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Fe(VI) as a New Chemical Agent for Treatment of Landfill Leachate

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✓ Fe(VI) as a new chemical agent for treatment of landfill leachate. ✓

By

Christopher Gravesen

A Master's Thesis Submitted to the Faculty of

Montclair State University

In Partial Fulfillment of the Requirements

For the Degree of

Master of Science

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School College of Science and Mathematics

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Abstract

More than 50% municipal solid waste (MSW) is being disposed of within landfills in the United States. Despite many benefits, landfills inevitably produce highly contaminated wastewater – landfill leachate. Landfill leachate represents one of the most challengeable wastewaters in the world. Refractory organic matters and ammonia nitrogen are two major persistent traditional contaminants in landfill leachate. The contaminants ought to be properly removed prior to discharge into natural water resources.

Ferrate (FeO_4^{2-}) is a potential water treatment chemical agent in which iron is in its +6 oxidation state. In engineering practice, Fe(VI) can concurrently function as oxidant, coagulant, adsorbent and disinfectant. Up to date, Fe(VI) has been intensively studied for treatment of drinking water, wastewater, and ballast water. However, few attempts have been made to apply Fe(VI) for treatment of landfill leachate.

The overall objective of this thesis was to evaluate ferrate as a new oxidizing agent for treatment of landfill leachate, with an emphasis on removal of two major leachate contaminants – refractory organic matters and ammonia nitrogen. The central hypothesis is that Fe(VI) has an adequately high oxidation capacity to simultaneously degrade refractory organic matters and ammonia in landfill leachate through oxidation, thereby providing a viable alternative for traditional landfill leachate treatment methods. To achieve the overall objective, bench scale tests were conducted in batch mode to pursue four specific objectives: 1) to evaluate the effects of Fe(VI) dose and pH on COD removal and $\text{NH}_3\text{-N}$ removal; 2) to evaluate the effect of chloride on Fe(VI) treatment of landfill leachate; 3) to evaluate the effect of initial $\text{NH}_3\text{-N}$ on Fe(VI) treatment of landfill leachate; and 4) to determine N transformation pathway during Fe(VI) oxidation of $\text{NH}_3\text{-N}$ under different conditions.

Results show that Fe(VI) could simultaneously reduce COD and $\text{NH}_3\text{-N}$ under different experimental conditions. In this study, up to 60% COD and 70% $\text{NH}_3\text{-N}$ were removed. Generally, the treatment efficiency was increased with the increasing dimensionless oxidant demand (DOD). COD removal was due to Fe(VI)-induced oxidation and iron sludge-induced coagulation/adsorption, while ammonia was reduced as a result of Fe(VI) oxidation, in addition to volatilization at high pH. The oxidized $\text{NH}_3\text{-N}$ was oxidized to nitrogen gas and $\text{NO}_3^- \text{-N}$. High pH and high DOD appeared to favor the $\text{NO}_3^- \text{-N}$ formation. At any condition, little $\text{NO}_2^- \text{-N}$ was produced. Cl^- (2,100 - 14,880 mg/L) played a complex role in the treatment. It somewhat increased COD removal at pH 4-5, but slightly slowed down the COD reduction at pH 9. At pH 4-5, a low Cl^- level (2,100 – 5,000 mg/L) could inhibit the $\text{NH}_3\text{-N}$ removal; however, more Cl^- improved $\text{NH}_3\text{-N}$ reduction at a high Cl^- range (5,000 – 14,880 mg/L). In contrast, at pH 9, more Cl^- favored the $\text{NH}_3\text{-N}$ removal. More initial $\text{NH}_3\text{-N}$ could compete with COD

for Fe(VI), thereby inhibiting the COD removal. Our results provide valuable information regarding the treatability of Fe(VI) for landfill leachate, and demonstrate that Fe(VI) is a potential treatment chemical for landfill leachate.

**FE(VI) AS A NEW CHEMICAL AGENT FOR TREATMENT
OF LANDFILL LEACHATE**

A THESIS

Submitted in partial fulfillment of the requirements

For the degree of Master of Science

By

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Montclair, NJ

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CHAPTER 1. INTRODUCTION

1.1 Production of Landfill Leachate and Its Treatment

1.1.1 Landfill and landfill leachate

The amount of municipal solid waste (MSW) has increasingly grown over the past decades, as a result of the exponential population, social civilization growth, and changes in the productivity and consumption habits (Foo et al. 2009). For example, the global municipal solid waste production went up from 1.3 to 1.7 billion tons from 1994 to 2008, corresponding to an increase of 31.1 %. In the United States, the total weight of MSW produced in 2010 was almost 250 million tons. Since landfills were used in 1960s, it remained the dominant MSW disposal method, relative to incineration and recycling, in the United States and many other countries in the world partially due to an economic perspective (Schiopu et al. 2010).

Landfill is defined as a land disposal site employing an engineered method of disposing of solid waste on land in a manner that minimizes environmental hazards by spreading the solid waste on the smallest practical volume. Despite many benefits, landfills bring two major negative environmental impacts: emission of greenhouse gases and production of landfill leachate. The former can contribute to global warming, while the latter has a potential to contaminate the sites nearby landfills. Landfill leachate is an extremely contaminated wastewater formed from landfills due to different actions such as rainwater percolation, biochemical, chemical and physical reactions, and inherent moisture content of the waste (Renou et al. 2008). Generally speaking, more precipitation can lead to more leachate, whereas higher evaporation causes less leachate. With certain hazardous chemicals, landfill leachate, if not properly treated or managed, can pollute soil, groundwater and surface water underlying or around landfills. Ezyske and Deng (2012) reported that ~25% of Superfund sites in the State of New Jersey, which has the most Superfund sites in the United States, were caused by landfill leachate.

1.1.2 Chemical composition of landfill leachate

Species and strength of pollutants in MSW landfill leachate are highly influenced by landfill age. Generally, leachate can be grouped into young and old (mature) leachates that are produced from landfills with < 5 and ≥ 5 years, respectively. Young leachate is produced in the acidic phase of landfills under which most organic wastes are anaerobically degraded into highly biodegradable organic acids. Old leachate is formed during methanogenic phase of landfills in which organic wastes are further degraded into neutral or weakly basic recalcitrant organic compounds, and methane starts to be produced.

Composition of landfill leachate is extremely complex. Kjeldsen et al. (2002) categorized them into 4 different groups: 1) dissolved organic matter (DOM); 2) inorganic macrocomponents, 3) heavy metals and 4) xenobiotic organic compounds.

- 1) DOM: DOM is the products of anaerobic degradation of organic fraction in MSW, as quantified by chemical oxygen demand (COD) or total organic carbon (TOC). In young leachate, biodegradable organic acids are dominant, such as volatile fatty acids. In old leachate, the major fraction of DOM is biologically recalcitrant humic-like substances.
- 2) Inorganic macrocomponents: they are cations and anions that may largely exist in landfill leachate, such as calcium (Ca^{2+}), magnesium (Mg^{2+}), sodium (Na^+), potassium (K^+), ammonium (NH_4^+), ferrous ion (Fe^{2+}), manganese (Mn^{2+}), chloride (Cl^-), sulfate (SO_4^{2-}) and bicarbonate (HCO_3^-).
- 3) Heavy metals: the heavy metals may include cadmium (Cd^{2+}), chromium (Cr^{3+}), copper (Cu^{2+}), lead (Pb^{2+}), nickel (Ni^{2+}) and zinc (Zn^{2+}). The heavy metals have a high concentration in young leachate because most of them are much more soluble in the acidic condition of young leachate. However, their concentrations may be significantly reduced in neutral or weakly basic old leachate.
- 4) Xenobiotic organic compounds: different from DOM, they are the compounds originally existing in MSW, rather than the byproduct of solid waste degradation. Xenobiotic organic compounds typically have high toxicity though their levels are much less than concentrations of the aforementioned DOM. Typical xenobiotic organic compounds in landfill leachate include aromatic hydrocarbons from household and industries, phenols, chlorinated aliphatics, pesticides and plastizers.

The typical compositions of untreated sewage and landfill leachate are summarized in Table 1.1. As seen, COD and $\text{NH}_3\text{-N}$ are two major pollutants in landfill leachate, compared with untreated sewage. Both of them represent DOM and ammonia in landfill leachate. COD in young leachate is biodegradable, and can cause oxygen depletion if leachate is directly discharge into natural water systems. COD in old leachate is refractory, and cannot be degraded by traditional wastewater treatment plants. NH_3 in leachate is so high that it may directly kill certain aquatic life. Certainly, NH_3 also contributes to the nutrient nitrogen into water, if it is not effectively removed prior to discharge, and can cause eutrophication in surface water. In addition, various metals are found in leachate.

