (Bhanu Prakash Vellanki, Bill Batchelor, & Ahmed Abdel-Wahab, 2013; S. H. Yoon, Abdel-Wahab, & Batchelor, 2011). The primarily reducing radicals for the pollutant degradation are \( e_{aq}^- \), in addition to hydrogen atoms (H\( \cdot \)) and others (e.g. sulfite radical anions (SO\( _3^- \))). ARPs have proven promising results for a broad range of contaminants, such as PFAS (Bao et al., 2018), perchlorate (Vellanki & Batchelor, 2013), nitrate (Bensalah, Nicola, & Abdel-Wahab, 2014), chromium (VI)(Moussavi, Jiani, & Shekoohiyan, 2015; Xie et al., 2017), and 2, 4, 6-trichlorophenol (Yazdanbakhsh, Eslami, Moussavi, Rafiee, & Sheikmohammadi, 2018).

Fundamentally different from AOPs, ARPs degrade water pollutants through chemical reduction due to the formation of strongly reductive \( e_{aq}^- \). For water treatment, \( e_{aq}^- \) can be generated through activation of a specific reducing agent (B. P. Vellanki, B. Batchelor, & A. Abdel-Wahab, 2013). However, the most frequently used options include UV irradiation of sulfite (UV/SO\( _3^{2-} \)) and UV irradiation of iodide (UV/I\( ^- \)). For example, the major reactions for the generation of \( e_{aq}^- \) in the UV/SO\( _3^{2-} \) and UV/I\( ^- \) systems are shown in Eq. (1-8) and Eq. (1-9) – (1-12), respectively.

\[
\text{SO}_3^{2-} + h\nu \rightarrow \text{SO}_3^- + e_{aq}^- \quad (1-8)
\]

\[
\text{I}^- + \text{H}_2\text{O} + h\nu \rightarrow \text{I}_2\text{O}^-* \quad (1-9)
\]

\[
\text{I}_2\text{O}^-* \rightarrow \text{I}^- + \text{H}_2\text{O} \quad (1-10)
\]

\[
\text{I}_2\text{O}^-* \rightarrow (\text{I}^-, e^-) + \text{H}_2\text{O} \quad (1-11)
\]

\[
(\text{I}^-, e^-) \rightarrow \text{I}^- + e_{aq}^- \quad (1-12)
\]
The process performance relies heavily on operating parameters (e.g., UV wavelength, UV lamp power, irradiation duration, and the reductant dose) and solution chemistry conditions (pH, dissolved oxygen, and the presence of co-existing water matrix constituents such as nitrate). Discussion on the effects of different factors on ARP degradation of PFAS was recently reviewed elsewhere (Cui et al., 2020a). It should be noted that e\textsubscript{aq} -based ARPs have not been attempted for industrial wastewater treatment, though they exhibit a strong capability for degradation of different recalcitrant pollutants.

*Hydrated electrons.* Pulse radiolysis or ionization of a specific solute can produce excess electrons (Devonshire & Weiss, 1968; Larsen, Glover, & Schwartz, 2010). Negative charges of the secondary electrons polarize adjoining water molecules to bound the electrons for generation of a metastable species, which is called e\textsubscript{aq} (Edwin J Hart, 1964; Larsen et al., 2010). It should be noted that e\textsubscript{aq} is a potent reducing agent having a reduction potential of -2.9 V (Schwarz, 1981; Swallow, 1973). Hydrated electrons have an extremely short lifespan (half-life < 300 microseconds (Edwin J Hart, 1964)). They typically attack target pollutants through a one-electron transfer mechanism (Edwin James Hart & Anbar, 1970).
2. OBJECTIVES

The long-term goal of this study is to develop innovative treatment technologies for mitigation of LOM impacts for supporting sustainability in waste management and wastewater treatment. The purpose of this thesis study aimed to investigate technical performance of the joint use of AOP and ARP for treatment of mature landfill leachate with a focus on alleviation of LOM. Because raw landfill leachate typically has substantial UV quenching substances and AOP can effectively abate the unwanted organics, the UV-based ARP was located after AOP treatment for better photo utilization. In this study, the Fenton process and UV/sulfite were chosen as the AOP and ARP, respectively. The central hypothesis is that hydroxyl radicals generated from the Fenton process can effectively degrade refractory LOM via chemic oxidation, before the residual recalcitrant degradation products can be further decomposed by hydrated electrons produced from the UV/sulfite via chemical reduction. To achieve the main goal, three specific objectives were pursued:

1) The Fenton treatment was optimized for removal of LOM. The optimization was achieved through sequential determination of the optimal levels of three operating factors, including pH, \([\text{H}_2\text{O}_2]:[\text{Fe}^{2+}]\), and the Fenton reagents’ doses.

2) Effects of pH and sulfite dose on the UV/sulfite treatment of Fenton-treated LOM were evaluated to determine the technical feasibility of ARP for alleviation of LOM.

3) Molecular weight distribution and hydrophobicity analyses were conducted to characterize LOM for better understanding the LOM decomposition during the AOP and ARP phases.
3. EXPERIMENTAL

3.1 Chemical Reagents and Landfill

All the reagents used in the thesis research were at least analytical grade, except as noted. The leachate sample was collected from an active municipal landfill in Pennsylvania (PA), United States. The landfill operation began in 1988. The daily volume of the leachate generated is now approximately 400 m$^3$. Once collected, the leachate was delivered to Montclair State University’s Innovative Water Treatment and Reuse Laboratory and stored at 4 °C in a cold room until use. In order to minimize the influence of particulate matter, the leachate samples were filtered using 0.45 µm Durapore® PVDF membrane filters (Millipore, Bedford, MA, USA). Basic quality of the raw leachate is as follows: pH = 8.50; COD = 3160 mg/L; UV$_{254}$ abs. = 12.700 cm$^{-1}$; DOC = 713 mg/L. The initial pH suggests that the leachate was a mature one. Ultrapure water (> 18.0 MΩ·cm) produced from a Milli-Q® water purification system (Milli-Q Direct 8) was used to prepare stock solutions or make dilution.

