Optimization of leachate treatment using persulfate/H$_2$O$_2$ based advanced oxidation process: case study: Deir El-Balah Landfill Site, Gaza Strip, Palestine

Ahmed H. Hilles, Salem S. Abu Amr, Rim A. Hussein, Anwar I. Arafa and Olfat D. El-Sebaie

ABSTRACT

The objective of this study was to investigate the performance of employing H$_2$O$_2$ reagent in persulfate activation to treat stabilized landfill leachate. A central composite design (CCD) with response surface methodology (RSM) was applied to evaluate the relationships between operating variables, such as persulfate and H$_2$O$_2$ dosages, pH, and reaction time, to identify the optimum operating conditions. Quadratic models for the following two responses proved to be significant with very low probabilities (\(<0.0001\)): chemical oxygen demand (COD) and NH$_3$-N removal. The obtained optimum conditions included a reaction time of 116 min, 4.97 g S$_2$O$_8^{2-}$, 7.29 g H$_2$O$_2$ dosage and pH 11. The experimental results were corresponding well with predicted models (COD and NH$_3$-N removal rates of 81% and 83%, respectively). The results obtained in the stabilized leachate treatment were compared with those from other treatment processes, such as persulfate only and H$_2$O$_2$ only, to evaluate its effectiveness. The combined method (i.e., S$_2$O$_8^{2-}$/H$_2$O$_2$) achieved higher removal efficiencies for COD and NH$_3$-N compared with other studied applications.

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Key words | activation, advanced oxidation, hydrogen peroxide, landfill leachate, persulfate

INTRODUCTION

Sanitary landfill is recognized as the most common and desirable method of urban solid waste management and as the most economical and environmentally acceptable method of municipal and industrial solid waste disposal (Tengrui et al. 2007). However, sanitary landfill generates a large volume of heavily polluted leachate (Zazouil & Yousefi 2008). Leachate is mainly released from waste deposited in a landfill due to successive biological, chemical, and physical processes (Alslaibi et al. 2010). The quality and quantity of the water formed at landfills depend on several factors, including seasonal weather variations, land filling technique, phase sequencing, piling, and compaction method (Amonkrane et al. 1997; Trebouet et al. 2001). Landfill leachate is a high-strength wastewater that is very difficult to manage. The leachate generated from stabilized landfills (age >10 years) is typically characterized by large amounts of organic contaminants measured as chemical oxygen demand (COD), biochemical oxygen demand (BOD$_3$), ammonia, halogenated hydrocarbons, suspended solids, significant concentration of heavy metals, and many other hazardous chemicals identified as potential sources of ground and surface water contamination (Schrab et al. 1993; Christensen et al. 2001; Aziz et al. 2009; Foul et al. 2009). A number of leachate treatment techniques, including biological, physical, and chemical processes, have been applied (Baig & Liechti 2001; Goi et al. 2009). Given its oxidation efficiency, persulfate is one of the chemical processes that have recently received considerable attention in landfill leachate treatment because of its oxidation potential and ability to reduce leachate strength and the amount of non-biodegradable organics (Huling & Pivetz 2006; Deng & Eyzske 2011; Watts 2011; Liu et al. 2012). Several applications of persulfate on landfill leachate treatment have been conducted. Abu Amr et al. (2015) obtained 39% and 22% removal rates for COD and NH$_3$-N, respectively, during the oxidation of stabilized leachate using persulfate alone. Accordingly, the performance of persulfate alone in stabilized leachate treatment is low; its effectiveness can be improved using...
advanced oxidant materials and techniques. Using ozone (O<sub>3</sub>) in advanced oxidation process (AOP) during the persulfate oxidation, Abu Amr et al. (2013) achieved a COD removal rate ranging from 39% (persulfate alone) to 72%. Liu et al. (2012) used iron (Fe<sup>2+</sup>) and copper (Cu<sup>2+</sup>) ions for persulfate activation and reported that iron ions would be more efficient than Cu<sup>2+</sup> ions for persulfate activation. However, the use of persulfate and H<sub>2</sub>O<sub>2</sub> in AOPs still has low efficiency for removal of ammonia, which exists in high level in leachate and is extremely toxic to the environment and aquatic organisms (Bashir et al. 2014). The scope and benefit of this new method is the increase in the oxidation potential using H<sub>2</sub>O<sub>2</sub> to activate persulfate and initiate sulfate radicals during the oxidation of stabilized leachate. In the present study, the interaction and statistical relationships among four independent factors (H<sub>2</sub>O<sub>2</sub> dosage, S<sub>2</sub>O<sub>8</sub><sup>2-</sup> dosage, pH variation and reaction time) for the treatment of anaerobic stabilized leachate were assessed through response surface methodology (RSM). RSM is a mathematical and statistical technique that is useful for the optimization of chemical reactions and industrial processes, and is commonly used for experimental designs. The main objectives of the present study include the following:

