



Application of Cavitation Ozonation Process on Recalcitrant Organic Matter Degradation from Stabilized Landfill Leachate

Setyo Sarwanto Moersidik^{1*}, Letti Annasari¹, Rudi Nugroho²

¹*Environmental Engineering Study Program, Department of Civil Engineering, Faculty of Engineering, Universitas Indonesia, Kampus UI Depok, Depok 16424, Indonesia*

²*Center of Technology for the Environment, Agency for the Assessment and Application of Technology, Geosystem technology 820 Building, Puspiptek, Serpong, Tangerang 15314, Indonesia*

Abstract. Treatment of stabilized leachate as a by-product of municipal landfills has been a significant challenge as the leachate contains recalcitrant organic matter which has low biodegradability. In this study, the efficacy of the advanced oxidation process using cavitation-ozonation to remove recalcitrant organic matter in leachate samples from TPST Bantar Gebang was evaluated. Several operational and process parameters including pH, ozone flowrate, and contact time were varied to determine the best conditions for removing recalcitrant organic matter represented by Chemical Oxygen Demand (COD). This study determined the optimum operating conditions for the cavitation-ozonation process: pH 11, ozone discharge of 3 L/min, and contact time of 30 minutes. The result was a COD removal efficiency of 20.37%, an increase of 52.06% in the concentration of BOD₅, and a 90% increase in the ratio of BOD₅ to COD. This study has shown that cavitation-ozonation is an effective pre-treatment, as it increases the biodegradability of stabilized leachate and reduces the load on subsequent treatment processes.

Keywords: Cavitation-ozonation; Leachate treatment; Recalcitrant organic matter

1. Introduction

Population growth has led to considerable increases in municipal solid waste generation. This is a significant concern as, without proper management, the amounts of solid waste burden the environmental carrying capacity. Improper management of solid waste creates leachate—water that leaks into the cells of waste. When it percolates the leachate brings dissolved and suspended material from the waste decomposition process (Tchobanoglous et al., 1993). Several factors significantly influence the generation leachate in landfills: the water content in the waste, precipitation, evaporation, the composition of organic waste, operational mode, and groundwater flows (Xing et al., 2013). Leachate should not be discharged into water bodies without being treated to remove high levels of organic matter. The possible results when leachate is not managed properly include fires and explosions, unpleasant odors, and the pollution of groundwater and surface water (Abd El-Salam and Abu-Zuid, 2015).

The Bantar Gebang Integrated Waste Treatment Area (TPST Bantar Gebang) in Bekasi, West Java, is one of the largest landfill sites in Indonesia. Every day it receives 6500–7000

*Corresponding author's email: ssarwanto@eng.ui.ac.id, Tel.: +62-21-7270029, Fax.: +62-21-7270028
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tons of municipal waste generated in Indonesia's Capital, Jakarta (Azanella, 2018). Bantar Gebang has five landfill zones and four leachate treatment plants (LTPs), where leachate is treated before being discharged into water bodies. The fundamental challenge in leachate treatment is to reduce the high concentration of recalcitrant organic compounds, which are difficult to degrade biologically. The presence of these compounds is indicated by high levels of chemical oxygen demand (COD) and a low ratio of biochemical oxygen demand (BOD) to COD (Gulyas, 1997). A predominant composition of recalcitrant organic compounds (> 60% of total organic carbon) is humic acids (Monje-Ramirez and Velásquez, 2004).