3.2 The Fenton Treatment Experiments

The Fenton treatment was first implemented to treat the raw leachate. In a typical run, the initial leachate pH was adjusted to a designated level using 2 N sulfuric acid (H$_2$SO$_4$, Fisher Scientific, Pittsburgh, PA, USA). The Fenton treatment was carried out in 1 L glass beakers on magnetic stirrers at room temperature (20 ± 1 °C) and atmospheric pressure.

Once an appropriate weight of granular ferrous sulfate (FeSO$_4$·7H$_2$O, Fisher Scientific, Pittsburgh, PA, USA) or a designated volume of FeSO$_4$ stock solution together with hydrogen peroxide solution (H$_2$O$_2$, 30% w/w, Fisher Scientific, Pittsburgh, PA, USA) was added, the treatment was initiated. The rapid mixing guaranteed a completely mixed state over the
treatment. After 30 minutes, the reaction was stopped through adjustment of the pH to 9.0 using 5 N potassium hydroxide solution (KOH, Fisher Scientific, Pittsburgh, PA, USA). At such a pH condition, almost all the formed Fe$^{3+}$ and any remaining Fe$^{2+}$ was transformed to iron (hydro)oxide solids. The following precipitation and settling proceeded for 24 hours to allow for the sedimentation of the most iron (hydro)oxide particles and the depletion of any residual H$_2$O$_2$ via self-decomposition. Thereafter, the supernatant was decanted and filtered through 0.45 µm PVDF membranes before further treatment or analyses.

The Fenton treatment process was optimized using the procedure reported in detail elsewhere (Yang Deng, 2007a; Chanil Jung, Yang Deng, Renzun Zhao, & Kevin Torrens, 2017). First, the optimal pH was determined by fixing dimensionless oxidant demand (DOD) at 0.125 and [H$_2$O$_2$]:[Fe$^{2+}$] at 4 and varying the initial pH at 4.50, 5.00, 5.50, 6.00, and 6.50. DOD is defined as the equivalent ratio of the added oxidant to the initial COD (COD$_0$) (Eq.(3-1)) (Chanil Jung et al., 2017).

$$DOD = \frac{\text{Chemical equivalent of oxidant}}{\text{Chemical equivalent of COD}_0}$$  \hspace{1cm} (3-1)

Specifically, for the Fenton treatment, DOD can be computed as follows (Eq.(3-2)) (Jung et al., 2017).

$$DOD = \frac{0.471H_2O_2}{COD_0}$$  \hspace{1cm} (3-2)

DOD was used in order to facilitate the performance comparison of chemical oxidation technologies for treatment of different leachate samples with different initial COD. After the optimal pH was determined, another set of the Fenton treatment tests were conducted to evaluate the effect of [H$_2$O$_2$]:[Fe$^{2+}$] by fixing the pH at the best level and DOD at 0.25 and varying [H$_2$O$_2$]:[Fe$^{2+}$] at 1, 2, 3, 4, and 5. The results allowed for determination of the most favorable
[H$_2$O$_2$]:[Fe$^{2+}$]. Finally, the effect of the Fenton reagents’ doses was assessed by fixing pH and [H$_2$O$_2$]:[Fe$^{2+}$] at their respective optimal levels and varying DOD at 0.125, 0.25, 0.50, 1.0, and 1.50.

3.3 ARP Treatment Tests

ARP treatment was carried out in a quartz photochemical reactor (model 7880-60, ACE Glass) that was integrated with a low-pressure UV lamp (15W) and had a treatment capacity of 15 mL. Before the treatment, leachate was continuously bubbled with N$_2$ gas for 15 minutes to repel DO and then an appropriate weight of sodium sulfite (Na$_2$SO$_3$, Millipore Sigma, Burlington, MA, USA) was added to the leachate. The treatment was launched once the UV lamp was turned on. In this study, the sulfite dose was quantified using a ratio of molar concentration of sulfite to initial UV$_{254}$ abs. (i.e., [SO$_3^{2-}$]/initial UV$_{254}$ abs.). In order for laboratory safety, the reactor was completely covered by aluminum foil throughout the treatment. The treatment proceeded for 4 hours, within which all the added sulfite was depleted. Absence or presence of sulfite was confirmed using the following method. A volume of 0.1 ml Betadine solution (10% Povidone-iodine) was added to 15 mg/L starch solution ((C$_6$H$_{10}$O$_5$)$_n$, Sigma-Aldrich, Burlington, MA, USA) before the solution color became blue. After one milliliter of treated sample was added to the mixed solution, sulfite remained if the solution became white with foggy appearance; otherwise, sulfite was depleted. In the tests to evaluate effect of solution pH, initial pH was adjusted with 10 mM KOH to 8.5, 9.0, 9.5, 10.0, and 10.5, while [SO$_3^{2-}$]/initial UV$_{254}$ abs. was fixed at [SO$_3^{2-}$]/initial UV$_{254}$ abs. = 0.8. Following the treatment, the treated leachate was collected for analyses. In the tests to assess the effect of sulfite dose, pH was fixed at 9.5, while [SO$_3^{2-}$]/initial UV$_{254}$ abs. was varied at 0.03, 0.15, 0.38, 0.75, 1.50, 3.00, 6.00, and 9.00.
3.4 Analyses