1. To investigate the efficiency of persulfate/H<sub>2</sub>O<sub>2</sub> oxidation process for treating of anaerobic stabilized leachate.
2. To develop an equation of COD and NH<sub>3</sub>-N removal efficiency from anaerobic stabilized leachate with respect to operational conditions (i.e., H<sub>2</sub>O<sub>2</sub> dosage, S<sub>2</sub>O<sub>8</sub><sup>2-</sup> dosage, pH and reaction time using RSM and central composite design (CCD)).
3. To determine the optimum operational conditions of the studied application.

**MATERIALS AND METHODS**

**Leachate sampling and characteristics**

Leachate samples were collected from the leachate collection pond at Deir El-Balah Landfill Site (DBLS), at Deir El-Balah City, Middle Governorate in Gaza Strip, Palestine. The leachate samples were collected from an anaerobic pond located in the area of the landfill that continues to receive waste. The DBLS has an area of 7 ha, receiving approximately 450 tons of municipal solid waste daily, and is considered as an anaerobic landfill. In this study, the leachate samples were collected six times manually from February 2014 to June 2014. For each time collected samples were placed in 2 L plastic containers. The samples were immediately transported to the laboratory, characterized, and cooled to 4 °C to minimize the biological and chemical reactions. The average characteristics of the leachate used in the experiments are summarized in Table 1. Sample collection and preservation were performed in accordance with *Standard Methods for the Examination of Water and Wastewater* (APHA 2005).

**Experimental procedures**

For each run, a persulfate reagent as sodium persulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>8</sub> M = 238 g/mol) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub> 37%) were employed for advanced oxidation of leachate samples. Persulfate and H<sub>2</sub>O<sub>2</sub> dosages were determined as COD/S<sub>2</sub>O<sub>8</sub><sup>2-</sup>/C<sub>0</sub>/H<sub>2</sub>O<sub>2</sub> ratio (g/g/g) by using different ratios that were added to the leachate until the best efficiencies of COD and NH<sub>3</sub>-N removal were obtained. An orbital shaker (Luckham R100/TW Rotatest Shaker 340 × 245 mm) was used for sample shaking at 350 rpm. All

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Results</th>
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<tbody>
<tr>
<td>COD (mg/L)</td>
<td>19,180–20,448</td>
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<tr>
<td>BOD (mg/L)</td>
<td>1,821</td>
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<tr>
<td>BOD/COD ratio</td>
<td>0.09</td>
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<tr>
<td>EC (μS)</td>
<td>40,800</td>
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<tr>
<td>TDS (mg/L)</td>
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<tr>
<td>Nitrate (mg/L)</td>
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</tr>
<tr>
<td>Ammonia (mg/L)</td>
<td>2,478</td>
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<tr>
<td>Sulfate (mg/L)</td>
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<tr>
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<tr>
<td>Copper (Cu) (mg/L)</td>
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<tr>
<td>Lead (Pb) (mg/L)</td>
<td>0.143</td>
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<tr>
<td>Nickel (Ni) (mg/L)</td>
<td>4.63</td>
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<tr>
<td>Manganese (Mn) (mg/L)</td>
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</tr>
<tr>
<td>Cadmium (Cd) (mg/L)</td>
<td>0.259</td>
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</tbody>
</table>

