Environmental Health Engineering and Management Journal 2016, 3(1), 35–40

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Environmental Health
Engineering and
Management Journal

Original Article

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Treatment of mature landfill leachate by chemical precipitation and Fenton advanced oxidation process

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Abstract

Background: Mature landfill leachate is a complicated mixture which is resistant to biological treatment processes. The treatment of mature landfill leachate by struvite precipitation and Fenton oxidation was the main objective of the current research.

Methods: Struvite with the phosphate/ammonia/magnesium molar ratio of 1/1/1.05 was considered during all experiments. Five initial pHs of 3, 4, 5, 6, and 7, four different H_2O_2/Fe mass ratios of 50, 100, 200, and 400, and reaction times of 20, 40, 80, 120, and 160 minutes were examined for the Fenton oxidation process.

Results: A leachate sample with average chemical oxygen demand (COD), BOD_5, and NH_4 concentrations of 7350, 2220, and 2280 mg L^{-1}, respectively, and a BOD_5/COD ratio of 0.3 was introduced to the chemical precipitation unit. An NH_4 removal efficiency of 87% was obtained at pH 8.5 for struvite precipitation. Under optimum conditions of Fenton oxidation, including pH 3, an H_2O_2/Fe^{2+} mass ratio of 200, and a reaction time of 160 min, more than 95% COD and BOD_5 removal was observed.

Conclusion: Struvite precipitation and Fenton oxidation are reliable and efficient alternatives for mature landfill treatment.

Keywords: Landfill, Leachate, Fenton oxidation, Chemical precipitation, Advanced oxidation

Citation: Jaafarzadeh Haghighi Fard NA, Jorfi S, Ahmadi M, Mirali S, Kujlu R. Treatment of mature landfill leachate by chemical precipitation and Fenton advanced oxidation process. Environmental Health Engineering and Management Journal 2016; 3(1): 35–40.

Article History:
Received: 13 January 2016
Accepted: 28 February 2016
ePublished: 15 March 2016

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Introduction

Final disposal of solid wastes is the most common operation of solid waste management in countries worldwide (1). The physical, biological, and chemical reactions in landfill wastes as well as percolated water and the initial moisture of waste through the landfill contents result in the production of a highly concentrated dark liquid named landfill leachate (2). A wide variation of leachate constituents are observed depending on the type of solid waste and its characteristics, moisture content, site hydrology, waste porosity and compaction; engineering operations like daily and final cover designs and bottom linings; environmental effects on landfill site; landfill age and operation performances (3,4). Leachate can contaminate soil and water resources and affect human health both directly and indirectly by spreading hazardous constituents. Traditional leachate treatment methods including surface ponds or chemical treatment processes are inefficient and expensive; they produce a considerable amount of excess sludge and can cause environmental pollution (5). The age of the landfill and the composition of the leachate strongly direct the treatment alternatives for leachate management. For instance, young leachates are characterized by high biochemical oxygen demand/chemical oxygen demand (BOD_5/COD) ratios which make the biological treatment processes feasible and reliable options (6,7). Studies have revealed that old leachate has a lower BOD_5/COD ratio; thus, other treatment schemes such as advanced oxidation processes should be considered to overcome the low biodegradability and refractory toxics (1,8). Advanced oxidation processes (AOP) involve the generation of free reactive radicals, mainly the hydroxyl radical through the chemical, electrochemical, or photochemical reactions between conventional oxidants such as ozone, hydrogen peroxide, and UV irradiation (5). Hydroxyl radicals react with the target pollutant, and that leads to the mineralization or partial oxidation of contaminants according to the dosage of free radicals and other operational parameters (9). Fenton oxidation has been widely considered for the treatment of refractory substances, like low biodegradable mature landfill leachate contents. In the Fenton reaction, ferrous ions react with H_2O_2 under optimum pH to generate highly oxidative hydroxyl radicals in a liquid medium (10,11). According to general Equation 1, the conventional Fenton process is defined as the reaction of Fe^{2+} and H_2O_2 for the production of hydroxyl radicals (one of the strongest oxidants, E = 2.73 V) (12), which is widely studied for the treatment of landfill leachate (9).