Leachate pH was measured with a Thermo Scientific Orion 5-Star Plus pH meter. COD was spectrophotometrically determined after chemical digestion using HACH test kits (20–1,500 mg/L range, HACH, Loveland, CO, USA). UV absorbance and DOC were analyzed using a DR5000 UV/Vis spectrophotometer (HACH, Loveland, CO, USA) and a total organic carbon (TOC) analyzer (TOC-L-CPH, Shimadzu Corp., Kyoto, Japan), respectively. In order to determine molecular weight distribution of LOM, 200 mL leachate was successively filtered using different MW cut-off ultrafiltration membranes (100 and 10kDa, Millipore, Billerica, MA) in a 200 mL Amicon ultrafiltration stirred cell (Amicon 8200, Millipore, Billerica, USA) under the pressure of nitrogen gas. In order to determine hydrophobic and hydrophilic fractions of LOM, the samples were isolated into fulvic acid (FA), humic-acid (HA) and hydrophilic (HPI) fractions using a solid phase extraction (SPE) method (J. B. Christensen, Jensen, Grøn, Filip, & Christensen, 1998). This method is briefly introduced as below. Following filtration with 0.45 µm nitrocellulose microfiltration membranes (Millipore), leachate was acidified to pH 2.0 using 10 M hydrochloric acid (HCl). At pH 2.0, HAs precipitated and then were separated with centrifugation at 4,500 rpm for 15 min. The HA isolates were collected and then re-dissolved in 0.05 M NaOH. The liquid after centrifugation went through nonionic Supelite™ DAX-8 resins (20-60 mesh), to which FAs were absorbed (Thurman & Malcolm, 1981). DOM remaining in the liquid was HPIs. Ammonia nitrogen (NH$_3$-N), nitrate nitrogen (NO$_3$-N), and nitrite nitrogen (NO$_2$-N) were analyzed using the HACH Ammonia Nitrogen Reagent Set (0.02-2.50 mg/L NH$_3$-N, the Nessler method), the HACH test kits (NitraVer 5 Nitrate Reagent Powder Pillows, 0.3-30.0 mg/L NO$_3$-N), and the HACH test kits (NitriVer® 3 Nitrite Reagent Powder Pillows, 0.002-0.300 mg/L NO$_2$-N, the Diazotization method), respectively. All the treatment tests were
performed, at least, in duplicate. All the analytical results reported here represent the mean of these replicate samples, while the error bars in figures are one standard deviation of these measurements.
4. RESULTS AND DISCUSSION

4.1 Treatment of Landfill Leachate with the Fenton Process

4.1.1 Effect of pH

Effect of leachate pH on the Fenton treatment of landfill leachate is shown in Figure 4-1. COD and UV$_{254}$ absorbance were used as aggregate indicators to evaluate the treatment efficiencies of LOM. Solution pH was varied between 4.5 and 6.5, while DOD was fixed at 0.125 and [H$_2$O$_2$]:[Fe$^{2+}$] was maintained at 4:1. DOD was recently proposed as a dimensionless chemical dose for industrial wastewater treatment (C. Jung, Y. Deng, R. Zhao, & K. Torrens, 2017). It can facilitate comparison of the treatment performance of different chemical technologies for treatment of wastewater with different initial organic concentrations.

At DOD = 1.0, the added oxidant can theoretically remove all the COD in wastewater, provided that: 1) the oxidant is capable of totally oxidizing the organic contaminants; 2) chemical oxidation proceeds via electron transfer only; 3) the oxidant is not consumed by any side reactions such as reactions with co-existing chemical species; and 4) self-decomposition of the oxidant does not occur.

As seen, the residual COD stabilized at 1904-1942 mg/L at pH 4.50-5.00, but gradually increased to 2742 mg/L at pH 6.50. Residual UV$_{254}$ absorbance exhibited a similar pattern with pH. UV$_{254}$ absorbance after the Fenton treatment slightly decreased from 6.850 cm$^{-1}$ at pH 4.5 to 5.700 cm$^{-1}$ at pH 5.5, before dramatically raised to 11.750 cm$^{-1}$ at pH 6.5. Alleviation of organic matter in landfill leachate is ascribed to the formation of •OH through the Fenton chemistry. •OH is recognized as the most powerful oxidizing agent in engineered water treatment systems (Yang Deng & Zhao, 2015; C. Huang et al., 1993). •OH can attack organic molecules via different reaction mechanisms, such as radical addition, hydrogen abstraction, electron transfer,
and radical combination (G Tchobanoglous et al., 2014). These reactions can lead to the formation of carbon-centered radicals (R’), which can promptly and irreversibly react with DO as follows (Yang Deng & Englehardt, 2006).

\[ R' + O_2 \rightarrow R(-H^+) + HO_2^- \]  
\[ R' + O_2 \rightarrow R-OO' \rightarrow R-O' \]

R’, R-OO’, and R-O’ produced can couple or disproportionate to form relatively stable intermediates, which may further react with •OH and DO to initiate further decomposition and even mineralization to produce inorganic water and CO₂.

Based on these findings, pH 5.0 was selected as the optimal pH level, considering that both COD and UV₂₅₄ absorbance remained at very low levels. The best pH would be used in the ensuing experiments. In the Fenton treatment of an industrial wastewater, the optimal pH typically exists, above or below which the organics removal efficiencies decline. The underlying mechanisms were comprehensively discussed (Yang Deng & Englehardt, 2006; Chanil Jung et al., 2017). A too acidic pH can disfavor the LOM removal in three aspects: 1) more H⁺ scavenges •OH (W. Tang & Huang, 1996); 2) the fraction of [Fe(H₂O)]²⁺ is increased with the pH decrease and the Fe(II) species sluggishly reacts with H₂O₂ (Gallard, de Laat, & Legube, 1998); and 3) a too low pH inhibits the reaction between Fe³⁺ and H₂O₂ to produce less Fe²⁺ and thus yield less •OH (Pignatello, 1992). On the other hand, LOM removal is also suppressed at a too high pH due to the five reasons: 1) Fe²⁺ can precipitate to form iron (hydro)oxides, particularly at pH > 5.0, which do not participate in the Fenton reactions (Bigda, 1995); 2) less H⁺ can inhibit decomposition of H₂O₂ in the Fenton process, so that less •OH is produced (Walling, 1975); 3) H₂O₂ self-decomposition is enhanced with the increasing pH, particularly at
pH > 5.0, to cause H\textsubscript{2}O\textsubscript{2} consumption; 4) the concentrations of CO\textsubscript{3}\textsuperscript{2-} and HCO\textsubscript{3}\textsuperscript{-} increase with pH, both of which are scavengers for •OH; and 5) the reduction potential of •OH declines with the increasing pH (E\textsubscript{0}=2.8 V at pH 0 vs. E\textsubscript{14}=1.95 V at pH 14) (Vogelpohl & Kim, 2004).