EC: electrical conductivity, TDS: total dissolved solids.
experiments were carried out at room temperature (28°C) using 50 mL samples in polyethylene bottles with a 250 mL capacity. pH was adjusted to the desired values using 5 M sulfuric acid solution and 5 M sodium hydroxide solution.

**Analytical methods**

COD and NH$_3$-N were tested immediately before and after the experiments, and the difference between the measurements of test values are presented as the average of the three measurements, respectively. The removal efficiencies of COD and NH$_3$-N were obtained using the following equation:

\[
\text{Removal (\%)} = \left( \frac{C_i - C_f}{C_i} \right) \times 100
\]

where $C_i$ and $C_f$ refer to the initial and final COD and NH$_3$-N concentrations, respectively.

**Experimental design and analysis**

Design expert software (version 6.0.7) was used for the statistical design of the experiments and data analysis. In the present study, a CCD and RSM were applied to optimize the experimental parameters and assess the relationships between four significant independent variables, as presented in Table 2: (1) persulfate dosage, (2) H$_2$O$_2$ dosage, (3) variation in pH, and (4) reaction time.

The COD and NH$_3$-N removal were the dependent variables (responses) during oxidation. Performance was evaluated by analyzing the COD and NH$_3$-N removal efficiencies. Each independent variable was varied over three levels between −1 and +1 at the determined ranges based on a set of preliminary experiments. The total number of experiments conducted for the four factors was 30 ($=2^k + 2k + 6$, where $k$ is the number of factors = 4); to assess the pure error, 24 experiments were enhanced with six replications. Because there were only three levels for each factor, the appropriate model followed a quadratic pattern, as shown in Equation (2):

\[
Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{j} X_j^2 + \sum_{i<j}^{k} \beta_{ij} X_i X_j + e_i
\]

where $Y$ is the response, $X_1$ and $X_2$ are the variables, $\beta$ is the regression coefficient, $k$ is the number of factors studied and optimized in the experiment, and $e$ is the random error.

Analysis of variance (ANOVA) was used for the graphical analysis of the data to determine the interactions between the process variables and responses obtained. The quality of fit in the polynomial model was expressed by the coefficient of determination ($R^2$), and its statistical significance was verified by an F-test in the same program. The model terms were evaluated based on the $P$-value (probability) corresponding to a 95% confidence level.

**RESULTS AND DISCUSSION**

A total of 30 runs were performed using the CCD experimental design; interactions between the four independent variables were considered in each run to investigate the validity of treating anaerobic stabilized leachate using H$_2$O$_2$ and persulfate during advanced oxidation. H$_2$O$_2$ reagent was used to activate persulfate during the oxidation of the leachate. As shown in Table 3, the removal efficiencies ranged from 38 to 81% for COD and 36 to 83% for ammonia. In the /S$_2$O$_8^{2-}$/H$_2$O$_2$ system, S$_2$O$_8^{2-}$ releases sulfate radicals (Equations (3) and (4)), which have powerful oxidation effect on organics (Huling & Pivetz 2006; Deng & Eyzske 2011; House 1962).

\[
\text{S}_2\text{O}_8^{2-} + 2\text{H}^+ + 2e^- \rightarrow 2\text{HSO}_4^{-}
\]

\[
\text{SO}_4^{2-} + \text{HO}^* \rightarrow \text{SO}_4^{2-} + \text{OH}^-
\]

The initiation of sulfate radicals during persulfate oxidation can be significantly enhanced by using some
applications, such as heat and UV radiation (Equation (5)) (Block et al. 2004; Gao et al. 2012).