Various technologies have been implemented to remove recalcitrant organic compounds from stabilized leachate. Coagulation and chemical oxidation, chemical precipitation, activated carbon adsorption, and membrane processes are methods that have been recommended for treating stabilized leachate (Amokrane et al., 1997). Using FeCl_3 as the coagulant has been shown to remove about 82% to 85% of COD in leachate (Long et al., 2017). A three-step treatment process of aerobic activated sludge biological pre-oxidation (ASBO), coagulation/sedimentation, and photo-oxidation through a photo-Fenton (PF) reaction has yielded BOD concentrations less than 150 mg/L at the effluent (Silva et al., 2017). Nurrohman and Wardjito (2016) reported satisfactory results from combining physical and chemical treatments to remove low biodegradable contaminants using a flotation and coagulation process, whereas Lubis et al. (2019) removed more than 90% of COD in low biodegradable wastewater using electrocoagulation. Recently, there has been increased use of advanced oxidation processes (AOPs) to remove organic contaminants from wastewater. This is because the high oxidative power of ozone increases the biodegradability of the wastewater (Zhou et al., 2010, Gautam et al., 2019, Karamah et al., 2019). Pure ozone (O_3) and a combined gas ($\text{O}_3/\text{H}_2\text{O}_2$) were applied to increase the biodegradability of leachate. This increased the maximum BOD_5 by about 110% (Wang et al., 2004). Another study suggested that the AOP of raw leachate could remove 16% to 33% COD from raw leachate (Xu et al., 2018). The combination of photoelectron-oxidation (PEO) and NaClO enhanced Fe^{2+} coagulation was able to remove 75% to 85% of COD (Qiao et al., 2018). Removing up to 85% of COD was achieved by combining ozone with hydrogen peroxide or persulfate in the AOP process (Gautam et al., 2019).

There are critical benefits of applying AOP to treat landfill leachate. First, it does not generate harmful byproduct compounds into the environment. Second, the process is relatively rapid and more efficient in improving the biodegradability of the organic contaminant (Yasar et al., 2006). In this process, recalcitrant organic compounds are converted into stable organic compounds with a lower molecular weight. Third, AOP also oxidizes organic compounds into the most stable oxidation form, CO_2 and water, by complete mineralization. Thus, its biodegradability increases. Fourth, the remaining unreacted ozone is released into the air and decomposes into oxygen (Sharma et al., 2011).

Several studies have shown that leachate produced from TPST Bantar Gebang was contaminating the surrounding aquatic environment, and they suggested stricter operation of the landfill and more effective leachate treatment (Pujiindiyati and Sidauruk, 2015; Pujiindiyati et al., 2019). Unfortunately, despite its several benefits, there have been very few studies of the application of AOP to remove recalcitrant organic matter. Hence, this study aimed to evaluate the applicability of using the cavitation-ozonation process to treat leachate generated from landfills in Indonesia.

The evaluation was based on sets of experiments using samples collected from TPST Bantar Gebang. Outcomes included removal of recalcitrant organic compounds and improvement of the biodegradability of organic compounds. This process was carried out under alkaline conditions to encourage the formation of hydroxyl radicals, a strong oxidizing agent.

2. Methods

2.1. Sample and Analysis

The AOP reactor used in this study was designed with a diameter of 0.3 m and a height of 1.5 m (see Figure 1). It can process up to 34 L volume of leachate, considering the possibility of foaming during the cavitation–ozonation process. Ozone was produced by an ozone generator with a capacity of 1.2 g/h, and it was injected into the reactor (Moersidik et al., 2020).