Table 1.1 Comparison of chemical compositions of untreated sewage and landfill leachate (Kjeldsen et al., 2002; Reinhart and Grosh 1998)

Indicators	Untreated Sewage			Landfill Leachate	
	Weak	Medium	Strong	Young (<2 yrs)	Old (>5 yrs)
pH				4.5 – 7.5	6.6 – 7.5
TSS (mg/L)	100	220	350	200 – 2,000	100 - 400
Hardness (mg/L CaCO ₃)				300 – 10,000	200 - 500
Alkalinity (mg/Ls CaCO ₃)	50	100	200	470-57,850	
Cl ⁻ (mg/L)	30	50	100	0-77,000	
SO ₄ ²⁻ (mg/L)	20	30	50	8-1,400	
Total P (mg/L)	4	8	15	5 - 100	5-10
TDS (mg/L)	250	500	850	3,90-44,900	
COD (mg/L)	250	500	1,000	3,000- 60,000	100 - 500
BOD ₅ /COD	>0.9			0.6 - 1.0	0 - 0.3
TOC (mg/L)	80	160	290	1,500 - 20,000	80 - 160
NH ₃ -N (mg/L)	12	25	50	10 - 800	20-40
As (mg/L)				0.0002-1.6	
Ba ²⁺ (mg/L)				0.08-5	
Ca ²⁺ (mg/L)				0.0007-0.15	
Pb (mg/L)				0.005-1.6	
Hg (mg/L)				0.0002-0.05	
Ni (mg/L)				0.02-2.227	

1.1.3 Conventional treatment methods for landfill leachate

Three conventional treatment methods are commonly used for landfill leachate, including: 1) leachate transfer, which involves co-treatment of sewage and landfill leachate or leachate recirculation within landfills; 2) biological treatment, aerobic or anaerobic; and 3) physicochemical methods such as chemical oxidation, adsorption,

chemical precipitation, coagulation/flocculation, sedimentation/flotation and air stripping (Renou et al. 2008).

1.1.3.1 Leachate transfer. Leachate transfer includes two options. The first one is co-treatment of sewage and landfill leachate in public owned treatment works (POTWs). That is, landfill leachate is directly sent to POTWs, then mixed with sewage, and finally co-treated in the POTW treatment units. This treatment method fully utilizes the existing municipal resources, thus significantly reducing operational & maintenance (O&M) costs. However, two drawbacks have greatly limited the application of this co-treatment option. First, as a result of toxicity due to presence of certain toxic metals, organic compounds, or ammonia, the leachate introduction may disturb microbial operation within POTWs. Second, the effluent quality may not be satisfied with discharge regulations (Cecen et al. 2004). Diamadopoulos et al. (1997) used a sequencing batch reactor (SBR) for treatment of a mixed sewage and leachate solution (v:v = 9:1), and achieved 95% COD removal and 50% NH₃-N removal. However, the effluent COD and NH₃-N significantly increased with the increasing fraction of leachate in the mixed solution.

The other option is leachate recirculation, in which the collected leachate is pumped back to the landfill. Obviously, this method is also cost-effective. The recycled leachate increases the moisture of MSW in landfills, and distributes nutrients and enzymes between methanogens and solid/liquids (Bae et al. 1998). Therefore, the leachate COD and produced methane can be somewhat reduced (Bae et al. 1998). Furthermore, the recirculation converts a traditional landfill into a bio-landfill that significantly shortens the time required for stabilization from several decades to 2-3 years (Reinhart et al. 1996). However, leachate recirculation is also limited in a few aspects. First, recirculation inadequately removes leachate pollutants to meet with discharge regulations. Second, a too high recirculation rate can produce high concentration of organic acids that is toxic to methanogens in landfills (Ledakowic et al. 2004). Finally, a high volume of leachate for recirculation may generate problems such as moisture oversaturation, ponding, and acidic condition (Chan et al. 2002; San et al. 2001).

1.1.3.2 Biological treatment. Biological treatment is a common way to remove biodegradable organic compounds in leachate. The biodegradable organic matters in leachate can be removed by microbe-induced degradation (suspended or attached growth), which decompose the organic compounds into carbon dioxide and water under aerobic conditions, and into carbon dioxide and methane under anaerobic conditions (Lema et al. 1988). Especially when the ratio of BOD₅ to COD (BOD₅ : COD) is >0.5 in young leachates, POTWs can readily break down organic matters in leachate. However, a large number of refractory compounds existing in landfill leachate, which are humic-like substances, cannot be effectively treated by this option (Renou et al. 2008). This is the major drawback of biological treatment.

1.1.3.3 Physicochemical treatment. Different physicochemical treatment methods are used for landfill leachate with different emphasis on different pollutants in landfill leachate, including coagulation, precipitation, adsorption, air stripping, membrane processes and chemical oxidation (Renou et al. 2008).

Coagulation is to destabilize colloidal particles in water via compression of double electrical layers. In this process, the colloids come out of suspension in the form of floc/flakes, when a coagulant agent is added. Coagulant used include iron chloride (FeCl_3) and iron sulfate (FeSO_4). However, coagulation only efficiently removes turbidity in leachate, but reduce little COD and $\text{NH}_3\text{-N}$ (Renou et al. 2008). Precipitation is to add certain chemicals into water and transfer soluble contaminants of concern into settleable solids. This is a very common method to remove metals in water (Gosh et al. 2011). Similar to coagulation, precipitation is not effective for removal of COD and $\text{NH}_3\text{-N}$ in landfill leachate. Both coagulation and precipitation can eventually produce large amounts of undesirable sludge required for proper disposal.

Adsorption is a process to transfer soluble contaminants of concern to the phase of solid sorbent. Activated carbon (AC) is the most frequently used sorbent for landfill leachate. AC-based adsorption treatment method, via physical and chemical bonds, is recognized as the most efficient and promising fundamental approach in the wastewater treatment process (Foo et al. 2009). Prior studies demonstrated that AC was effective for organic and inorganic contaminants in leachate, especially COD and NH_3 . Foo et al. (2009) found that addition of granular activated carbon could remove 91-95% COD and ~40% $\text{NH}_3\text{-N}$ in leachate. However, costs associated with the expensive adsorption materials, in addition to regeneration of spent AC, challenge this option in practice.

Air stripping is a process that transfers volatile chemicals from water phase into air phase via continuous injection of air into water. Air stripping used for leachate treatment is targeted at removal of volatile organic compounds (VOCs) (e.g. BTEX) and ammonia. Marttinen et al. 2002 achieved 89 % NH_3 reduction at $\text{pH}=11$ and 20°C within 24 hours, and Cheung et al. 1997 also removed 93% ammonia from an old leachate. However, stripping is only a physical process that transfer pollutants from one phase to another, and cannot provide a true solution to destruct leachate pollutants, particularly toxic chemicals. Therefore, air pollution may be a byproduct of this treatment.

Chemical oxidation is a commonly used method for landfill leachate, in which oxidizing agents are added to landfill leachate, and chemically destruct leachate pollutants via redox reactions. Chemical oxidation is expected to decompose toxic organic compounds into less toxic molecules, and even into non-toxic inorganic carbon dioxide and water (mineralization), as long as oxidizing agents used are adequately strong. On the other hand, ammonia is anticipated to be removed through oxidation into nitrogen gas, nitrite or nitrate. Although many oxidizing agents have been attempted,

most of them such as KMnO_4 are not able to effectively oxidize recalcitrant organic matters in landfill leachate. Just few oxidants exhibit encouraging results, including ozonation and advanced oxidation processes (AOPs). Ozonation is to utilize ozone gas (O_3) as a major oxidant. Haapea et al. (2002) reported that O_3 alone was able to remove 50-70% of COD from landfill leachate. But O_3 can only be produced on-site, and has an extremely high operational cost. Moreover, the low solubility of O_3 usually causes a low utilization efficiency of added O_3 .

The other promising oxidation is AOP in which hydroxyl radicals ($\cdot\text{OH}$) are produced as the principal oxidizing agent. $\cdot\text{OH}$ has an unpaired electron, and thus exhibits an extremely high oxidation capacity. It has the highest redox potential of 2.9 V in engineered system, and can unselectively oxidize almost all the chemicals in water. Due to its very short lifetime (\sim a few ms), $\cdot\text{OH}$ can only be produced in-situ. Different methods can produce $\cdot\text{OH}$ in water, including UV/ O_3 , $\text{O}_3/\text{H}_2\text{O}_2$, UV/ H_2O_2 and Fenton process ($\text{H}_2\text{O}_2/\text{Fe}^{2+}$). Deng (2009) reviewed different AOPs for treatment of landfill leachate, and found that AOPs typically removed \sim 70% COD from a typical mature leachate. However, AOPs cannot remove any ammonia nitrogen via oxidation owing to the low rate constant of the reaction between $\cdot\text{OH}$ and NH_3 in water (Deng, 2009).