\[
S_{2}O_{8}^{2-} + \text{Initiator} \rightarrow SO_4^{2-} + SO_4^{2-}
\]  

(5)

Although persulfate reagent can act as a direct oxidant, its reaction rates are limited for high recalcitrant contaminants (Renaud & Sibi 2001). The effect of alkalinity on persulfate activation was presented by Furman et al. (2011). At high pH, persulfate can be activated by initiating sulfate radical under the effect of the hydroxyl radical (Equation (6)) (Ocampo 2009).

\[
S_{2}O_{8}^{2-} + OH^+ \rightarrow HSO_4^{+} + SO_4^{2-} + \frac{1}{2}O_2
\]  

(6)

Consequently, persulfate at high pH led to the release of sulfate radicals under the direct reaction of hydroxyl radical. Accordingly, in our new reaction system, H$_2$O$_2$ plays a significant role by increasing hydroxyl radicals in the solution, which directly activate persulfate and initiate sulfate radicals. The removal efficiencies of the target parameters were improved by increasing both S$_2$O$_8^{2-}$ and H$_2$O$_2$ dosage. Heat resulting from H$_2$O$_2$ decomposition also contributes to initiating sulfate radicals. During oxidation, the temperature was monitored and ranged between 40 and 50°C. An increase in temperature was observed when H$_2$O$_2$ dosage was increased. However, an excess amount of S$_2$O$_8^{2-}$ would consume hydroxyl radicals and inhibit the reaction. Nevertheless, sulfate radicals would still have an ability to oxidize the target pollutants despite the increase in S$_2$O$_8^{2-}$ dosage. Deng & Ezyske (2011) achieved 91% and 100% removal efficiencies for COD and ammonia, respectively, from leachate using a sulfate radical oxidation at pH 4. The effect of initial pH variation was examined to determine the optimal pH for the S$_2$O$_8$/H$_2$O$_2$ system. The removal of COD and ammonia was increased by increasing pH. Although persulfate oxidation would be more active at high pH, however, higher removal for COD and NH$_3$-N has been reported at reported at low pH (4) with high temperature (40°C) and higher persulfate dosage (Deng & Ezyske 2011). The reaction time was varied between 10 min and 120 min to determine optimal experimental conditions. The results demonstrated that the degradation of organics and ammonia of leachate were improved as reaction time increased.

Analysis of variance

Table 4 presents the ANOVA regression parameters for the predicted response models and other statistical parameters for COD and NH$_3$-N removal. The data given in this table demonstrate that all of the models were significant at a 5% confidence level, given that the $P$-values were less than 0.05. The coefficients of determination obtained in the current study for the COD and NH$_3$-N removal rates ($R^2 = 0.965$ and 0.968 respectively) were all higher than 0.80. A high $R^2$ value represents good agreement between the
calculated and observed results and shows a desirable and reasonable agreement with the adjusted $R^2$. The pred. $R^2$ of 0.8777 and 0.882 is in reasonable agreement with the adjusted $R^2$ of 0.9388 and 0.942 for COD and NH$_3$-N, respectively (Joglekar & May 1997; Nordin et al. 2004). The adequate precision (AP) ratio of the models varied between 24.1 and 29.3, which is desirable and adequate ($>4$).

In the current study, two quadratic models were significant model terms (Table 4). Insignificant model terms, which have limited influence, were excluded from the study to improve the models. Based on the results obtained, the response surface models constructed for predicting COD and NH$_3$-N removal efficiency were considered reasonable. The final regression models, in terms of their coded factors, are presented in Equations (7) and (8).