$$Fe^{2+} + H_2O_2 \rightarrow Fe_3OH^{-} + OH^{*} \qquad (1)$$

According to Equation 1 the reaction will be completed under acidic conditions, because the presence of H^+ ions is necessary for the decomposition of H_2O_2 (3). The major benefits of Fenton oxidation include low costs, ease of access, relative safety, effectiveness, and simple design and operation (13,14). Another specification of leachate is a high concentration of NH_4-N as a usual characteristic of landfill leachate which adversely affects the treatment process. It is a major toxicant to biological reactions in activated sludge processes; it is also a significant radical scavenger in AOP. Therefore, pretreatment prior to any treatment system can enhance treatment efficiency and lower operational technical problems and costs, especially in Fenton reactions (15). Studies have shown that struvite precipitation is one of the most effective alternatives for removing high ammonium concentrations from landfill leachates, which provides a rapid reaction rate and considerable removal efficiency (16). Struvite is a white crystalline compound chemically consisting of magnesium, ammonium, and phosphorus in equal molar concentrations ($MgNH_4PO_4.6H_2O$) (17). Struvite formation reaction is presented in Equation 2:

$$Mg^{2+} + NH_4^{+} + PO_4^{-} \rightarrow MgNH_4PO_4.6H_2O \qquad (2)$$

The removal of ammonium by means of struvite precipitation has been successfully applied in different industrial wastewaters and landfill leachate (18-20). Based on a literature review, there is no report on the application of integrated struvite precipitation and Fenton oxidation for leachate treatment. In the current study, pre-treatment by struvite precipitation and Fenton oxidation before biological treatment for mature landfill leachate was evaluated.

Materials and Methods

Materials

The $MgCl_2.6H_2O$, $NaH_2PO_4.2H_2O$, NaOH, H_2SO_4, $FeSO_4$, and H_2O_2 used in this study were of analytical grade and purchased from Merck, Germany.

Characterization of landfill leachate

A municipal solid waste landfill was the source of leachate sampling. Twelve samples were collected monthly for one year. The characteristics of COD, BOD_5, NH_3-N, and pH were analyzed and the averages of the three repetitions are presented in Table 1. Samples were transferred to the laboratory in a cold box at 4°C.

Chemical precipitation experiment

Chemical precipitation with struvite was considered for the removal of NH_4. A Plexiglas container, with a total volume of 20 L and equipped with a variable speed mixer was considered as the reactor for struvite precipitation. The experiments were carried out in batch mode. $MgCl_2.6H_2O$ and $NaH_2PO_4.2H_2O$ were used as magnesium and phosphate sources. The ratio of phosphate to ammonia to magnesium was adjusted stoichiometrically to 1/1/1.05 mol during all experiments (16) and pH values were adjusted to 7, 7.3, 7.6, 7.9, 8.2, and 8.5 using either NaOH or H_2SO_4. After the time required for completion of the reaction had passed, the contents of the reactor were allowed to settle for 30 minutes and then passed through a Millipore membrane filter with a pore size of 0.45 µm for use in performing analytical tests. The supernatant was withdrawn and directed to the Fenton reaction reactor. For the following experiments performed with the Fenton process, the pH had to be adjusted.

Fenton reaction

Conventional Fenton oxidation with Fe^{2+} was considered for the destruction of refractory organics. Operational parameters including pH values of 3, 4, 5, 6, and 7, H_2O_2/Fe mass ratios of 50, 100, 200, and 400, and reaction times of 20, 40, 80, 120, and 160 minutes were examined consecutively. Five initial pHs in a constant reaction time of 40 minutes and a H_2O_2/Fe mass ratio of 50 were investigated. In the second step, different H_2O_2/Fe mass ratios were examined to evaluate the effect of H_2O_2/Fe increase on the enhancement of process efficiency in a constant reaction time of 40 minutes and optimum pH. The H_2O_2 concentration of 40 mM (35%, Merck, analytical grade) was constant and the iron was varied. Reaction times of 20, 40, 80, 120, and 160 minutes were examined in the predetermined optimum pH and H_2O_2/Fe. All mineralization reactions for each defined condition were performed in 500 ml flasks in batch mode. The flasks were sealed with aluminum crimp caps and shaken on a shaker at 150 rpm. All experiments were conducted at room temperature (23 ± 4°C). A summary of experimental conditions and related identification code are presented in Table 2. All ana-