**Figure 4-1** Effect of pH on residual COD and UV\textsubscript{254} absorbance after the Fenton treatment of landfill leachate (Experimental conditions: initial COD = 3160 mg/L; initial UV254 absorbance = 12.700 cm\textsuperscript{-1}; DOD = 0.125; and [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] = 4:1)

4.1.2 Effect of [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}]

Effect of [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] on the Fenton treatment of LOM is shown in **Figure 4-2**. In this study, [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] was varied from 1:1, 2:1, 3:1, 4:1, to 5:1, while pH was adjusted to the optimal level (i.e., 5.0) and DOD was fixed at 0.125. As seen, residual COD and UV\textsubscript{254} absorbance had nearly same patterns with [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}]. As [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] increased from 1:0 to 2:0, residual COD and UV\textsubscript{254} absorbance declined from the initial 2095 mg/L and 7.700 cm\textsuperscript{-1} to 1661 mg/L and 5.095 cm\textsuperscript{-1}, respectively. When [H\textsubscript{2}O\textsubscript{2}]:[Fe\textsuperscript{2+}] increased from 2:1 to 4:1, COD
narrowly varied between 1661 and 1982 mg/L, while UV$_{254}$ absorbance ranged within 5.095 and 6.910 cm$^{-1}$. As [H$_2$O$_2$]:[Fe$^{2+}$] further increased to 5:1, residual COD and UV$_{254}$ absorbance increased to 2703 mg/L and 10.550 cm$^{-1}$, respectively. The optimal [H$_2$O$_2$]:[Fe$^{2+}$] was selected at 2:1, which would be used in the following experiments.

In the Fenton chemistry, the Fenton reagents (i.e., Fe$^{2+}$ and H$_2$O$_2$) are the both reactants to generate •OH as shown in Eq.(1-1). However, either of the both is also •OH scavenger that can promptly react with •OH to produce less reactive species (Eq. (1-3) and (1-4)). Therefore, excessive Fe$^{2+}$ or H$_2$O$_2$ can rapidly consume •OH and thus lead to a low net yield of •OH.

![Figure 4-2](image-url)

**Figure 4-2** Effect of [H$_2$O$_2$]:[Fe$^{2+}$] on residual COD and UV$_{254}$ absorbance after the Fenton treatment of landfill leachate (Experimental conditions: pH = 5.00; initial COD = 3160 mg/L; initial UV254 absorbance = 12.700 cm$^{-1}$; and DOD = 0.125)
4.1.3 Effect of the Fenton reagents’ doses

Effect of the Fenton reagents’ doses on the LOM removal is illustrated in Figure 4-3. Hydrogen peroxide was dosed at DOD = 0.125, 0.25, 0.50, 1.0, and 1.50, while pH and $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$ were fixed at their respective optimal levels (i.e., pH 5.0 and $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}] = 2:1$). DOD indirectly reflects the dose of $\text{H}_2\text{O}_2$ that is a key factor affecting the maximum theoretical yield of $\bullet\text{OH}$. As shown in Figure 4-3, as DOD was increased from 0.125 to 1.0, residual COD declined from 1988 to 700 mg/L. With the further increasing DOD to 1.50, residual COD was not obviously altered. On the other hand, the remaining UV$_{254}$ absorbance slightly increased from 7.000 to 2.884 cm$^{-1}$ with the DOD increase from 0.125 to 0.25, then sharply decreased to 3.180 at DOD = 1.0. The further DOD increase from 1.0 to 1.5 did not translate into any significant alleviation of UV$_{254}$ absorbance, suggesting that the residual LOM is recalcitrant to chemical oxidation (C. Jung et al., 2017). Based on the removal patterns of COD and UV$_{254}$ absorbance, the optimal DOD of 1.0, rather than the DOD of 1.5, was chosen. The DOD of 1.5 was not selected because: 1) the DOD increase from 1.0 to 1.5 did not lead to a significant increase in the removals of COD or UV$_{254}$ absorbance; and 2) too much iron sludge required for disposal is produced at a too high DOD.

In Section 4.1, three successive steps were used to optimize the Fenton treatment of landfill leachate. That is, the most favorable pH, $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}]$, and DOD were sequentially determined. In this study, the optimal operational conditions for the Fenton treatment are as follows: pH 5.0, $[\text{H}_2\text{O}_2]/[\text{Fe}^{2+}] = 2:1$, and DOD = 1.0. At the optimal experimental conditions, the maximum removal efficiencies of COD and UV$_{254}$ absorbance were achieved at 78% and 75%, respectively. The leachate treated by the Fenton process would be used as influent for advanced reduction treatment.
4.2 ARP

4.2.1 Feasibility studies of ARP for degradation of the Fenton-treated LOM

In Section 4.2, all the influent for the ARP treatment was the landfill leachate treated by the Fenton process at the optimal experimental conditions. In this study, we did not use COD, because we found that a positive error in the measured COD always existed after the ARP treatment. The error might be caused by the formation of unwanted reducing species that enabled an overestimation of COD during the COD tests.

The initial effort was made to examine whether ARP can provide a significant removal for LOM in the Fenton-treated leachate. The difference after different treatment conditions might be demonstrated from the leachate color, as shown in Figure 4-4. Leachate, before and after
filtration, appeared brown due to the presence of considerable humic-like LOM. Following the Fenton treatment, leachate color was closely colorless, suggesting a substantial alleviation in chromophores due to the LOM degradation. The color was not significantly altered after the ARP treatment.