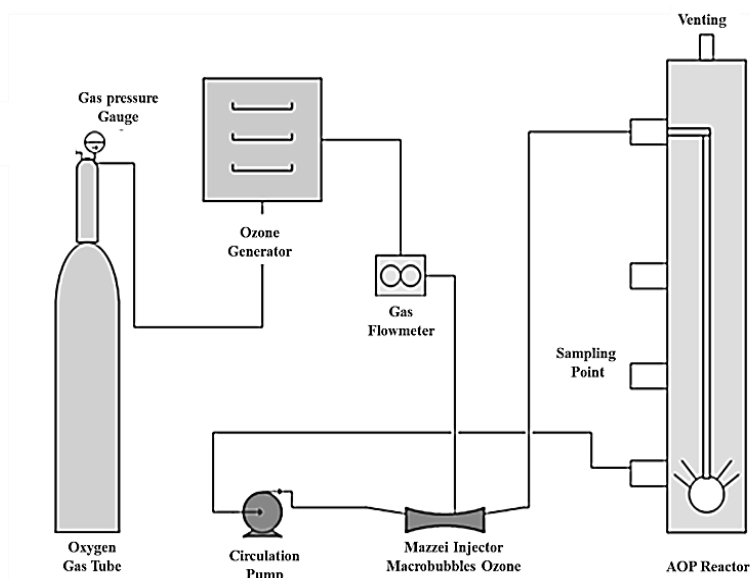


Figure 1 The AOP reactor used in this study, adapted from Moersidik et al. (2020)

This experiment used a sample of landfill leachate generated from landfill zone 3 at TPST Bantar Gebang, which was inactive and closed for disposal. It had an area of 25.41 Ha with 2,787,904.08 m³ of solid waste. A 1000-litre sample was taken from the inlet leachate treatment plant (IPAS 3) during the day in the rainy season. Approximately 5 litres of the leachate sample was taken to the Regional Environmental Laboratory of Jakarta for characteristic analysis.

For sample analysis, approximately 50 ml of leachate that had been processed in specific pH, ozone discharge, and contact time was collected from the valve at the bottom of the reactor. Then the pH, temperature, and COD concentration in the sample were measured. The pH was determined using a pH meter and pH paper, temperature was measured with a thermometer, and COD was determined by using a HAACH spectrophotometer, type DR 2800.

2.2. Experimental Set-Up

The AOP reactor system used in this study was adapted from our previous study on electroplating wastewater treatment (Moersidik et al., 2020). The reactor system comprised an acrylic reactor 0.3 m in diameter and 1.5 m high. The reactor can accommodate up to 100 litres of leachate. However, in this study, only 34 litres of leachate was processed to anticipate effects of foaming during the cavitation–ozonation process.

Ozone was injected into the reactor with a 0.5-inch-diameter venturi injector. Ozone was produced in situ using Ozotech OZ4PC10-F ozone generators. The production capacity of this ozone generator was 1.2 g O₃/h with oxygen feed gas. The reactor was also equipped with a sampling port 5 cm from the bottom of the reactor.

For the operation of the reactor, the variables are pH sample, ozone discharge (Q O₃), and contact time (t). The optimal operating conditions for the cavitation–ozonation process were determined by looking at the highest percentage of COD removal according to the factorial design applied. This was factorial design levels I and II, presented in Table 1 and Table 2. The variations of contact time used for levels I and II were determined by referring to previous studies where, during a contact time of 240 min, the effectivity of COD removal was seen in the first 60 minutes (Ntampou et al., 2006; Tizaoui et al., 2007). Thus, it was expected that during the operation period, a significant decrease in COD concentration could be observed until the condition became stable.

The significance of each independent variable was evaluated by analysis of variance (ANOVA) and multiple regression analysis. This resulted in a multiple linear regression Equation 1:

$$y = B_0 + B_1x_1 + B_2x_2 + B_3x_3 \quad (1)$$

where y is the response (percentage of COD removal), B₀ is the intercept, B₀, B₁, and B₂ are the coefficient values of the intercept, pH, Q O₃, and time, respectively, and x₁, x₂, and x₃ are the values of pH, Q O₃, and time.

Table 1 Factorial design level I

| Experimental condition (C) | pH | Q O ₃ (L/min) | t (min) |
|----------------------------|------|--------------------------|---------|
| C1 | 8.3 | 2.0 | 60 |
| C2 | 8.3 | 3.5 | 60 |
| C3 | 11.0 | 2.0 | 60 |
| C4 | 11.0 | 3.5 | 60 |
| C5 | 8.3 | 2.0 | 150 |
| C6 | 8.3 | 3.5 | 150 |
| C7 | 11.0 | 2.0 | 150 |
| C8 | 11.0 | 3.5 | 150 |