1.2 Ferrate Chemistry

Ferrate (Fe(VI)) is the iron anion $\text{Fe}^{\text{VI}}\text{O}_4^{2-}$ in which iron has a +6 valence. The most common ferrate salt is potassium ferrate (K_2FeO_4) that is relatively easily prepared (Sharma, 2002). Ferrate has a tetrahedral structure in solid crystals such as K_2FeO_4 according to an x-ray analysis (Kamachi et al. 2005). It has four equivalent oxygen atoms covalently bonded to the Fe^{6+} in the center. Fe (VI) is known to have a high redox potential of up to 2.2 V, thereby indicating an oxidative capability over that of O_3 . In water, different Fe(VI) species prevail at different pH, as shown in Figure 1.1. H_2FeO_4^- , HFeO_4^- and FeO_4^{2-} are the dominant Fe (VI) species at < 3.7 , $3.7-7.8$ and > 7.8 , respectively (Lee et al. 2004).

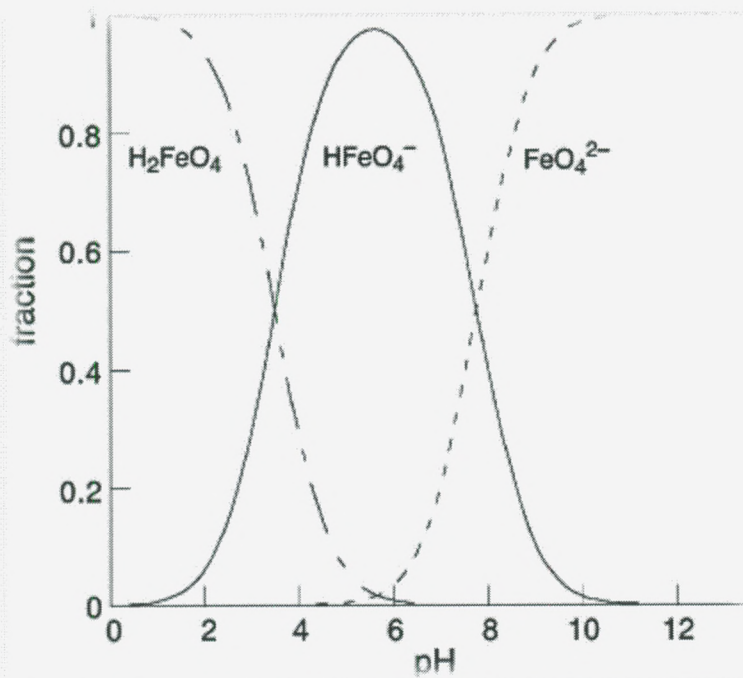


Figure 1.1 Distribution of the diprotonated ferrate (H_2FeO_4), monoprotated ferrate (HFeO_4^-) and nonprotonated ferrate (FeO_4^{2-}) in an aqueous solution at different pH. (From Kamachi et al. 2005)

Once Fe(VI) is added to water, oxidation first occurs so that Fe(VI) is reduced to Fe(III). As a result, organic compounds and reducing inorganic agents (e.g. ammonia) can be degraded. At acidic, basic and neutral conditions, Fe(VI)-induced oxidation half reactions can be summarized as follows.



Meanwhile, Fe(VI) plays an important role in disinfection. Subsequent to Fe(VI) oxidation, the produced Fe(III) immediately forms precipitate, especially at a neutral or weak condition, that can remove contaminants in water via coagulation and adsorption (Batarseh et al. 2007). Therefore, during Fe(VI) treatment, Fe(VI) functions as oxidant, disinfectant, coagulant, and adsorbent.

Fe(VI) has been studied for treatment of drinking water, sewage, and ballast water (Jiang et al., 2006). Results are encouraging. For example, Jiang et al. (2006) applied 0.55 mM Fe(VI) to treat sewage, and removed 38% COD within 2 hours at pH 5-7. The only study to apply Fe(VI) for treatment of landfill leachate was conducted at University of Central Florida (Batarseh et al. 2007). Batarseh et al. (2007) used liquid sodium ferrate to treat 12-yr-old and 20-yr-old leachates, and found that Fe(VI) could work over pH 2-9,

and the optimal pH was under 5.0. Their results showed that 1 g/L Fe(VI) removed 54 % COD from the 12 year old leachate, and 56 % COD from the 20 year old leachate. However, ammonia reduction and N transformation were not studied in this study.

Fe (VI) oxidation of ammonia (NH_3) was investigated by Sharma et al. (1998). The oxidation effectiveness was found to be determined by pH and molar ratio of Fe(VI) to ammonia. Within six hours, Fe(VI) removed up to 22 % ammonia, and the most favorable conditions were pH 9.0 and $[\text{Fe(VI)}] : [\text{NH}_3] = 1$. The finding is not in agreement with prior studies that actually showed a higher oxidation rate of ammonia with decreasing pH. However, little knowledge is known on Fe(VI) oxidation of ammonia in a very complex waste matrix such as landfill leachate.

CHAPTER 2. OBJECTIVES AND HYPOTHESIS

The principal objective of this study was to evaluate ferrate as a new oxidizing agent for treatment of landfill leachate, with an emphasis on removal of two major leachate contaminants – refractory organic matters and ammonia nitrogen. The central hypothesis is that Fe(VI) has an adequately high oxidation capacity to simultaneously degrade refractory organic matters and ammonia in landfill leachate through oxidation, thereby providing a viable alternative for traditional landfill leachate treatment methods. To achieve the overall objective, the following four specific objectives were pursued.

- 1) To evaluate the effects of Fe(VI) dose and pH on COD removal and NH₃-N removal;
- 2) To evaluate the effect of chloride on Fe(VI) treatment of landfill leachate;
- 3) To evaluate the effect of initial NH₃-N on Fe(VI) treatment of landfill leachate;
- 4) To determine N transformation pathway during Fe(VI) oxidation of NH₃-N under different conditions.

CHAPTER 3 EXPERIMENTS

3.1 Sample Collection

The leachate was collected from New Jersey Meadowland Commissions (NJMC) on July 2nd of 2012. The leachate was originally stored in an underground storage tank before it was pumped to Passaic Valley Sewerage Commission (Newark, New Jersey) (see Figure 3.1). The sample is a mixed leachate solution from Landfill 1-A and 1-E operated by NJWC. Once collected, the leachate samples were stored in two bottles with zero headspace and immediately transported to Environmental Geochemistry Laboratory (EGL) at Montclair State University (MSU). The sample was stored at 4 °C in a refrigerator prior to use. The sample collected is shown in Figure 3.2.

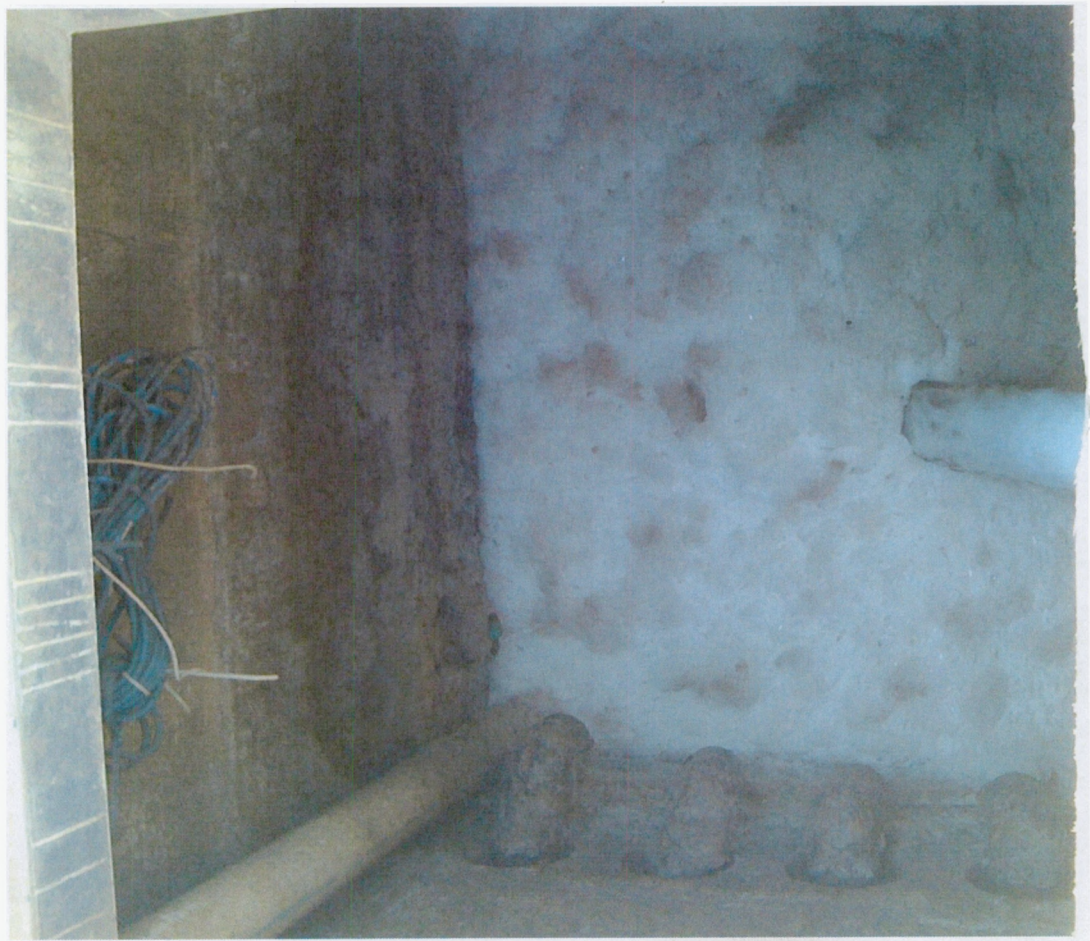


Figure 3.1 Landfill leachate collected from an underground storage tank operated by New Jersey Meadowland Commissions.