Comparison of LOM at different experimental conditions is shown in Figure 4-5. The group “before treatment” represents the Fenton-treated leachate with the COD of 273 mg/L and UV$_{254}$ absorbance of 2.450 cm$^{-1}$. In the control with UV irradiation only in the absence of air, UV$_{254}$ absorbance dropped to 1.795 cm$^{-1}$. Under the UV irradiation of organic pollutants (R), excited substrates (R*) are generated (Eq. (4-3)), which can subsequently initiate the reactions between R* and certain UV-quenching moieties in LOM.

$$R + h\nu \rightarrow R^*$$ (4-3)

A slightly lower UV$_{254}$ absorbance (1.695 cm$^{-1}$) was observed in the group under the UV irradiation in the presence of air. When the leachate was open to air during irradiation, DO was maintained at a relatively high level due to the O$_2$ transfer from air to leachate. More reactive oxygen species (ROS) might be produced as shown in Eq. (4-1) and (4-2), which can lead to further degradation of LOM (Yang Deng, 2009), as evidenced by the lower UV$_{254}$ absorbance.

In contrast, the lowest UV$_{254}$ absorbance (i.e., 1.163 cm$^{-1}$) was achieved in the ARP group with a removal of 53% in comparison to the UV$_{254}$ absorbance of the Fenton-treated leachate. The better removal was ascribed to the generation of highly reducing hydrated electrons (Eq. (1-8)). The encouraging results indicate that e$_{aq}^-$ can react with certain UV-quenching functional groups and thus initiate the further degradation of LOM. Hydrated electrons react with organic molecules typically through an electron transfer mechanism (Cui, Gao, & Deng, 2020b).
However, we did not see any significant reduction in DOC in the UV irradiation group in the absence or presence of air or in the ARP group, indicating that LOM in the Fenton-treated leachate could not be mineralized by UV irradiation only or $e_{aq}$-driven reduction.

Figure 4-4 Comparison of different samples to demonstrate the difference in the color after each process (1: raw landfill leachate; 2: landfill leachate sample filtered with 0.45µm membrane; 3: leachate sample after the Fenton treatment at the optimal conditions; and 4: landfill leachate sample after the Fenton and ARP treatment ($[SO32-]/$initial UV$_{254}$ absorbance, pH = 9.5, and irradiation duration = 4 hours))
Figure 4-5 Comparison of residual COD and UV<sub>254</sub> absorbance for treatment of the Fenton-treated landfill leachate at different conditions (Before treatment: the leachate after the Fenton treatment at the optimal conditions; UV irradiation (no air): the control for ARP; UV irradiation (air): the group at the identical conditions with the control, except that the system was open to air during the UV irradiation; and ARP: the UV/SO<sub>3</sub><sup>2-</sup> system with the following conditions: [SO<sub>3</sub><sup>2-</sup>] / initial UV<sub>254</sub> absorbance = , pH = 9.5, and irradiation duration = 4 hours)

4.2.2 Effect of pH

Effect of pH on residual UV<sub>254</sub> absorbance after the ARP treatment is shown in Figure 4-6. As pH increased from 8.5 to 10.5, residual UV<sub>254</sub> absorbance narrowly varied between 1.220 and 1.300 cm<sup>-1</sup>. The finding suggests that the alleviation of UV<sub>254</sub> absorbance appeared to be independent of pH, which is not in agreement with the effect of pH in other ARP studies (Cui et al., 2020b). Generally, a more alkaline pH favors organic degradation during ARP because less H<sup>+</sup> scavenges e<sub>aq</sub> (Eq. (4-4) and (4-5)) at a higher pH to ensure more efficient utilization of e<sub>aq</sub> for the degradation of LOM (Buxton, Greenstock, Helman, & Ross, 1988).

\[
ed_{aq} + H^+ \rightarrow H\cdot \\
k = 2.3 \times 10^{10} \text{ M}^{-1}\cdot\text{s}^{-1}, \quad \text{(Buxton et al., 1988)} \]  

(4-4)
\[ e_{aq}^- + H^+ + H_2O \rightarrow H_2 + OH^- \quad k = 3.0 \times 10^{10} \text{ M}^{-1}\text{s}^{-1}, \quad \text{(Buxton et al., 1988)} \] (4-5)

However, the expected effect of pH was not observed in this study, likely because the generated \( e_{aq}^- \) at the lowest pH (i.e., pH 8.50) was sufficient for reactions with all the available UV-quenching functional groups in LOM. In this study, pH 9.5 was chosen for the following ARP experiments.

**Figure 4-6** Effect of pH on residual UV\(_{254}\) absorbance during the ARP treatment of the Fenton-treated landfill leachate (Experimental conditions: initial UV\(_{254}\) absorbance = 3.180 cm\(^{-1}\), [SO\(_3^{2-}\)]/initial UV\(_{254}\) absorbance = 0.8, and irradiation duration = 4 hours)

4.2.3 Effect of sulfite dose

Effect of sulfite dose on residual UV\(_{254}\) absorbance and DOC after ARP treatment is shown in **Figure 4-7**. Here sulfite dose is indicated as the ratio of molar sulfite concentration to the initial UV\(_{254}\) absorbance, i.e., [SO\(_3^{2-}\)]/initial UV\(_{254}\) absorbance. The parameter was proposed
similar to DOD, which would facilitate the comparison of ARP treatment of wastewaters with different initial UV$_{254}$ absorbance. Here, we select UV$_{254}$ absorbance, rather than COD used for DOD, in the definition of the sulfite dose, because UV$_{254}$ absorbance can be effectively mitigated in an ARP treatment, but COD could not be accurately measured after ARP, as mentioned above.