Table 2 Factorial design level II

| Experimental condition (C) | pH | Q O ₃ (L/min) | t (min) |
|----------------------------|------|--------------------------|---------|
| C9 | 11.0 | 3.0 | 30 |
| C10 | 11.0 | 4.0 | 30 |
| C11 | 11.0 | 3.0 | 90 |
| C12 | 11.0 | 4.0 | 90 |

2.3. Removal of Kinetics Model

The constants of the COD removal rate were determined by plotting COD concentration versus time. Three models including zero, first-, and second-order reaction kinetics (Equations 1, 2, and 3, respectively) were used to identify the most appropriate model.

$$\frac{dC}{dt} = -k \quad (2)$$

$$\frac{dC}{dt} = -kC \quad (3)$$

$$\frac{dC}{dt} = -kC^2 \quad (4)$$

where C is COD concentration, k is the degradation rate, and t is the measurement time. The best-fitting model was determined by linear regression from the plotted data based on the coefficient of determination value (R^2).

3. Results and Discussion

3.1. Characterization of Leachate

Table 3 shows the characteristics of leachate classified by age and its comparison with the characteristics of leachate from Zone 3 (Foo and Hameed, 2009).

Table 3 Classification of landfill leachate

| | Zone 3 landfill leachate (experimental data) | Young | Intermediate (Foo and Hameed, 2009) | Stabilized |
|--------------------------------------|---|-------------------------------|--|------------------------|
| Age (year) | > 10 | < 5 | 5-10 | > 10 |
| pH | 8.4 | < 6.5 | 6.5-7.5 | > 7.5 |
| COD (mg/L) | 3,168.32 | > 10,000 | 4000-10,000 | < 4000 |
| BOD (mg/L) | 281.98 | > 5,000 | 400-5000 | <400 |
| BOD ₅ /COD | 0.089 | 0.5-1.0 | 0.1-0.5 | < 0.1 |
| Organics | not checked | 80% volatile fatty acid (VFA) | 5%-30% VFA + humic and fulvic acids | humic and fulvic acids |
| NH ₃ -N (mg/L) | 576 | < 400 | N.A. | > 400 |
| TOC/COD | 0.413 | < 0.3 | 0.3-0.5 | > 0,5 |
| TKN (mg/L) | not checked | 100-200 | n.a. | n.a. |
| Heavy metal (mg/L) | low | low to medium | low | low |
| Biodegradability | low | high | medium | low |
| CO ₃ ²⁻ (mg/L) | 220.41 | n.a. | n.a. | n.a. |
| Cl ⁻ (mg/L) | 1975.67 | n.a. | n.a. | n.a. |
| SO ₄ ²⁻ (mg/L) | 189.44 | n.a. | n.a. | n.a. |

Qualitatively, the leachate from Zone 3 was dark brown or almost black and pungent. Based on the data in Table 1, the leachate contained a high concentration of ammonia (NH₃-N) and COD, but a low concentration of BOD₅. This corresponds to the characteristics of stable leachate from landfill aged over 10 years. Meanwhile, the concentrations of metals such as chromium hexavalent (Cr⁶⁺), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), manganese (Mn), nickel (Ni), silver (Ag), and zinc (Zn), and were low.

The pH of the leachate was 8.4. This alkaline pH value was helpful for treating leachate using AOPs. Leachate also contained concentrations of CaCO₃, chloride (Cl⁻), and sulfate (SO₄²⁻). According Tizaoui et al. (2007), carbonate (CO₃²⁻), chloride (Cl⁻), and sulfate (SO₄²⁻) in the advanced oxidation process may reduce the ion oxidation strength of the hydroxyl radicals (OH•). The leachate characterization test showed the following concentrations: carbonate (CaCO₃) was 220.41 mg/L, chloride (Cl⁻) was 1975.67 mg/L, and sulfate (SO₄²⁻) was 189.44 mg/L.