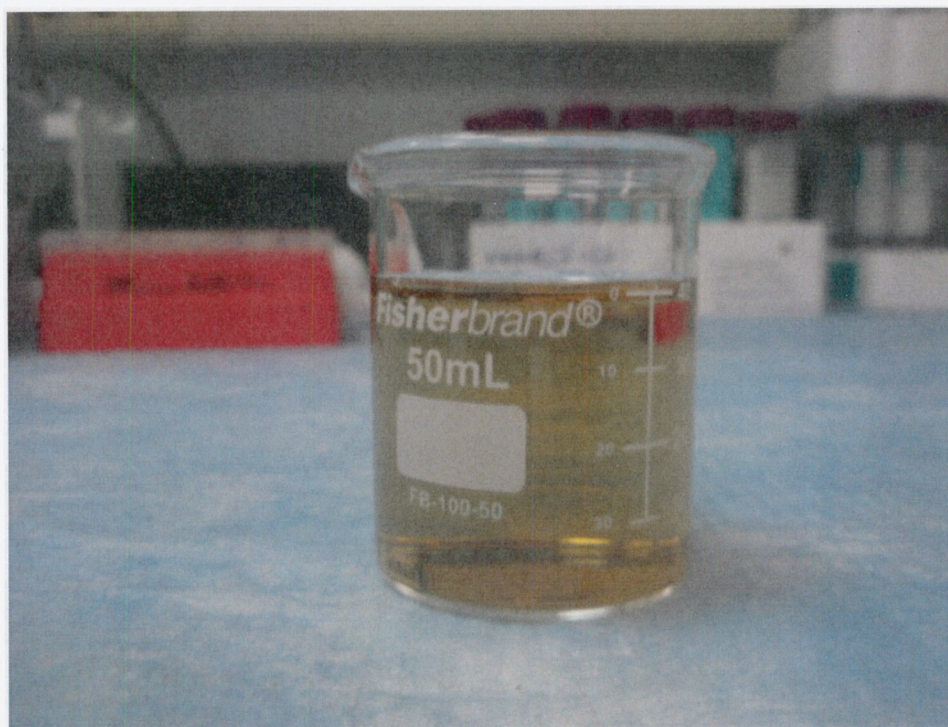


Figure 3.2 Landfill leachate collected for this study

3.2 Chemical Reagents

All the chemicals used were of analytical grade, at least, except as noted. Solution was prepared using deionized water from a Milli-Q water purification system (Elix®, Millipore). Potassium ferrate (K_2FeO_4) was purchased from Sigma-Aldrich (> 90%, Saint Louis, MO). All the other chemicals were obtained from Fischer Sci., including ammonia solution (Sigma-Aldrich, 28 % NH_3 in water, 99.99 % metal basis, Saint Louis, MO), silver nitrate ($AgNO_3$) (Alfa Aesar, 99.9 %, Ward Hill, MA) and sodium chloride ($NaCl$) (Alfa Aesar, > 99.0 %, Ward Hill, MA).

3.3 Experimental Procedure

Bench-scale batch tests were conducted in 200 mL beakers containing 50 mL landfill leachate. Reaction was started at least 1 hour after the leachate was moved out from the refrigerator, so that the leachate temperature went back to the room temperature. Beakers were placed on magnetic stirrers. A rapid stirring guaranteed the solution in a complete mixing state during reactions. Once certain amounts of K_2FeO_4 were added, the reaction was initiated. Solution pH was adjusted to a desirable level with concentrated H_2SO_4 and 1 N $NaOH$ solution, if needed. Because the used K_2FeO_4 powder contained some alkaline that ensured the stability of $Fe(VI)$, the leachate solution pH slightly increased subsequent addition of the dry K_2FeO_4 powder into leachate. Generally, $Fe(VI)$

decay was very slow at a basic condition, but became rapid at an acidic condition. Fe(VI) color was purple. Accompanied with the gradual consumption of Fe(VI), purple in the leachate faded, Fe(VI) was reduced to Fe(III), and then Fe(III) transformed to iron sludge. The rapid magnetic mixing was stopped until all the Fe(VI) was consumed (the purple disappeared). Two milliliters of uniformed mixed liquid sample was collected for COD analysis. And the remaining sample was kept in the beakers. Iron precipitates settled down to the bottom after a few hours. Thereafter, the clean supernatant was collected for further analysis.

In the tests to investigate the effect of chloride on the ferrate treatment, the chloride level in the leachate was adjusted as follows. To remove the chloride originally present in leachate, certain amounts of AgNO_3 was added to the leachate. A 2-min complete mixing allowed chloride to fully react with silver anion and then form insoluble AgCl precipitate. Thereafter, a 1-day settlement allowed all the produced AgCl particles to settle down to the bottom of the beakers. To increase the chloride level, appropriate amounts of NaCl was added to the leachate sample. A 10-min rapid mixing ensured that all the added NaCl crystals were fully dissolved.

In the tests to study the effect of ammonia nitrogen on the ferrate treatment, the $\text{NH}_3\text{-N}$ level in the leachate was adjusted as follows. To reduce the $\text{NH}_3\text{-N}$ originally present in the leachate sample, the leachate was being stirred continuously. Leachate $\text{NH}_3\text{-N}$ was measured every hour. The mixing was stopped until $\text{NH}_3\text{-N}$ dropped to a desirable level. To increase the $\text{NH}_3\text{-N}$ level in the leachate, certain volumes of ammonia solution were added to the leachate under a hood. After a 10-min rapid mixing, the beaker was sealed with parafilm, and stored at room temperature for 2 days to ensure that the solution reached a chemical equilibrium.

3.4 Analysis

Solution pH was measured using an Orion 5-Star pH/RDO/Conductivity Portable Meter. Generally, the absence or presence of Fe(VI) could be determined by solution color. Fe(VI) in water was purple. Once purple vanished, Fe(VI) largely decayed, and even depleted. COD (Reactor Digestion method), $\text{NH}_3\text{-N}$ (Nessler Method), $\text{NO}_2^- \text{-N}$ (Diazotization Method), $\text{NO}_3^- \text{-N}$ (Cadmium Reduction Method), and chloride (Mercuric Thiocyanate Method) were quantified using HACH test kits with a UV/Vis spectrophotometer (DR 5000, HACH). If the sample strength was beyond the limits of the test kits, the samples were properly diluted with Milli-Q water.

CHAPTER 4. RESULTS AND DISCUSSION

4.1 Characterization of Leachate Samples and Definition of Dimensionless Oxidant Dose (DOD)

Landfill leachate was a mixed sample of two leachates produced from Landfill 1-A and Landfill 1-E operated by New Jersey Meadowland Commissions. Basic water quality parameters of the leachate sample are shown in Table 4.1.

Table 4.1 Basic quality parameters of the used landfill leachate sample

pH	COD (mg/L)	NH ₃ -N (mg/L)	NO ₂ ⁻ (mg/L)	NO ₃ ⁻ (mg/L)	Cl ⁻ (mg/L)
8.06-8.10	1,321-1,372	385-454	0.05	0.0	2,100

The leachate pH was slightly over 8.00, thereby indicating that the leachate samples were typically mature.

In this study, we used Dimensionless Oxidation Dose (DOD) to represent chemical dose of Fe(VI). DOD is defined as Eq. (4.1).

$$DOD = \frac{\text{Equivalent of Fe(VI)}}{\text{Equivalent of COD}_0 + \text{Equivalent of NH}_3 - \text{N}_0} = \frac{\frac{C_{\text{Fe(VI)}}}{\text{Equivalent weight of Fe(VI)}}}{\frac{\text{COD}_0}{\text{Equivalent weight of COD}} + \frac{\text{NH}_3 - \text{N}_0}{\text{Equivalent weight of NH}_3 - \text{N}}} \quad (4.1)$$

Where, COD₀ and NH₃-N₀ are the initial COD and NH₃-N in landfill leachate; C_{Fe(VI)} is the mass dose of Fe(VI); and, equivalent weights of COD, NH₃-N, and Fe(VI) are 8, 4.7 (we assume that all NH₃-N is oxidized to N₂), and 18.7 g/eq., respectively. Theoretically, Fe(VI) at DOD = 1 just oxidizes all the COD and NH₃-N in the leachate, though it is not true in practice because a part of Fe(VI) is consumed by co-existing chemical species and/or Fe(VI) self-decay.