**COD removal (%)**

$$\text{COD removal} = 50.6 + 4.50A + 2.56B + 10.3C - 7.03A^2 - 5.53B^2 + 18.5D + 2.13AC + 5.00AD + 1.65 BC 3.25 BD - 2.50 CD$$

**NH$_3$-N removal (%)**

$$\text{NH}_3\text{-N removal} = 48.5 + 2.11A + 7.72C + 7.44D - 12.1A^2 + 7.38B^2 + 15.9D^2 + 1.69 AB + 1.94 AC + 0.0625 AD - 0.0625 BD + 2.44 CD$$

The adequate precision (AP) ratio of the models varied between 24.1 and 29.3, which is desirable and adequate ($>4$).

In the current study, two quadratic models were significant model terms (Table 4). Insignificant model terms, which have limited influence, were excluded from the study to improve the models. Based on the results obtained, the response surface models constructed for predicting COD and NH$_3$-N removal efficiency were considered reasonable. The final regression models, in terms of their coded factors, are presented in Equations (7) and (8).
software to confirm that the selected models provided an adequate approximation of the real system. Normal probability plots can be used in evaluating the models and predicting whether the residuals follow a normal distribution. Figure 1(a) and 1(b) demonstrates the normal probability plots for the standardized residuals for COD and NH₃-N. In the normal probability plots, as shown in Figure 1, the points fall along a straight line for each case. However, some scattering is expected even with normal data; thus, the data can be considered to be normally distributed in the responses of certain models. The relationship between predicted and experimental values of COD and NH₃-N removals is shown in Figure 2. It can be seen that the correlation between the predicted and experimental values indicates that the predicted and experimental values were in reasonable agreement. It means that the data fit well with the model and give a
convincingly good estimate of response for the system in the experimental range studied.

The perturbation plots in Figure 3(a) and 3(b) show the comparative effects of all independent variables on COD and NH$_3$-N removal. As shown in Figure 3, the sharp curvature of the three factors (A) persulfate dosage, (B) H$_2$O$_2$ dosage, and (C) pH indicates that these variables are very sensitive as concerns COD and NH$_3$-N removal efficiency, while the flat curve of contact time (D) shows that this is a less sensitive factor as regards COD and NH$_3$-N removal efficiency.

**Treatment efficiency and optimization process**

To assess the interactive relationships between independent variables and the responses of certain models, 3D surface response plots were utilized by Design Expert 6.0.7 software. The maximum levels of COD and NH$_3$-N removal were 82% and 85%, respectively (Figures 4 and 5). The effectiveness of combined persulfate and H$_2$O$_2$ in COD and NH$_3$-N removal was illustrated in Figures 4 and 5(a). The maximum removal value for COD was obtained at maximum persulfate and H$_2$O$_2$ dosages of 5.88 g and 8.63 g, respectively, pH 11 and reaction time 120 min. However, the best removal of ammonia was obtained at 4.2 g persulfate dosage. Persulfate and H$_2$O$_2$ concentrations were the two actual factors for improving COD and ammonia removal. The maximum persulfate dosage combined with the excess amount of H$_2$O$_2$ at higher pH (11) resulted in the formation of hydroxyl and sulfate radicals, which is considered as a further improvement of the oxidation process efficiency. However, an excess amount of sulfate ions participates in consuming an amount of hydroxyl radical. This condition inhibits a part of the oxidation and decreases the efficiency of the compound pollutant elimination in the treatment process (Tizaoui et al. 2007). Deng & Ezyske (2011) observed the same results after using sulfate radical for leachate treatment. The interaction between pH, persulfate and H$_2$O$_2$ is illustrated in Figures 4 and 5(c), 5(d) and 5(e); the removal efficiency was reduced at natural pH and was found to improve at lower and higher pH value using 4.2 g and 6.17 g persulfate and H$_2$O$_2$ dosage, respectively.