3.2. COD Removal in Optimum Conditions

A comparison of the efficiency of COD removal in some operating conditions is shown in Figure 2. From these experiments, the highest level of COD removal was 20.37% obtained in experiment 9 (shown in the bold bar in Figure 2, operating conditions: leachate pH 11, discharge Ozone 3.0 L/min, and contact time of 30 minutes).

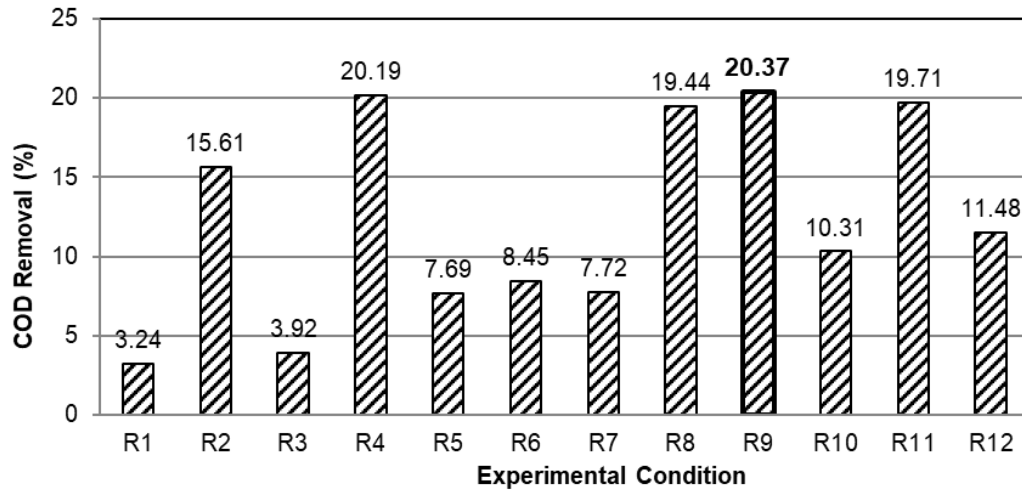


Figure 2 Efficiency comparison of COD removal in some operating conditions

Earlier research showed that using oxidation to treat leachate and wastewater, such as the ozonation method can reduce COD concentration by 27% to 45%, while combining ozonation with other treatments such as hydrogen peroxide or Fenton can raise its efficiency to 35% to 80% (Tizaoui et al., 2007; Ratanatamskul and Auesuntrachun, 2009; Amaral-Silva et al., 2016). Aljuboury and Palaniandy (2017) showed that using other advanced oxidation treatment methods such as solar photo-Fenton was effective at removing up to 84% of COD.

3.2.1. pH Influence in COD removal

Based on the results of research conducted at various discharge ozone and the contact time corresponding factorial design, the percentage of COD removal from leachate at pH 11 was higher than the percentage removed at the original pH (pH 8.3). This was similar to the findings by Cortez et al. (2010a) at alkaline pH. They found that more molecules of ozone decomposed to form hydroxyl ions (OH^-) and hydroxyl radicals ($\text{OH}\cdot$). Hydroxyl ions (OH^-) and hydroxyl radicals ($\text{OH}\cdot$) are both stronger oxidants than ozone (O_3), and they have unselective characteristics in nature. Thus, they can oxidize more types of compounds compared with Ozone (O_3). At pH 11, Cortez et al. (2010b) found the concentration of ozone to be 112 mg/L with operating conditions of 60 min contact time. This resulted in the highest COD removal, 40%.

3.2.2. Influence of ozone discharge on COD removal

Based on the results of Level I experiments, at ozone discharges of 2 L/min and 3.5 L/min; the higher COD removal was at an ozone discharge of 3.5 L/min. At ozone discharge 2 L/min, the concentration of ozone that contacted the leachate in the reactor was equal to 7.06 mg O_3 /L. At an ozone discharge of 3.5 L/min, the concentration of ozone that contacted the leachate was equal to 12.35 mg O_3 /L.