As seen, when $[\text{SO}_3^{2-}]/$initial UV$_{254}$ absorbance increased from 0.0 to 0.15, the remaining UV$_{254}$ absorbance sharply declined from 3.180 to 1.465 cm$^{-1}$, corresponding to a removal of 54%. With the increase of $[\text{SO}_3^{2-}]/$initial UV$_{254}$ absorbance to 0.38, UV$_{254}$ absorbance somewhat decreased to 1.163 cm$^{-1}$ (64% removal). However, at the $[\text{SO}_3^{2-}]/$initial UV$_{254}$ absorbance of 0.38-9.0, any significant change in the residual UV$_{254}$ absorbance was not observed. Meanwhile, DOC was measured at the different $[\text{SO}_3^{2-}]/$initial UV$_{254}$ absorbance. Obviously, DOC was not obviously decreased, again indicating that ARP cannot effectively mineralize LOM in the Fenton-treated leachate.

In order to further understand the alternation in the LOM characteristics, we measured specific UV absorbance (SUVA), which is defined as follows.

$$\text{SUVA} = \frac{\text{UV}_{254} \text{ absorbance}}{\text{DOC}} \times 100$$  \hspace{1cm} (4-6)

Here, SUVA indicates the UV absorbance at a specific wavelength per unit mass of DOC. It is a widely accepted surrogate parameter to characterize natural organic matter (NOM) in natural water. SUVA is also used to characterize UV quenching properties of LOM (Zhao et al., 2018). Generally, a SUVA of $>4.0$ L/g·m implies the presence of a majority of humic substances with aromatic and hydrophobic characters and with high molecular weight (MW), while a SUVA of $<2.0$ L/g·m suggests a high fraction of non-humic substances characterized with high aliphatic and low hydrophobic charterers and with low MW (Edzwald & Tobiason, 2010). A SUVA between
the two cut-off levels means that the organics are mixture of both humic and non-humic substances. As seen in Figure 4-8, the initial SUVA of 1.164 L/(mg·m) suggests that the LOM in the Fenton-treated leachate was mostly non-humic substances. When the [SO$_3^{2-}$]/initial UV$_{254}$ absorbance increased to 0.03, SUVA dramatically declined to 0.512 L/(mg·m), suggesting that ARP improved the hydrophilicity as a result of advanced reduction. As the [SO$_3^{2-}$]/initial UV$_{254}$ absorbance further increased to 9.0, SUVA was not markedly altered.

**Figure 4-7** Effect of sulfite dose on residual UV$_{254}$ absorbance and DOC during the ARP treatment of the Fenton-treated landfill leachate (Experimental conditions: initial UV$_{254}$ absorbance = 3.180 cm$^{-1}$, [SO$_3^{2-}$]/initial UV$_{254}$ absorbance = 0.00-9.00, pH 9.5, and irradiation duration = 4 hours)
Figure 4-8 Effect of sulfite dose on SUVA during the ARP treatment of the Fenton-treated landfill leachate (Experimental conditions: $[\text{SO}_3^{2-}] / \text{initial UV}_{254} \text{ absorbance} = 0.00-9.00$, pH 9.5, and irradiation duration = 4 hours)

4.2.4 Molecular weight distribution

LOM in different leachates was isolated into different MW groups using the ultrafiltration technique, including high (>100 kDa), medium (100-10 kDa), and low MW (<10 kDa) groups. UV$_{254}$ absorbance was measured for the different LOM fractions (Figure 4-9). The fractions of different MW groups in the overall LOM are shown in Figure 4-10. UV$_{254}$ absorbance of > 100 kDa, 100-10 kDa, and < 10 kDa was 0.900, 2.400, and 7.933 cm$^{-1}$, respectively, indicating that UV$_{254}$ absorbance in all the MW groups were significant. Particularly, the majority of UV absorbance at 254 nm (71%) originated from low MW groups. The finding agrees with the observation in SUVA (Figure 4-9) that SUVA (< 1.2164 L/(mg·m)) of the raw leachate suggests a high fraction of low MW compounds.
Following the Fenton treatment, the UV\textsubscript{254} absorbance of > 100 kDa, 100-10 kDa, and < 10 kDa was all alleviated to 0.283, 0.233, and 2.067 cm\(^{-1}\), respectively. The high MW group still remained the dominant fraction (80\%) in the overall LOM in terms of UV absorbance. These findings indicate that \textperiodcentered OH produced from AOP could effectively degrade LOM molecules in different MW groups, so that the remaining LOM molecules had simple structures.

After the ARP treatment (low dose), the UV\textsubscript{254} absorbance in the high MW slightly declined to 0.233 cm\(^{-1}\), with a removal of 17\%, indicating that e\(_{\text{aq}}\) truly reacted with UV-quenching moieties in the high MW molecules. Meanwhile, the UV\textsubscript{254} absorbance of the medium MW group went up to 0.617 cm\(^{-1}\), having an increase by 165\%. The significant improvement suggests that more UV-quenching moieties derived from the degraded high MW compounds than the ones that are transformed into the low MW group. For the low MW group, UV\textsubscript{254} absorbance went down to 0.600 cm\(^{-1}\), corresponding to a reduction of 71\%, indicating that e\(_{\text{aq}}\) preferentially reacted with UV quenching moieties in low MW molecules. Because substantial UV\textsubscript{254} absorbance was removed in the low MW group, the UV fraction of low MW group in overall LOM dropped to 41\%. In contrast, the portions of UV\textsubscript{254} from high and medium MW groups went up to 16\% and 43\%, respectively.