Table 1. Results of chemical precipitation with struvite for ammonia removal

[NH3][Mg][PO4] (ratio of moles)	pH	COD (mg L⁻¹)	BOD5 (mg L⁻¹)	NH3–N (mg L⁻¹)
Raw leachate	6.9	7350	2220	2280
1/1/1.05	7.2	6805	1905	471
1/1/1.05	7.5	6620	1830	430
1/1/1.05	7.8	6545	1747	358
1/1/1.05	8.1	6380	1668	330
1/1/1.05	8.5	6215	1602	286

lytical data are based on the averages of the experiments run in triplicate. The desired pH values were adjusted using either sulfuric acid (Merck, analytical grade) or sodium hydroxide (Merck, analytical grade). Before analysis, 0.5 ml of a sodium thiosulfate solution was added to each sample to quench the residual H_2O_2 concentration in the flask.

Analytical methods

All laboratory analyses of COD, BOD_5, NH_3-N, and alkalinity were conducted according to the *Standard Methods for Examination of Water and Wastewater* (21). The cadmium concentration was determined using atomic absorption spectroscopy (Model AAS vario6) (21). Samples were collected from the supernatant and then centrifuged at 5000 rpm for 20 minutes. Those used for NH_3-N and soluble COD investigations were filtered on glass-fibre filters (GF50). A pH meter (Hack, Germany) was used to measure pH.

Results

Mature leachate characteristics

The characteristics of mature leachate after nearly one year sampling and analysis are presented in Table 3. Results indicated that annual average COD and BOD_5 concentrations were 7350 and 2220 mg L^{-1}, respectively.

Struvite precipitation

The results of chemical precipitation with struvite on mature landfill leachate with a stoichiometric ammonium/magnesium/phosphate ratio of 1/1/1.05 in varying pHs ranging from 6.9 to 8.5 are presented in Table 1. The residual NH_3–N concentrations after chemical reaction for pH values of 7.2, 7.5, 7.8, 8.1, and 8.5 were 471, 430, 358, 330, and 286 mg L^{-1}, respectively. The highest removal efficiency of 87% was achieved with pH 8.5, and this was thus selected as the optimum pH level. At the end of the chemical reaction in pH 8.5, COD and BOD concentrations had been reduced to 6380 and 1668 mg L^{-1}, respectively, providing 13.2% and 24% removal, respectively (Figure 1).

Table 2. Different experimental conditions

Parameter	Variation range
pH	3–4–5–6–7
H_2O_2/Fe	50–100–200–400
Reaction time	20–40–80–100

Table 3. Average characteristics of landfill leachate

Parameter	Mean value
COD (mg L^{-1})	7350 ±410
BOD (mg L^{-1})	2220 ±260
BOD5/COD	0.3
NH_3–N (mg L^{-1})	2280 ±340
Alkalinity (mg $L^{-1}{}_{ac}$ $CaCO_3$)	4350 ±325
pH	6.9

Fenton advanced oxidation process

Optimum pH

The results of pH optimisation are presented in Figures 2A and 2B. The effluent COD and BOD_5 concentrations of the struvite precipitation unit entering the Fenton reactor were 6215 and 1602 mg L^{-1}, respectively. Since the best results of struvite precipitation occurred in pH 8.5, the desired pH was first adjusted to a defined value with sulphuric acid. The least remaining amounts of BOD and COD (1433 and 4496 mg L^{-1}, respectively) were observed at pH 3; therefore, pH 3 was selected as the optimum value.

Optimum H_2O_2/Fe^{2+}

Four different mass ratios of H_2O_2/Fe were examined at a constant COD concentration, optimum pH, and a reac-

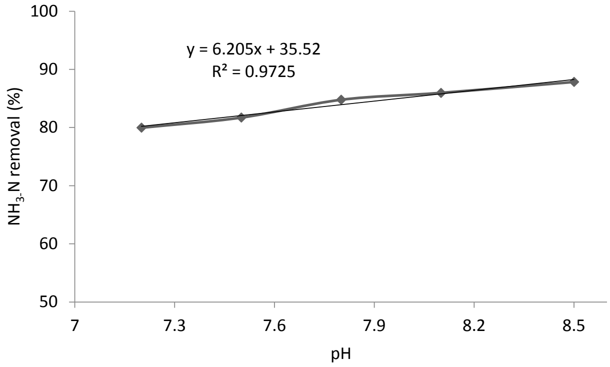

Figure 1. Ammonium removal in different pHs and constant chemical dosage.