4.2 Effects of pH and DOD

Residual COD and NH₃-N during Fe(VI) treatment of landfill leachate at different pH and DODs are shown in Figure 4.1 and 4.2, respectively. As seen in Figure 4.1, COD in landfill leachate gradually decreased from 1,372 mg/L to 548 and 616 mg/L at pH 4-5 and 9, respectively, with the increasing DOD from 0 to 3. That is, the overall COD removal efficiencies were 60% and 55%, respectively. This finding demonstrated that an acidic condition slightly favored the COD removal over a weakly alkaline condition.

Moreover, the residual COD in leachate and iron sludge after Fe(VI) treatment at pH 4-5 and 9 are also presented in Figure 4.1. Because Fe(VI) oxidation and iron sludge coagulation/adsorption might both remove COD from leachate, it is essential to understand removal contributions of the both mechanisms. The difference between initial COD and overall residual COD represents the overall COD removal due to oxidation and coagulation/adsorption; the difference between initial COD and residual COD in the mixed leachate and sludge represents the COD removal due to oxidation; and the difference between overall residual COD and COD in the mixed leachate and sludge represents the COD removal due to coagulation/adsorption. As shown, the COD removal due to oxidation was slightly higher at pH 9 than pH 4-5, at any particular DOD except DOD = 3 under which COD removal due to oxidation were almost identical at the both pH. In contrast, pH 4-5 achieved slightly greater COD removal due to coagulation over the tested DOD range.

Meanwhile, $\text{NH}_3\text{-N}$ removal also dropped from 454 mg/L to 307 and 137 mg/L at pH 4-5 and pH 9, respectively, with the increasing DOD from 0 to 3, as shown in Figure 4.2. That is, at DOD = 3, $\text{NH}_3\text{-N}$ was removed by 32% and 70% at pH 4-5 and pH 9, respectively. At any specific DOD, the weakly alkaline condition reduced ~ 100 mg/L more $\text{NH}_3\text{-N}$ than the acidic condition. This finding was ascribed to different fractions of ammonia species at different pH. At low pH, more $\text{NH}_3\text{-N}$ existed in the form of NH_4^+ that is non-volatile anion in water. However, N in the very volatile NH_3 gradually became dominant in $\text{NH}_3\text{-N}$ with the pH increase. It is not surprising that more $\text{NH}_3\text{-N}$ was removed at higher pH because more $\text{NH}_3\text{-N}$ existed in the form of NH_3 that could be lost via volatilization. In contrast, volatilization could not be a major reason for the NH_3 loss at low pH, because the non-volatile NH_4^+ was the prevailing $\text{NH}_3\text{-N}$ species at low pH. Hence, the NH_3 removal at pH 4-5 was due to Fe(VI) oxidation alone. Overall, the NH_3 removal was as a result of Fe(VI) oxidation at an acidic condition, or oxidation and volatilization at an alkaline condition.

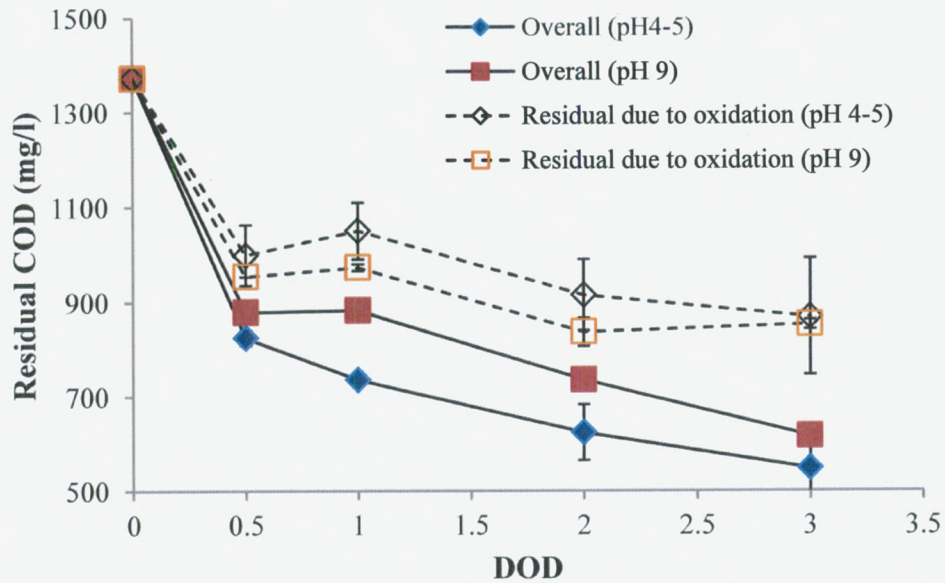


Figure 4.1. Residual COD vs. DOD during Fe(VI) treatment of landfill leachate at different pH.

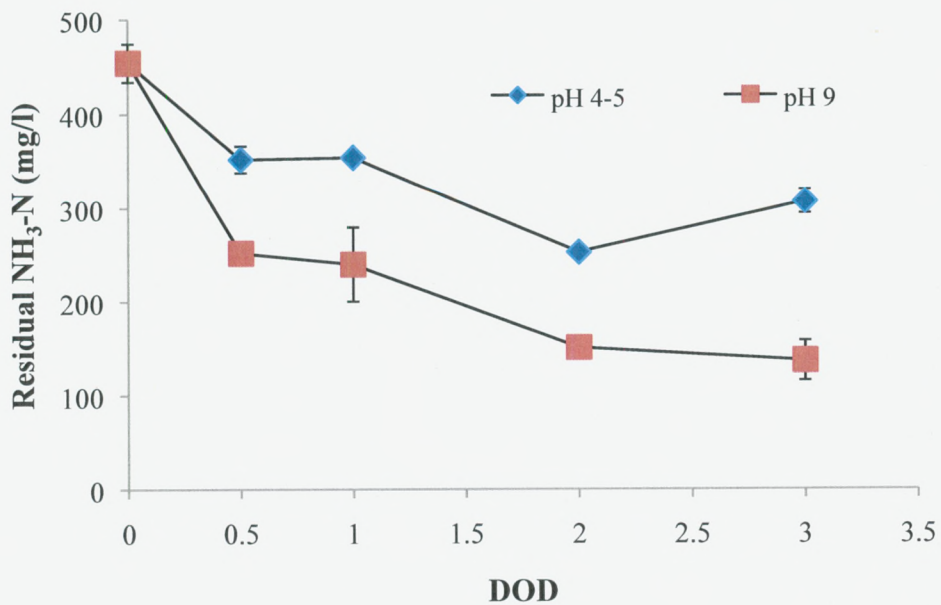


Figure 4.2. Residual NH₃-N vs. DOD during Fe(VI) treatment of landfill leachate at different pH.

Our finding revealed that NH₃-N was decreased partially by oxidation, as demonstrated in Figure 4.2. The transformation pathways of the N in NH₃-N were extremely interesting. Generally, NH₃ could be oxidized to N₂ gas that could escape into

air once produced, NO_2^- and NO_3^- . To quantitatively understand the transformation of $\text{NH}_3\text{-N}$ during Fe(VI) treatment of leachate, besides $\text{NH}_3\text{-N}$, nitrite nitrogen (NO_2^- -N) and nitrate nitrogen (NO_3^- -N) were also measured when Fe(VI) was used to treat the leachate. Results at pH 4-5 and pH 9 are shown in Figures 4.3 and 4.4, respectively. Of note, we did not directly measure the production of N_2 . Because organic nitrogen content was ignorable before and during treatment, $\text{N}_2\text{-N}$ could be estimated through the following equation.

$$\text{N}_2\text{-N}_m = (\text{NH}_3\text{-N}_0 + \text{NO}_2^- \text{-N}_0 + \text{NO}_3^- \text{-N}_0) - (\text{NH}_3\text{-N}_m + \text{NO}_2^- \text{-N}_m + \text{NO}_3^- \text{-N}_m) \quad (4.2)$$

Where, $\text{N}_2\text{-N}_m$ is the $\text{N}_2\text{-N}$ produced at $\text{DOD} = m$; $\text{NH}_3\text{-N}_0$, $\text{NO}_2^- \text{-N}_0$, and $\text{NO}_3^- \text{-N}_0$ are the initial ammonia nitrogen, nitrite nitrogen, and nitrate nitrogen, respectively; and, $\text{NH}_3\text{-N}_m$, $\text{NO}_2^- \text{-N}_m$, and $\text{NO}_3^- \text{-N}_m$ are ammonia nitrogen, nitrite nitrogen, and nitrate nitrogen of the leachate treated at $\text{DOD} = m$, respectively.