When the sulfite dose was further increased as shown in the “Fenton treatment + ARP (high dose)”, the UV\textsubscript{254} absorbance in the high MW group was not much different (0.283 cm\(^{-1}\)) from that at the ARP with a low sulfite dose. However, the UV\textsubscript{254} absorbance in the medium MW group dropped down to 0.217 cm\(^{-1}\), much lower than 0.617 cm\(^{-1}\) in the ARP (low sulfite dose), while the UV\textsubscript{254} absorbance in the low MW group increased to 0.917 cm\(^{-1}\), greater than 0.600 cm\(^{-1}\) in the ARP group (low sulfite dose).
Two lines of information can be obtained from the mentioned observations. First, ARP can further reduce the overall UV\textsubscript{254} absorbance, regardless of the low or high sulfite dose. Specifically, the initial UV\textsubscript{254} absorbance (2.583 cm\textsuperscript{-1}) in the Fenton-treated LOM was further reduced to 1.450 cm\textsuperscript{-1} and 1.417 cm\textsuperscript{-1} by ARP at the low and high sulfite doses, respectively. Therefore, $e_{aq}^{-}$ truly reacted with UV-quenching functional groups in the LOM compounds. Second, we did not see any reduction of the overall UV\textsubscript{254} absorbance between the low and high sulfite doses, but only noticed the re-distribution of UV\textsubscript{254} absorbance among the three MW groups. Therefore, the additional sulfite between the low and high sulfite dose appeared not to further reacted with UV-quenching moieties but could continue to degrade high MW LOM compounds into low MW.

**Figure 4-9** UV\textsubscript{254} absorbance in different MW groups of different landfill leachates (Raw leachate: the leachate before the Fenton treatment; Fenton treatment: the leachate treated by the Fenton process at the optimal condition (pH 5.0; DOD = 1.0; and $[\text{H}_2\text{O}_2] : [\text{Fe}^{2+}] = 2:1$); Fenton treatment + ARP (low dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at $[\text{SO}_4^{2-}]$:initial UV\textsubscript{254} abs. = 0.15 (pH 9.5); and Fenton treatment + ARP (high dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at $[\text{SO}_4^{2-}]$:initial UV\textsubscript{254} abs. = 6.00 (pH 9.5).
Figure 4-10 Fractions of different MW groups in the overall LOM in terms of UV$_{254}$ absorbance for different landfill leachates ((a) Raw leachate: the leachate before the Fenton treatment; (b) Fenton treatment: the leachate treated by the Fenton process at the optimal condition (pH 5.0; DOD = 1.0; and [H$_2$O$_2$]:[Fe$^{2+}$] = 2:1); (c) Fenton treatment + ARP (low dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at [SO$_4^{2-}$]:initial UV$_{254}$ abs. = 0.15 (pH 9.5); and (d) Fenton treatment + ARP (high dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at [SO$_4^{2-}$]:initial UV$_{254}$ abs. = 6.00 (pH 9.5).
4.2.5 Hydrophobic properties

UV$_{254}$ absorbance of hydrophobic and hydrophilic LOM was measured. Hydrophobic LOM included humic acid (HA) and fulvic acid (FA). As seen in Figure 4-11, UV$_{254}$ absorbance of HA, FA, and HPI was 0.315, 8.610, and 2.590 cm$^{-1}$, respectively, in the raw leachate. A majority of UV$_{254}$ absorbance (78%) was contributed from hydrophobic LOM (3% and 75% were from HA and FA, respectively). After the Fenton treatment, UV$_{254}$ absorbance of HA was almost constant, but UV$_{254}$ absorbance of FA and HPI dramatically declined to 1.372 and 0.714 cm$^{-1}$, respectively.

After the further treatment by ARP (the low dose), UV$_{254}$ absorbance of HA and FA declined to 0.098 and 0.350 cm$^{-1}$, while UV$_{254}$ absorbance of HPI almost remained constant, in comparison with the UV$_{254}$ absorbance in the Fenton-treated leachate. The findings clearly show that e$_{aq}^-$ readily reacted with certain UV quenching moieties in the both hydrophobic fractions (i.e., HA and FA), but was almost ineffective for alleviating UV absorbing properties of HPI. When the sulfite dose was considerably increased in ARP, we did not see any significant change in the overall UV$_{254}$ absorbance or the fractions of UV absorbance in hydrophobic and hydrophilic LOM.
Figure 4-11 UV$_{254}$ absorbance of HA, FA, and HPI of different landfill leachates (Raw leachate: the leachate before the Fenton treatment; Fenton treatment: the leachate treated by the Fenton process at the optimal condition (pH 5.0; DOD = 1.0; and [H$_2$O$_2$]:[Fe$^{2+}$] = 2:1); Fenton treatment + ARP (low dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at [SO$_4^{2-}$]:initial UV$_{254}$ abs. = 0.15 (pH 9.5); and Fenton treatment + ARP (high dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at [SO$_4^{2-}$]:initial UV$_{254}$ abs. = 6.00 (pH 9.5).
Figure 4-12 Fractions of FA, HA, and HPI in the overall LOM in terms of UV$_{254}$ absorbance for different landfill leachates: (a) Raw leachate: the leachate before the Fenton treatment; (b) Fenton treatment: the leachate treated by the Fenton process at the optimal condition (pH 5.0; DOD = 1.0; and $[\text{H}_2\text{O}_2]:[\text{Fe}^{2+}] = 2:1$); (c) Fenton treatment + ARP (low dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at $[\text{SO}_4^{2-}]:$initial UV$_{254}$ abs. = 0.15 (pH 9.5); and (d) Fenton treatment + ARP (high dose): the leachate treated by the Fenton process at the optimal conditions and then ARP at $[\text{SO}_4^{2-}]:$initial UV$_{254}$ abs. = 6.00 (pH 9.5).
5. SUMMARY, IMPLICATION, AND RECOMMENDATION

5.1 Major Findings

This thesis research focused on the combination of AOP and ARP for mitigation of LOM. Particularly, this study represents the first scientific and engineering attempt to apply ARP to leachate treatment for addressing LOM issues. Because leachate typically contains substantial UV quenching substances, the Fenton treatment, as the selected AOP in this study, was arranged prior to UV-based ARP in order to ensure a high utilization efficiency of the ARP treatment. Major findings from this thesis research are summarized as follows.