Higher COD removal was caused by a higher concentration of contacted ozone at an ozone discharge of 3.5 L/min This also affected the higher solubility of ozone in water. Karamah et al. (2010) investigated the mass transfer phenomena that happen in the microbubble ozonation process. Their results showed that an increase in the flow rate of ozone increased the kinetic energy of the ozone. This made it easier to dissolve the ozone in water. Higher solubility of ozone in water increases the chances that hydroxyl radicals will form, resulting in higher degradation of recalcitrant organic compounds and a greater reduction in COD concentration.

Level II experiments were conducted at ozone discharge rates of 3 L/min and 4 L/min. The highest percentage of COD removal occurred at an ozone discharge of 3 L/min. At that

discharge rate, the concentration of ozone that contacted the leachate in the reactor was equal to 10.59 mg O₃/L. At an ozone discharge of 4 L/min, the concentration of ozone that contacted the leachate was equal to 14.12 mg O₃/L. The increasing ozone concentration did not have a significant impact on the rate of ozone solubility in the leachate. The optimal COD removal generated at the lower ozone discharge occurred only when the ozone discharge was 3 L/min. It is assumed that the higher the ozone discharge, the more complex the turbulence during the contact of ozone with water. This disrupts the ozone dissolving process. This higher ozone discharge allegedly also reduces the duration of the leachate contact. Thus, not all the ozone was dissolved in water. Ozone which was not dissolved was partially separated back into the air. This assumption must be proven through further research.

3.2.3. Influence of contact time on COD removal

From this research, the highest COD removal occurred with a contact time of 30 minutes. In this study, the advanced oxidation process that used microbubble cavitation-ozonation to contact the ozone with leachate was performed by a semi-batch system. In that system, a small amount of leachate was recirculated through an injector so it contacted the ozone. Re-circulation was done by using a pump. During this continuous re-circulation process, the temperature continued to rise. In the first 30 minutes, the temperature rose only 1.5°C from the initial leachate temperature of 27.5°C. However, as contact time increased, the temperature also rose. At 150 minutes of contact time, the leachate temperature increased by 7.5°C. This reduced the ozone solubility of leachate.

Ozone stability in the formation of hydroxyl radicals was influenced by temperature (Sawyer et al., 2003). Some results showed that an increase in temperature from 0°C to 30°C led to reduced solubility of ozone in water and increased the ozone decomposition rate (Munter, 2001). At temperatures between 15°C and 20°C, ozone half-life in water was between 15 min and 20 min, whereas between 30°C and 35°C, the half-life was between 8 min and 12 min (Lenntech, 2014).

Thus, it can be concluded that with 30 min of contact time, a rise in temperature affects the solubility of ozone in water. On the other hand, rising temperatures also accelerate the decomposition of ozone into hydroxyl radicals, which were reactive but less competitive to react. However, in this study, the reduced ozone solubility was predicted to have more significant effects on the leachate than increasing the number of hydroxyl radicals, thus reducing the efficiency of COD removal.

3.2.4. The significance of operating variables in COD removal

The results of the ANOVA and multiple regression analysis ($\alpha = .05$) are shown in Table 4. The multiple regression analysis could explain 77% of the variability of COD removal as a value of R² of 0.776 was obtained.

Table 4 Results of ANOVA and multiple regression analysis on the operating variables in COD removal

| Variables | Unstandardized coefficients | | Standardized coefficients | t | p value |
|------------|-----------------------------|------------|---------------------------|--------|---------|
| | B | Std. Error | B | | |
| (Constant) | -22.676 | 12.529 | | -1.810 | .145 |
| pH | 1.504 | 1.102 | 0.323 | 1.365 | .244 |
| Q | 6.860 | 1.983 | 0.819 | 3.460 | .026 |
| t | 0.001 | 0.033 | 0.006 | 0.025 | .981 |

The results showed that the standardized coefficient value of ozone flowrate was highest (0.819) compared to the values from variations of pH and operation time (0.323 and 0.006, respectively). This suggests that the ozone flowrate is the most influential variable in removing COD in wastewater. Furthermore, the ozone flowrate was the only variable that made a statistically significant difference; its p value was below .05. This suggests that broader ranges in the values of pH and operation time should be investigated and analyzed to increase their significance in removing COD from wastewater.