Optimization was carried out to determine optimum values of COD and NH$_3$-N removal efficiency using Design Expert 6.0.7. According to the software optimization step, the desired goal for each operational condition (i.e., persulfate dosage, H$_2$O$_2$ dosage, reaction time, pH) was chosen ‘within’ the range, while persulfate dosage was chosen within minimum. The responses (COD and NH$_3$-N) were defined as maximum to achieve the highest performance for COD and NH$_3$-N removal. The program combined the individual desirability into a single number, and then searched to optimize this function based on the response goal. Thus, the optimum working conditions and respective removal efficiencies were established (Table 5): about 82.1 and 85.4% removal of COD and NH$_3$-N is predicted, respectively, based on the model and under optimized operational conditions. The desirability function value was found to be 1 for these optimum conditions. An additional laboratory

![Figure 3](https://iwaponline.com/wst/article-pdf/73/1/102/464483/wst073010102.pdf)
experiment was then performed to confirm optimum results. The said conducted laboratory experiments agreed with the predicted response, and values of 80% and 82% removal efficiency of COD and NH₃-N were obtained, respectively.
Figure 5  |  Response surface for NH\textsubscript{3}-N removal efficiency as a function of persulfate dosage (4.97 g), H\textsubscript{2}O\textsubscript{2} dosage (7.29 g), pH (11) and reaction time (116 min) (persulfate/H\textsubscript{2}O\textsubscript{2} oxidation).
To investigate the effectiveness of combined persulfate and H$_2$O$_2$ reagents, leachate sample was treated with persulfate only and H$_2$O$_2$ only (Figure 6). The efficiency of persulfate only was found to be limited for the removal of COD and ammonia (45%, and 46%, respectively). These lower values were attributed to the high concentration of organics in the leachate containing a considerable amount of inactivated persulfate from the beginning of the reaction. Thus, the persulfate oxidation is more efficient for leachate treatment than H$_2$O$_2$ only. Although the performance of sequential oxidation using persulfate followed by H$_2$O$_2$ was improved, the effectiveness of simultaneous S$_2$O$_8^{2-}$/C0/H$_2$O$_2$ system was higher than other applications (80% and 82% removal for COD and ammonia, respectively).

In comparison with ozone/persulfate oxidation, Abu Amr et al. (2016) used ozone in activation of persulfate for stabilized leachate treatment. The results achieved 70% and 72% removal efficiencies for COD and NH$_3$-N, respectively. The optimal reaction time consumed in the ozone/persulfate process was 210 min, i.e., higher than that in the current new process. Besides, the performance of S$_2$O$_8^{2-}$/H$_2$O$_2$ in removing COD and NH$_3$-N was much higher. Furthermore, the new treatment process (persulfate/H$_2$O$_2$) was also found to be more efficient than ozone/H$_2$O$_2$ for stabilized leachate treatment. Tizaoui et al. (2007) obtained 50% and 94% removal efficiencies for COD and color removal, respectively.

**CONCLUSION**

In the current study, the performance of employing H$_2$O$_2$ as advanced oxidation for the treatment of solid waste leachate by S$_2$O$_8^{2-}$/H$_2$O$_2$ was investigated. The optimum application for the treatment was conducted with respect to operational conditions, namely, S$_2$O$_8^{2-}$ and H$_2$O$_2$ concentration, pH variation, and reaction time. The performance of combined H$_2$O$_2$ and S$_2$O$_8^{2-}$ was more efficient for stabilized leachate treatment than the S$_2$O$_8^{2-}$ system and the H$_2$O$_2$ system. Accordingly, the new treatment increased the oxidation potential of the organics and achieved better ammonia removal efficiency during oxidation. Furthermore, the performance of the new process was found to be more efficient for leachate treatment than the ozone/persulfate oxidation process.

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<thead>
<tr>
<th>Table 5</th>
<th>Optimization results for COD and NH$_3$-N maximum removal efficiency</th>
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<tbody>
<tr>
<td>Persulfate (g)</td>
<td>H$_2$O$_2$ (g)</td>
</tr>
<tr>
<td>4.96</td>
<td>7.29</td>
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<td>Lab. experiment</td>
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*RT: reaction time.
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