1. The Fenton treatment can effectively remove COD and UV$_{254}$ absorbance at the optimal conditions, exhibiting a high capability for abatement of LOM. Performance of the Fenton treatment relies heavily on operational conditions, including leachate pH, [H$_2$O$_2$]:[Fe$^{2+}$], and DOD. In this study, the optimal conditions were pH 5.0, [H$_2$O$_2$]:[Fe$^{2+}$] = 2:1, and DOD = 1, at which the maximum COD and UV$_{254}$ absorbance removal efficiencies reached 78% and 75%, respectively.

2. The Fenton treatment can remove a majority of LOM, thus playing a key role in the LOM removal. However, the Fenton process cannot reduce LOM to a very low level because the residual LOM is extremely recalcitrant to advanced oxidation. In this study, the remaining COD and UV$_{254}$ absorbance were still as high as 695 mg/L and 3.175 cm$^{-1}$, respectively.

3. UV/sulfite ARP is noticeably effective for abating UV$_{254}$ absorbance of the refractory LOM in the Fenton-treated landfill leachate, but it could not mineralize these LOM compounds, as evidenced by the low DOC removal.
4. In this study, the ARP treatment performance was not markedly influenced by leachate pH. However, the sulfite dose affected the removal of \( \text{UV}_{254} \) absorbance. The best \( \text{UV}_{254} \) absorbance removal (64\%) was achieved at a low sulfite dose (\([\text{SO}_3^{2-}] / \text{initial } \text{UV}_{254} \) absorbance to 0.38 = 0.38) with the remaining \( \text{UV}_{254} \) absorbance at 1.163 \( \text{cm}^{-1} \). But the further increase of \([\text{SO}_3^{2-}] / \text{initial } \text{UV}_{254} \) absorbance to 6.0 could not translate into any significant improvement in the \( \text{UV}_{254} \) absorbance.

5. MW distributions studies show that the Fenton process could simultaneously and effectively degrade high, medium, and low MW LOM. In the following ARP at a low sulfite dose, high MW LOM was degraded to cause the increase in the \( \text{UV}_{254} \) absorbance of the medium MW LOM, while \( \text{UV}_{254} \) absorbance of low medium LOM declined. But at high sulfite dose, though the removal of overall \( \text{UV}_{254} \) absorbance was not significantly changed, ARP re-distributed contributions of different MW groups in the \( \text{UV}_{254} \) absorbance. \( \text{UV}_{254} \) absorbance in the medium MW group dropped, while the \( \text{UV}_{254} \) absorbance of the low MW group increased.

6. Hydrophobic and hydrophilic characterization tests showed that the Fenton treatment could substantially reduce \( \text{UV}_{254} \) absorbance of HA, FA, and HPI. However, the ARP appeared to alleviate \( \text{UV}_{254} \) absorbance due to hydrophobic LOM (i.e., HA and FA), but marginally decompose HPI.

5.2 Implications to the Wastewater and Solid Waste Industry

This thesis research provides an innovative leachate treatment design empowered by the joint use of AOP and ARP for addressing LOM issues. The major findings have an immediate and profound impact upon the solid waste industry and society as a whole. AOP treatment of landfill leachate has been intensively studied in the industry. However, the LOM in AOP-treated
leachate is highly recalcitrant to further chemical oxidation. Application of ARP subsequent to AOP can effectively degrade LOM, particularly alleviation of UV$_{254}$ absorbance, which is caused by UV quenching substances.

Discharge of landfill leachate to municipal wastewater treatment plants is the most common leachate management option in the U.S. However, the UV blocking organics in leachate can significantly reduce the efficiency of UV disinfection. Given that over 20% of municipal wastewater treatment plants are using UV disinfection (the share is expected to rise), the UV quenching issue challenges the preferred practice. Undoubtedly, the treatment train comprising AOP and ARP provides a new design for surmounting the problem in the industry.

ARP may revolutionize the way leachate is treated due to the benefits unmatched by existing treatments. Mechanistically different from others, ARPs based on chemical reductive mechanisms aim to tackle the LOM issues that current practices cannot effectively or efficiently overcome. Meanwhile, the UV-based ARP can be easily accepted and deployed by the industrial community because of 1) commercially available UV equipment; 2) mature experience in UV system design, installation, operation, and maintenance; 3) potentially low costs (sodium sulfite < $0.40/lb); 4) non-toxic final products (sodium sulfate); and 5) no production of residuals required for disposal. This research was the first step toward the technology revolution and eventually finds a sustainable pathway to the issues confronted by our solid waste industry.

5.3 Recommendations

ARP is an emerging water and wastewater treatment technology. This thesis reports the first encouraging performance data on the ARP treatment of landfill leachate. This study deserves further investigation for development of the innovative leachate treatment technology. Recommendations are made below for the future studies.
1) Landfill leachate is a complex matrix with various pollutants. Beside LOM, others such as ammonia, nitrate, and certain toxic metals are present in landfill leachate. Fate of the other LOM constituents were not investigated. Possible variation of the leachate pollutants of concern can influence comprehensive evaluation of the technical feasibility of ARP for leachate treatment.

2) The joint AOP and ARP design was tested only with a landfill leachate. Because leachate quality is highly site-specific, more leachates from other landfill sites should be tested to demonstrate the technology viability.

3) In-depth mechanistic studies are needed to explore reactions of hydrated electrons and LOM at molecular levels. The fundamental information is essential to better utilize hydrated electrons for addressing LOM issues.

4) Effects of other operational factors on the ARP treatment need to be evaluated, such as UV irradiation intensity, reaction temperature, and dissolved oxygen.

5) Batch reactors were used in the thesis research. However, in realistic leachate treatment, a continuous flow reactor is more frequently used. ARP reactor design needs to be optimized.

6) Cost analyses are highly recommended for the future studies. The overall costs should include capital, operational, and maintenance expenses within an expected design lifetime. Results will provide key information on economic feasibility of the technology for landfill leachate treatment.
6. References


Zhao, R., Jung, C., Trzopek, A., Torrens, K., & Deng, Y. (2018). Characterization of ultraviolet-quenching dissolved organic matter (DOM) in mature and young leachates before and