3.3. Kinetics of Removing COD

Figure 3 shows the results for the COD removal kinetics. The best-fitted model ($r^2 \approx 1$) was obtained from the second-order model, with an r^2 value of 0.9681. The constant (k) of the COD removal rate was approximately $5 \times 10^{-8} \text{ M}^{-1} \cdot \text{s}^{-1}$. The second-order model was also found to be best fitted for the oxidation process on landfill leachate in previous studies, such as in solar photo-Fenton reaction with the constant (k) of the COD removal rate was $2.10^{-6} \text{ L/g} \cdot \text{min}$ (Aljuboury and Palaniandy, 2017). In (Deng et al., 2020) research that discussed the electrochemical oxidation process shows that 60–64% COD was removed within 1 h at a rate constant (k) about $5.83 \times 10^{-3} \text{ L/g} \cdot \text{min}$. Other studies have shown that the second-order model constant (k) of the COD removal rate using the ozonation process and $\text{O}_3/\text{H}_2\text{O}_2$ process was found to be $3.10^{-6} \text{ L/mg} \cdot \text{min}$ and $2.10^{-6} \text{ L/mg} \cdot \text{min}$, respectively (Ratanatamskul and Auesuntrachun, 2009).

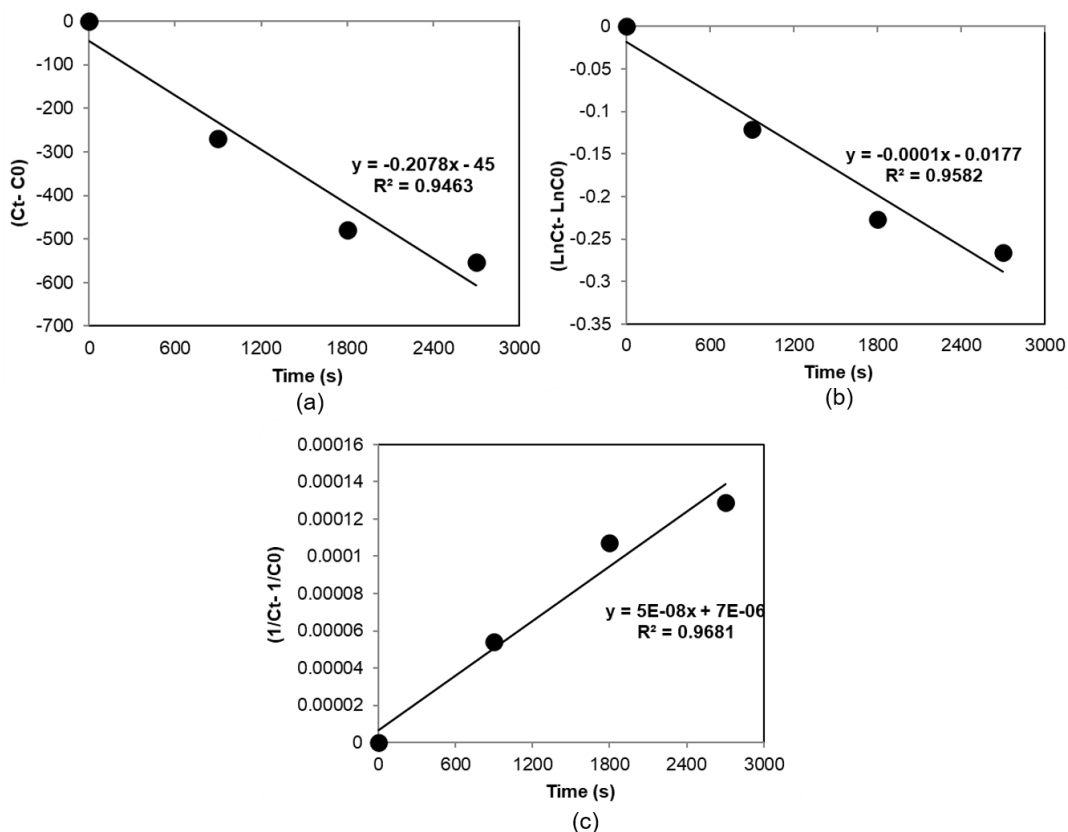


Figure 3 Kinetics of COD removal using the: (a) 0th order model; (b) 1st order model; and (c) 2nd order model

Organic compounds in the stabilized landfill leachate phase were recalcitrant organic compounds that were difficult to be degraded by usual biological processes because of its low ratio of BOD_5/COD , in this case, 0.098. Application of advanced oxidation process in

stabilized landfill leachate phase treatment was done to achieve 2 goals (Gao et al., 2015); to convert recalcitrant organic compounds into simpler organic compounds, and to oxidize organic compounds to their most stable oxidation form that was CO₂ and water by complete mineralization, and to improve leachate biodegradability. Thus, the cavitation–ozonation process conducted in this study could transform recalcitrant organic compounds in stabilized landfill leachate to organic compounds with lower molecular weight, thus increasing its biodegradability. It could be seen from the decrease of 20.37% COD and the increase of 52.06% BOD₅, increasing the ratio of BOD₅/COD was 0.188 (increased 1.9 times, or 90.95%).