Generally speaking, as DOD increased, $\text{NH}_3\text{-N}$ decreased, accompanied with the increase of $\text{NO}_3^- \text{-N}$ and $\text{N}_2\text{-N}$. Regardless of pH and DOD, the $\text{NO}_2^- \text{-N}$ was almost negligible ($< 0.05 \text{ mg/L}$). Therefore, N_2 and NO_3^- were the major oxidation products of NH_3 and/or NH_4^+ . At any particular DOD, more $\text{NO}_3^- \text{-N}$ was produced at pH 9 than pH 4-5. For example, at $\text{DOD} = 3$, the $\text{NO}_3^- \text{-N}$ levels at pH 4-5 and 9 were 38.5 and 107.5 mg/L , respectively. However, this finding appeared not to be in agreement with the activities of Fe(VI) species at the both pH. The major Fe(VI) species at pH 4-5 (HFeO_4^-) is more active than the dominant Fe(VI) species (FeO_4^{2-}) at pH 9. The possible reason is that NH_4^+ (the prevailing form of $\text{NH}_3\text{-N}$) was more readily oxidized by Fe(VI) than NH_3 (the dominant form of $\text{NH}_3^- \text{-N}$).

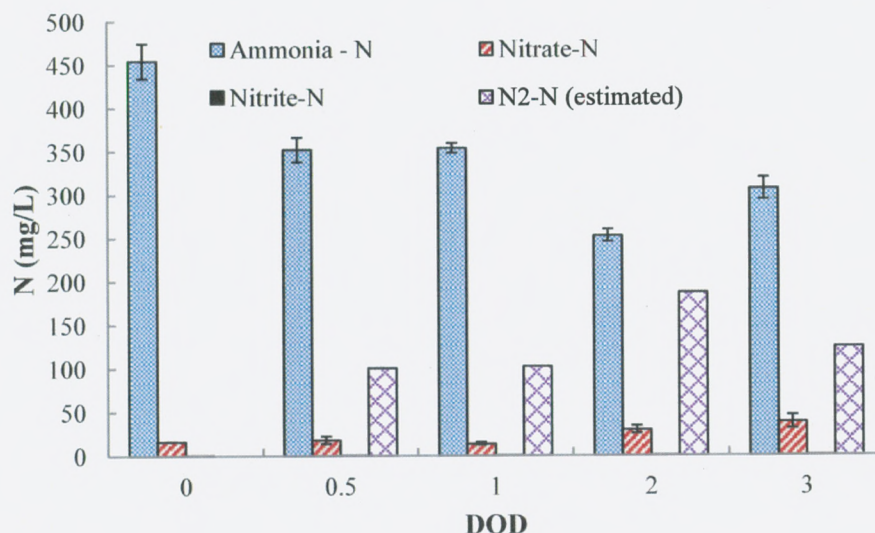


Figure 4.3 N transformation during Fe(VI) treatment of landfill leachate at pH 4-5.

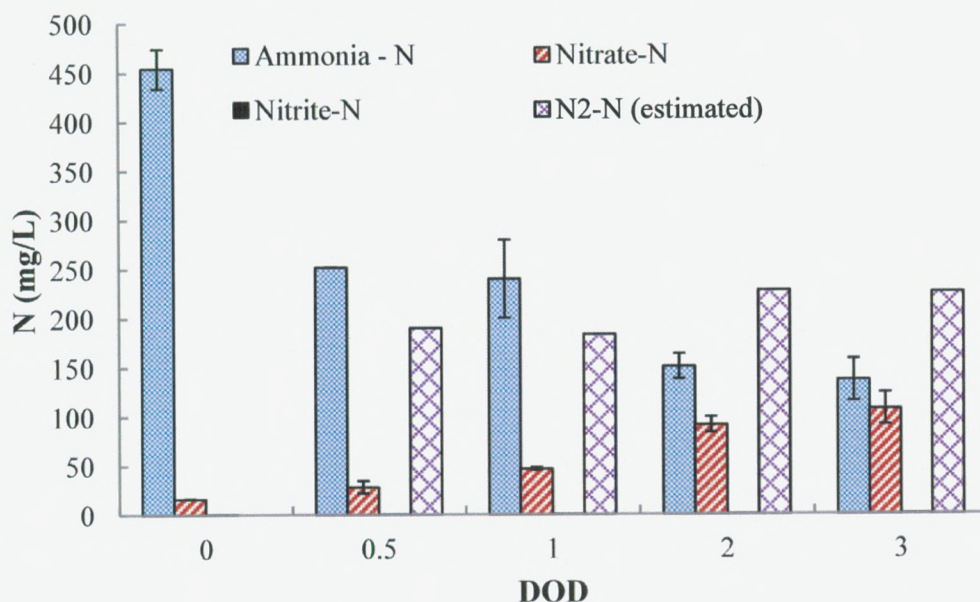


Figure 4.4 N transformation during Fe(VI) treatment of landfill leachate at pH 9.

4.3 Effect of Chloride (Cl⁻)

Chloride (Cl⁻) is a very common species found in landfill leachate. Early studies show that Cl⁻ may broadly range within 0 – 77,000 mg/L (Kjeldsen et al. 2002; Reinhart et al. 1998). Cl⁻ can rapidly react with certain oxidizing agents, and thus compete with target compounds for oxidants to inhibit oxidation efficiencies (Deng et al. 2012). Therefore, understanding of the Cl⁻ role in Fe(VI) treatment of landfill leachate is of importance. Residual COD and NH₃-N during Fe(V) treatment of landfill leachate at different Cl⁻ concentrations are shown in Figures 4.5 and 4.6, respectively. Of note, in order to achieve a leachate with 0 mg/L Cl⁻, AgNO₃ was added to precipitate all the Cl⁻. This precipitation process also reduced the initial COD from 1,372 to 676 mg/L, and increased the initial NH₃-N from 454 to 485 mg/L, respectively.

As seen in Figure 4.5, the effect of Cl⁻ (2,100 -14,880 mg/L) noticeably enhanced the COD removal at pH 4-5. When Cl⁻ was increased from 2,100 to 14,880 mg/L, the COD of Fe(VI)-treated leachate (DOD=2) dropped from 624 to 380 mg/L. However, Cl⁻ exhibited a small inhibiting role in the COD removal by Fe(VI) at pH 9. The residual COD after Fe(VI) treatment (DOD =2) increased from 736 to 810 mg/L with the increasing Cl⁻ from 2,100 to 14,880 mg/L. As shown in Figure 4.6, the effect of Cl⁻ on residual NH₃-N was a little complex. At pH 4-5, as Cl⁻ increased from 2,100 to 5,000 mg/L, the residual NH₃-N after Fe(VI) treatment increased from 253 to 333 mg/L.

However, the residual $\text{NH}_3\text{-N}$ slightly dropped to 271 mg/L when Cl^- further went up to 14,880 mg/L. In contrast, at pH 9, $\text{NH}_3\text{-N}$ decreased from 151 to 109 mg/L with the increasing Cl^- from 2,100 to 14,880 mg/L.

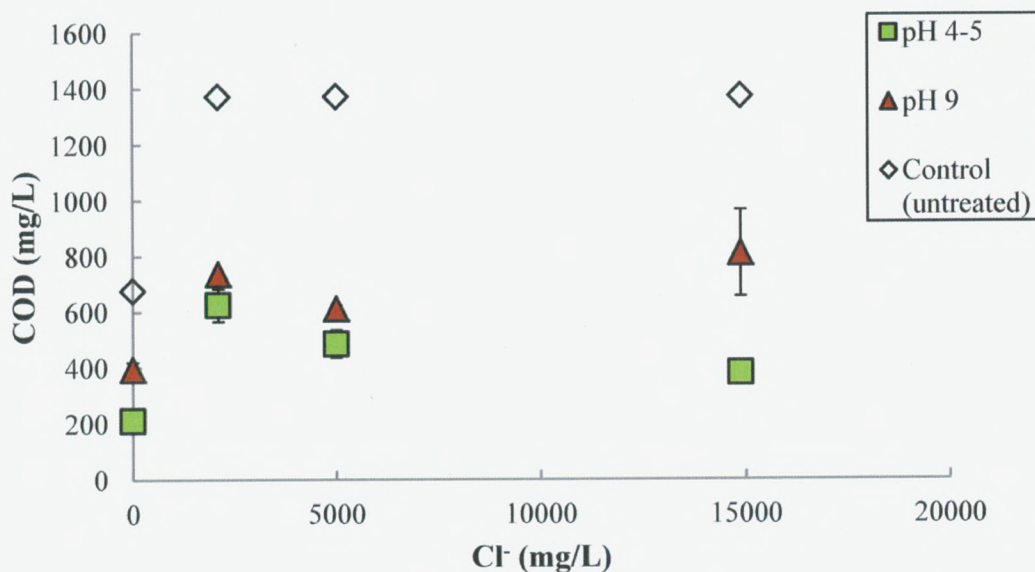


Figure 4.5 Residual COD after Fe(VI) treatment in presence of different Cl^- levels (DOD = 2).