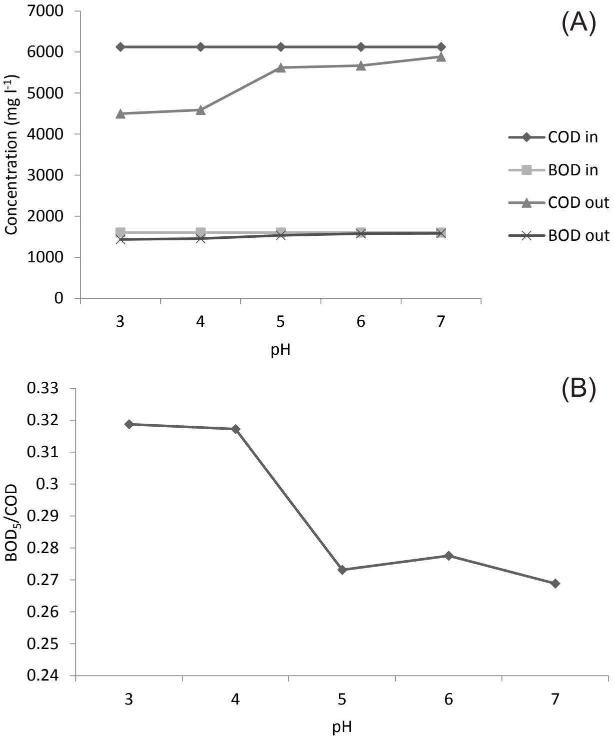

Figure 2. (A) Determination of optimum pH in constant reaction time of 40 minutes, H_2O_2/Fe ratio of 50, and feed characteristics; (B) BOD_5/COD ratio variations

tion time of 40 minutes (H_2O_2 concentration of 400 mM). The results of this step are shown in Figure 3. As seen in Figure 3A, removal of COD and BOD_5 did not improve at H_2O_2/Fe^{2+} ratios higher than 200. The BOD_5/COD ratio changed from 0.31 to 0.3 H_2O_2/Fe^{2+} m at a mass ratio of 200. Based on data obtained in this step, the H_2O_2/Fe^{2+} mass ratio of 200 was selected as the optimum value.

Optimum reaction time

The results of time variations are shown in Figures 4A and 4B. As was anticipated and can be seen in Figure 4A, the reaction time increase favoured removal efficiency. Residual BOD_5 and COD values after 160 minutes were 80 and 293 mg L^{-1}, respectively, and the BOD_5/COD ratio reached 0.27 at this time. It seems that all biodegradable constituents had been removed at this time and no further removal would occur. The COD removal values in reaction times of 20, 40, 80, 120, and 160 minutes were 3.8%, 56.6%, 86.5%, 88.7%, and 95.2%, respectively.

Discussion

Leachate resistance toward biological reactions

The BOD_5/COD ratio of less than 0.5 (exactly = 0.3) makes the direct application of biological treatment schemes difficult. On the other hand, the presence of refractory organics and toxics such as heavy metals necessitate the application of pre-treatments such as chemical precipitation and AOP. The low BOD_5/COD ratio has been reported by other researchers. Cortez et al (10) reported low BOD_5/COD ratios ranging from 0.01 to 0.15. Also, Li et al (22) reported the BOD_5/COD ratio of 0.05. The average ammonium concentration was relatively high and equal to 2280 mg L^{-1}. Scientific reports have identified NH_3–N as a major toxicant to biological processes and a radical scavenger for AOP. Therefore, the sufficient removal of NH_3–N before the following treatment is required (15,16).