Several studies of leachate ozonation showed similar trends. Amokrane et al. (1997) reported that an ozonation process removed between 20% and 50% of COD. Kurniawan et al. (2006) reported COD removal in the range of 25% to 85%. Cortez et al. (2010b) reported that biodegradability increased from 0.01 to 0.17 in the ozonation process with an ozone concentration of 112 mg O₃/L. Singh et al. (2014) reported that biodegradability increased from 0.02 to 0.20 in the ozonation process with an ozone concentration of 66.7 g O₃/m³. In this study, COD removal by the cavitation–ozonation process was equal to 20.37% because of the following factors: The hydroxyl radicals formation reaction rate that was slower than hydroxyl radicals reaction rate with ozone, high alkalinity (CaCO₃) in leachate, the Existence of chloride Ions (Cl⁻) and sulfate Ions (SO₄²⁻) in leachate, and high concentration of ammonia in leachate (Amokrane et al., 1997, Tizaoui et al., 2007).

4. Conclusions

In this study, the leachate from TPST Bantar Gebang was categorized as stabilized landfill leachate, characterized by a low BOD₅/COD ratio of 0.089 (< 0.1), pH 8.5 (> 7.5), COD concentration of 3186.32 mg/L (< 4000 mg/L), ammonia concentration of 576 mg/L (> 400 mg/L), and low metal concentration. The evaluation of the operational and process parameters of AOP in removing COD from leachate found that the optimal operating conditions for the cavitation–ozonation process were pH 11, ozone discharge of 3 L/min, and contact time of 30 min. Using those parameters, the efficiency of removing recalcitrant organic compounds (represented by COD) on the cavitation–ozonation process in optimal conditions was 20.37%, the increase in BOD₅ concentration was 52.06%, and the ratio of BOD₅/COD increased by 90% from 0.098 to 0.188. Finally, the analysis of the kinetics of the removal of recalcitrant organic compounds (represented by the COD parameter) on the cavitation–ozonation process was a second-order reaction. The reaction rate of COD removal (k) was 5×10⁻⁸ M⁻¹ s⁻¹. This study has shown the efficacy of the cavitation–ozonation process as a pre-treatment for stabilized leachate before biological treatment, as it increases the BOD₅ concentration resulting in leachate with more biodegradability.

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