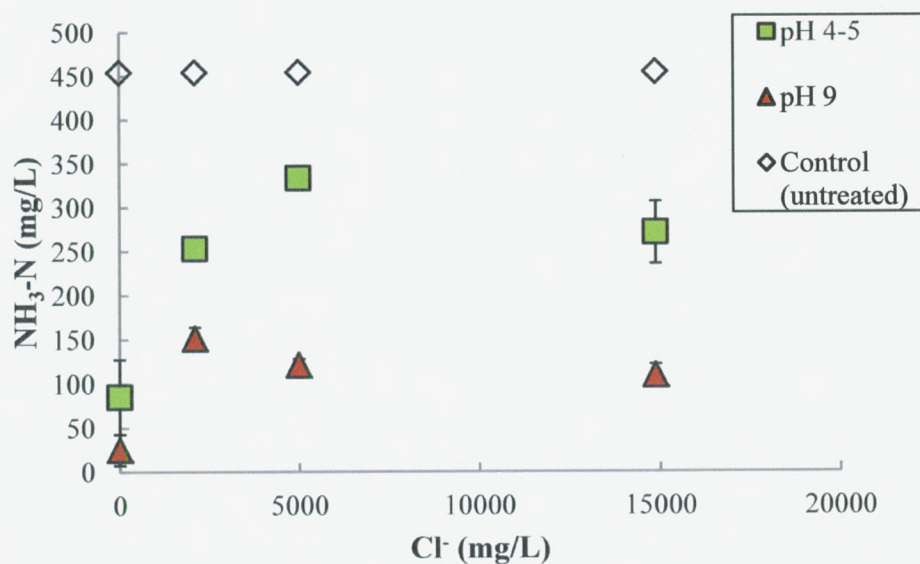
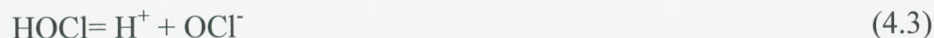
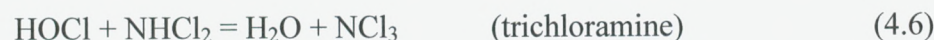
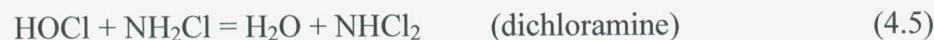
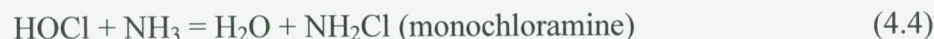


Figure 4.6 Residual $\text{NH}_3\text{-N}$ after Fe(VI) treatment in presence of different Cl^- levels (DOD = 2).

Effects of chloride on N transformation at pH 4-5 and pH 9 during Fe(VI) treatment of leachate are shown in Figure 4.7 and 4.8, respectively. At pH 4-5, when Cl^- increased from 2,100 to 5,000 mg/L, the produced NO_3^- -N increased from 30 to 47 mg/L. However, when Cl^- further went up to 14,880 mg/L, NO_3^- -N produced was almost zero. The finding may be ascribed to the different roles that Cl^- played. In the certain oxidants (e.g. Fe(VI)), Cl^- might transform to active chlorine species, such as hypochlorite (OCl^-) and hypochlorous acid (HOCl). At pH 4-5 (acidic condition), the dominant form of the chlorine species is HOCl that is more oxidative than OCl^- , as follows.



The HOCl might enhance NH_3 -N oxidation into NO_3^- -N. Therefore, more NO_3^- -N was produced at 5,000 mg/L Cl^- than 2,100 mg/L Cl^- . However, when much active HOCl was produced, its reactions with NH_3 might prevail, which led to the production of N_2 , as follows.



In contrast, different N transformation pattern was observed at pH 9. Much more NO_3^- -N was produced (80-91 mg/L) at pH 9, and the NO_3^- -N production appeared to be independent of the Cl^- concentration. At pH 9 (basic condition), the major Fe(VI) and chlorine species are FeO_4^{2-} and OCl^- , respectively. The mechanisms behind the finding are not clear.

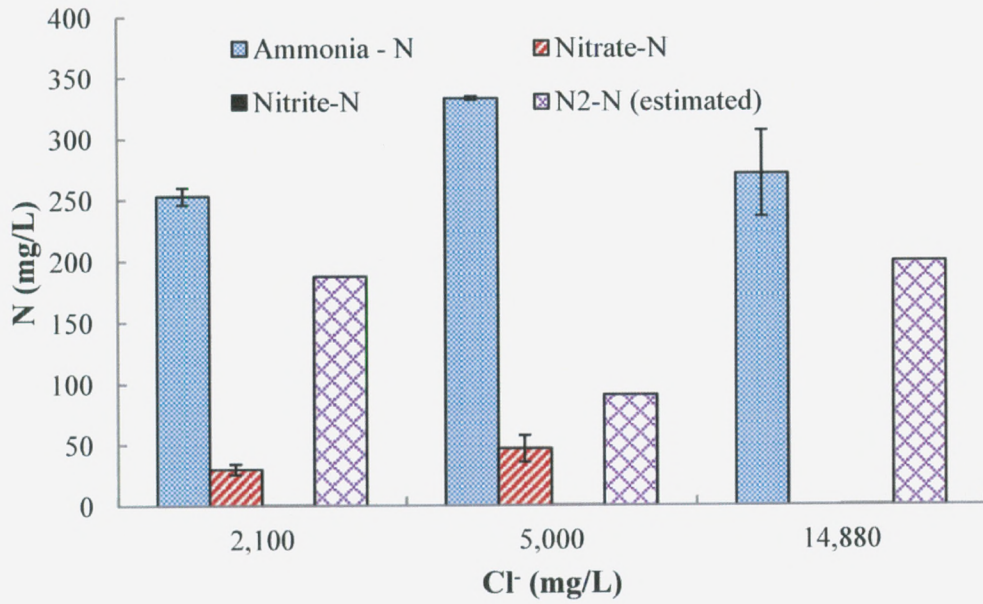


Figure 4.7 N transformation during Fe(VI) treatment of landfill leachate at pH 4-5 and different Cl⁻ levels (DOD = 2).

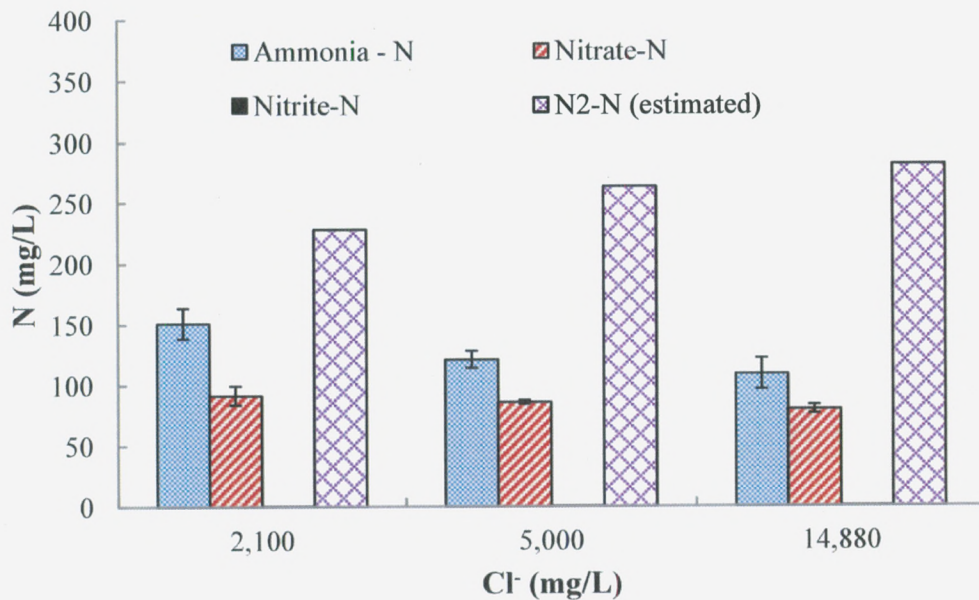


Figure 4.8 N transformation during Fe(VI) treatment of landfill leachate at pH 9 and different Cl⁻ levels (DOD = 2).