Effect of pH on struvite precipitation

As seen in Figure 1, the pH increase enhanced the removal of ammonia linearly with an R^2 coefficient of 97.2. These results are in agreement with those of Jaafarzadeh et al (16) who reported an 89% NH_3–N removal with a pH increase from 7.62 to 7.84 (concentration decreased from 2162 to 229 mg L^{-1}). Similar findings have been reported in literature (18,23).

Effect of pH on Fenton oxidation

Many reports have specified pH as the chief factor affecting the performance of the Fenton process for contaminant degradation because of the necessity of keeping ferrous ions in soluble form to continuously react with H_2O_2 (24-26). A pH below optimum value can inhibit the reaction, because at extremely low pH values, the formed $[Fe(H_2O)]^{2+}$ reacts relatively slowly with H_2O_2 and less hydroxyl radical production occurs. Moreover, the scavenging effect of H^+ on hydroxyl radical can interfere with the reaction between Fe^{3+} and H_2O_2. The BOD_5/COD ratio which accounts for the biodegradability potential of wastewater was 0.31 at pH 3 and decreased to 0.26 at a pH value of 7. This verifies the higher degradation rate of lower pHs, which leads to more COD conversion and the production of many intermediates that might be bio-

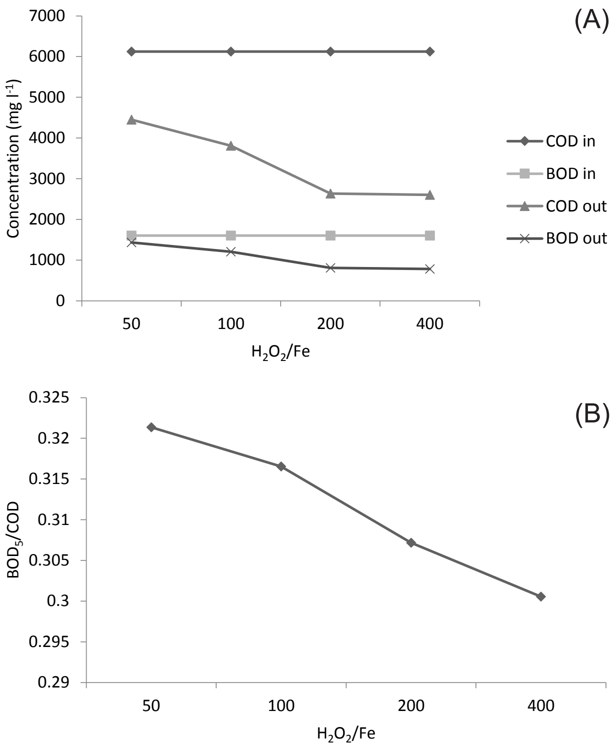

Figure 3. (A) Determination of optimum H_2O_2/Fe in optimum pH 3 and constant reaction time of 40 minutes and feed characteristics; (B) BOD_5/COD ratio variations.

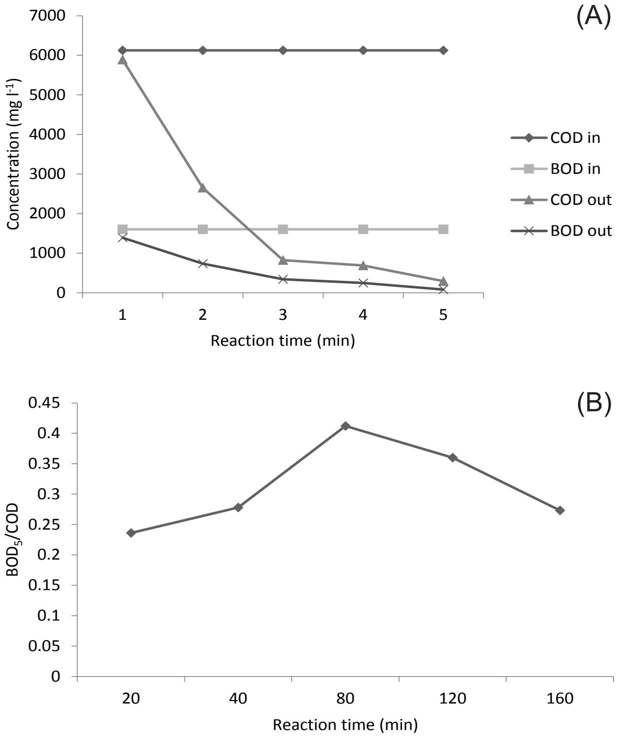

Figure 4. (A) Determination of optimum reaction time in optimum pH 3, H_2O_2/Fe ratio of 200, and constant feed characteristics; (B) BOD_5/COD ratio variations