4.4 Effect of Initial NH₃-N

Effects of initial NH₃-N levels on residual COD and NH₃-N of the Fe(VI)-treated leachate are shown in Figure 4.9 and 4.10, respectively. At pH 4-5, when the Fe(VI) dose is fixed, the residual COD was increased from 422, 624, to 818 mg/L with the increasing NH₃-N from 167, 454 to 1,540 mg/L. The negative effect of initial NH₃-N is ascribed to the competition of NH₃-N with COD for Fe(VI). With the increasing initial NH₃-N, more Fe(VI) was consumed for oxidation of ammonia, so that COD removal was inhibited. As demonstrated in Figure 4.10, at pH 4-5, the removed NH₃-N were 36, 201, and 330 mg/L at the initial NH₃-N = 167, 454, and 1,540 mg/L, respectively. In contrast, different patterns in residual COD and NH₃-N were observed at pH 9. At a fixed Fe(VI) dose, the residual COD slightly varied between 736 and 857 mg/L within the initial NH₃-N range of 167 – 1,540 mg/L. And the NH₃-N was removed by 102, 303, and 1,180 mg/L with the initial NH₃-N = 167, 454, and 1,540 mg/L, respectively. The dramatic improvement of NH₃-N might be because much NH₃-N was removed by volatilization of NH₃ molecules, which had a much high fraction at a basic condition.

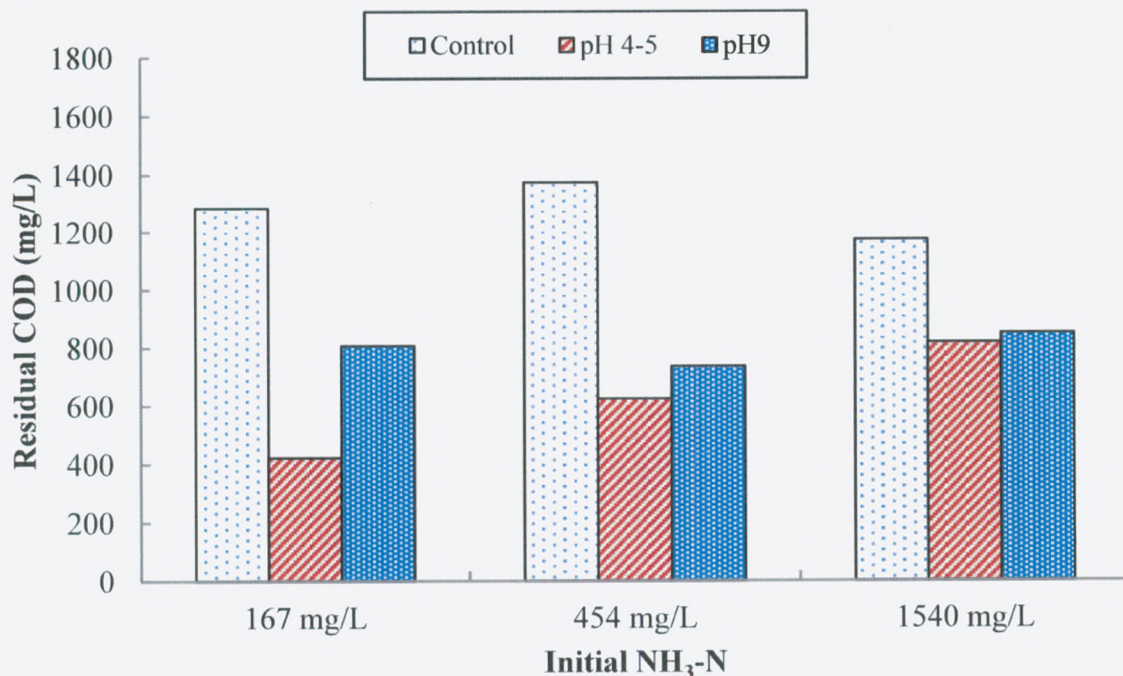


Figure 4.9 Residual COD after Fe(VI) treatment in presence of different initial NH₃-N levels. DOD = 2 at initial NH₃-N = 454 mg/L).

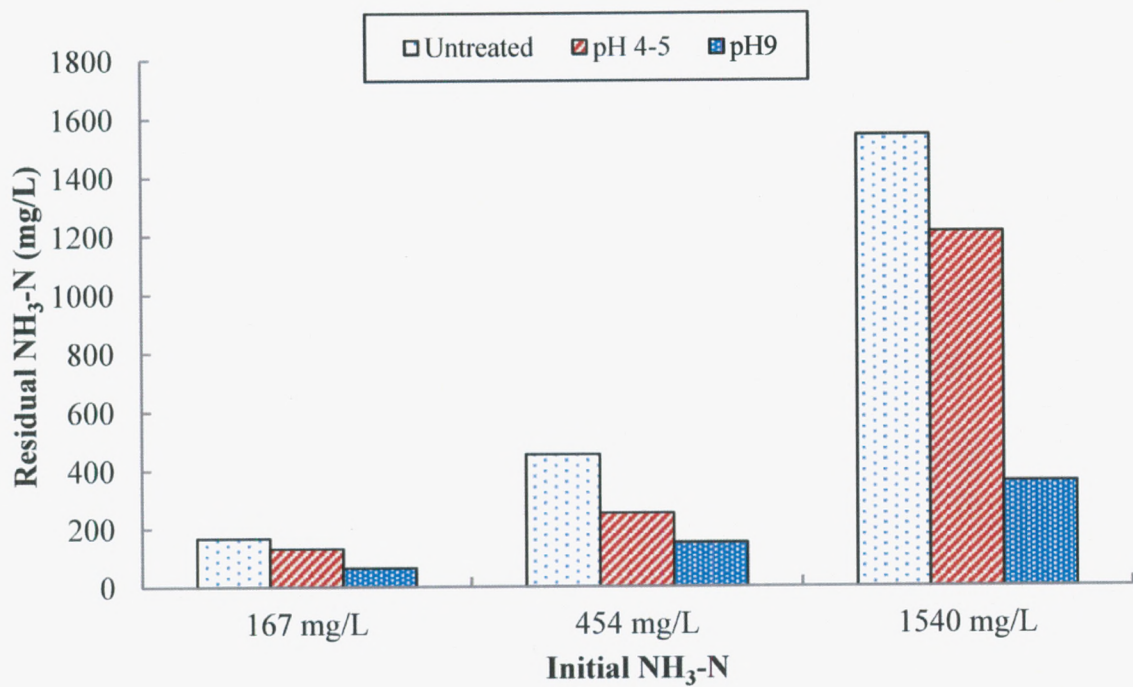


Figure 4.10 Residual NH₃-N after Fe(VI) treatment in presence of different initial NH₃-N levels. DOD = 2 at initial NH₃-N = 454 mg/L).

CHAPTER 5. CONCLUSIONS AND SUGGESTION

This study demonstrated the treatment patterns of Fe(VI) for landfill leachate. The major conclusions include:

- 1) Fe(VI) could provide moderate removal in COD and NH₃-N at acidic and basic conditions. In this study, at a maximum, 60% COD and 70% NH₃-N were reduced.
- 2) The removal of COD and NH₃-N were increased with the increasing DOD. In this study, DOD = 1- 2 appeared to be the optimal range of Fe(VI) dose. The increase in treatment efficiencies was marginal when DOD was beyond 2.
- 3) COD removal was due to Fe(VI)-induced oxidation and/or iron sludge-induced coagulation/adsorption, while NH₃-N were removed as a result of Fe(VI) oxidation, in addition to volatilization at basic condition.
- 4) At any condition, little NO₂⁻ was produced as a result of NH₃-N oxidation.
- 5) The oxidized NH₃-N was transformed to N₂ and NO₃⁻. Higher Fe(VI) dose and higher pH favored the production of NO₃⁻-N.
- 6) The role of Cl⁻ (2,100 -14,880 mg/L), a very common leachate solute, in Fe(VI) treatment of leachate was complex. Cl⁻ somewhat increased COD removal at pH 4-5, but slightly slowed down the COD reduction at pH 9. At pH 4-5, a low Cl⁻ level (2,100 – 5,000 mg/L) could inhibit the NH₃-N removal; however, more Cl⁻ improved NH₃-N reduction at a high Cl⁻ range (5,000 – 14,880 mg/L). In contrast, at pH 9, high Cl⁻ concentration favored the NH₃-N removal.
- 7) NH₃-N could compete with COD for Fe(VI). Therefore, higher initial NH₃-N might inhibit COD reduction.

Overall, our findings demonstrates that Fe(VI) is a potential chemical agent for treatment of landfill leachate. Refractory leachate organic matters and ammonia nitrogen can be simultaneously removed. To further develop the Fe(VI)-based oxidation technology for leachate treatment, suggestion is proposed for future study.

- 1) Better experimental device and design are needed to accurately measure the NH₃-N loss due to volatilization;
- 2) Because commercial ferrate salt contains base, the solution pH was not easy to control during reactions. High purity of Fe(VI) salt should be prepared in the future tests, so that pH is more readily controlled.

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