degradable and need a longer reaction time to complete mineralisation. For the full treatment of leachate by Fenton oxidation, pH 3 can be introduced as the best value. If the Fenton is considered as a pre-treatment before biological processes, however, higher values of 6 or 7 can be considered because of the bacterial functions in neutral pH ranges. This finding was in agreement with those of similar studies (27,28).

Effect of H_2O_2 and Fe^{2+} dosages

Excess amounts of Fe^{2+} and H_2O_2 reagents may adversely affect the process, leading to high TDS, high electrical conductivity (EC) of the effluent and an excessive chemical sludge production (29). Moreover, if a biological treatment has been considered following the AOP, residual H_2O_2 can destroy bacterial metabolism, raise operational costs, and produce gas bubbles which prevent sedimentation of the sludge flocs (24). Similar studies have concluded that the degradation rate of organics increases linearly with H_2O_2 concentration until the threshold value is reached, at which point the addition of H_2O_2 does not improve the degradation rate flocs (24). The least BOD_5 and COD concentrations of 762 and 2637 mg L^{-1} were observed with an H_2O_2/Fe^{2+} mass ratio of 200, which is in agreement with the overall trend obtained by Lopez et al (13). The initial COD concentration was reduced from 10 540 to 4180 mg L^{-1} at an H_2O_2/Fe^{2+} mass ratio of 12, and higher ratios did not improve the removal (13). Therefore, it was concluded that the addition of Fe to a higher than threshold value does not significantly enhance process efficiency.

No significant BOD_5/COD ratio variations were observed in this step; the ratio was decreased from 0.32 at an H_2O_2/Fe^{2+} mass ratio of 50 to 0.3 at an H_2O_2/Fe^{2+} mass ratio of 400 (Figure 3B). This is probably attributable to the limited reaction time and equal transformation rate for BOD_5 and COD components, the presence of refractory compounds, and the reaction intermediates. The highest BOD_5/COD ratio of 0.41 was observed in 80, which was the highest value in all experiments. It can be explained by the partial oxidation of refractory organics and the conversion of some slowly degradable organics to readily biodegradable compounds. AOPs are characterised by rapid partial degradation and time intensive mineralisation (12). Considering a supplement biologic treatment, a contact time of 80 minutes can be determined as the optimum value. If Fenton oxidation is the unique treatment scheme, a higher reaction time would yield a higher removal amount; therefore, the contact time of 160 minutes with more than 95% COD removal is the optimum value.

Conclusion

According to the experimental results, it can be concluded that struvite precipitation followed by Fenton oxidation is a reliable, feasible, and efficient alternate pre-treatment and also post-treatment of mature landfill leachate.

The experimental data obtained in this study are summarized below.

- Struvite precipitation led to 87% NH_4 removal at pH 8.5 (concentration of 2280 was reduced to 286 mg L^{-1}).
- The optimum operational conditions were pH = 3, H_2O_2/Fe^{2+} mass ratio = 200, and reaction time = 80 minutes for Fenton oxidation as a pre-treatment for biological processes and a reaction time = 160 minutes when used as a post-treatment.
- The residual BOD_5 and COD concentrations at optimum conditions were 80 and 293 mg L^{-1}, respectively, providing 95% removal.
- The BOD_5/COD ratio of 0.41 could be achieved with a pH of 3, an H^2O^2/Fe^{2+} mass ratio of 200, and a reaction time of 80 minutes.

Acknowledgments

The authors would like to thank Ahvaz Jundishapur University of Medical Sciences for their support of this study.

Ethical issues

There were no ethical issues in any phase of the experiments or in the writing of this article.

Authors' contributions

This statement certifies that all authors were involved in all stages of preparing this article. It is warranted that this article has not been previously published and is not under consideration for publication elsewhere. On behalf of all co-authors, the corresponding author shall bear full responsibility for the submission.

Competing interests

The authors declare that they have no conflicts of interest.

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