

Environment

Evaluation of different ozone dosages in landfill leachate treatment

Avaliação de diferentes dosagens de ozônio no tratamento de lixiviado de aterro sanitário

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ABSTRACT

Landfill leachate treatment is challenging due to its heterogeneity and the presence of recalcitrant compounds in the effluent. Treatment processes with high removal efficiencies are required to meet the legal standards for effluent discharge into water bodies. This study aimed to characterize landfill leachate and evaluate the influence of different ozone dosages on the alteration of its physicochemical parameters. The leachate was collected from a landfill in the Candiota- RS municipality and characterized. The evaluated parameters included pH, true color, BOD (Biochemical Oxygen Demand), COD (Chemical Oxygen Demand), BOD₅/COD ratio, phosphorus, and total suspended solids, as well as the phytotoxicity of both raw and treated leachate. High removal values were observed for true color (75%), phosphorus (96%), and total suspended solids (90%) of the leachate when a dosage of 174.8 mg O₃ L⁻¹ was applied. These results demonstrate that satisfactory treatment of landfill leachate was achieved with low ozone concentrations. However, concerning organic matter and toxicity, it was found that the applied doses were insufficient to degrade all organic matter and reduce toxicity. Overall, ozone treatment is a promising alternative and can be applied as a pre-treatment for landfill leachate, with its biodegradability further enhanced through biological treatment, which is already common in landfills.

Keywords: Advanced oxidative processes; Ozonation; Real effluent

RESUMO

O tratamento de lixiviado de aterro sanitário é um desafio devido heterogeneidade e presença de compostos recalcitrantes no efluente. Para enquadrar o efluente aos padrões legais de lançamento

de efluentes em corpos hídricos, são necessários tratamentos com altas eficiências de remoção. O objetivo deste estudo foi caracterizar o lixiviado de aterro sanitário e avaliar a influência de diferentes dosagens de ozônio na alteração de seus parâmetros físico-químicos. O lixiviado foi coletado em aterro sanitário situado no município de Candiota – RS e caracterizado. Os parâmetros avaliados foram: pH, cor verdadeira, DBO, DQO, relação DBO_5/DQO , fósforo, sólidos suspensos totais, além de fitotoxicidade do lixiviado bruto e tratado. Pode-se observar altos valores de remoção de cor verdadeira (75%), fósforo (96%) e sólidos suspensos totais (90%) do lixiviado quando aplicada a dosagem de $174,8 \text{ mg O}_3 \text{ L}^{-1}$. Estes resultados demonstram que com baixas concentrações de ozônio atingiu-se resultados muito satisfatórios no tratamento do lixiviado de aterro sanitário. Com relação à orgânica e toxicidade, verificou-se que as doses aplicadas foram insuficientes para degradar toda a matéria orgânica e diminuir a toxicidade. Em termos gerais, a ozonização é uma alternativa promissora e pode ser aplicada como pré-tratamento do lixiviado, e ter sua biodegradabilidade aumentada com o tratamento biológico, que já é usual em aterros sanitários.

Palavras-chave: Processos oxidativos avançados; Ozonização; Efluente real

1 INTRODUCTION

Managing municipal solid waste is one of the major challenges in large cities (Hoss et al., 2022). In 2021 alone, Brazil generated approximately 81.8 million tons of municipal solid waste annually. Only 46.4 million tons of waste were collected and disposed of in sanitary landfills (ABRELPE, 2022). The waste disposed of in landfills undergoes decomposition processes, generating by-products such as gases and toxic liquids, which can harm health and the environment if not properly controlled (Gomes et al., 2022; Dantas et al., 2021). A significant impact is associated with the liquid from waste decomposition, as it poses a risk of contaminating surface and groundwater (Naveen et al., 2017; Aharoni et al., 2017).

According to NBR 8849/1985, the liquid that results from the decomposition of substances in solid waste is referred to as leachate. Leachate can contain high concentrations of ammoniacal nitrogen, chlorides, organic matter, recalcitrant organic compounds, heavy metals, and humic and toxic substances (Gomes et al., 2022; Scandelai et al., 2021; Aharoni et al., 2017; Renou et al., 2008). The heterogeneity and variability of leachate characteristics over time (Scandelai et al., 2021), as well as the presence of recalcitrant compounds (Gomes and Schoenell, 2018) make it challenging to adopt a single-treatment approach (Dantas et al., 2021). Biological treatment systems

(aerobic and anaerobic) are more frequently implemented due to low operational and maintenance costs (Hoss et al., 2022). Additionally, biological treatments are suitable for fresh leachate with a high BOD/COD ratio (Gomes and Schoenell, 2018). However, with the aging of landfills, these systems need to be improved to achieve the legal parameters for discharge into water bodies (Dantas et al., 2021).

An alternative for leachate treatment is Advanced Oxidation Processes (AOPs), such as ozonation. This technology relies on the reaction of a strong oxidizing agent ($\bullet\text{OH}$ or O_3), capable of oxidizing difficult-to-remove organic molecules in biological treatment processes (Apha et al., 2005; Scandelai et al., 2021). The application of ozone has gained attention due to its ability to meet emission concentrations established by regulations (Preethi et al., 2009). Furthermore, ozonation promotes the removal of organic matter, increases dissolved oxygen levels, and removes color, odor, and turbidity (Gomes and Schoenell, 2018; Preethi et al., 2009). Thus, this study aimed to evaluate the influence of different ozone dosages on organic load, true color, pH, phosphorus, and total suspended solids.

2 METHODOLOGY

This study was conducted using real effluent from a landfill in Candiota, Rio Grande do Sul, Brazil. Since its establishment in 2011, the landfill has received approximately 700 tons of municipal solid waste daily from 20 municipalities in the southern region of Rio Grande do Sul.

The landfill has an effluent treatment system composed of biological and physicochemical processes. The sample was collected from the first pond using a simple sampling technique. After collection, the effluent was stored in 20-liter containers, refrigerated, and immediately transported to the laboratory, where it was kept refrigerated until analysis.

First, the physicochemical characterization of the raw leachate was performed. Subsequently, the leachate was treated with different ozone exposure times to evaluate the effect of various ozone dosages. The parameters evaluated included pH, true color

(UC), Biochemical Oxygen Demand (BOD) (mg L^{-1}), Chemical Oxygen Demand (COD) (mg L^{-1}), BOD_5/COD ratio (mg L^{-1}), phosphorus - P (mg L^{-1}), total suspended solids - TSS (mg L^{-1}), and phytotoxicity of both raw and treated leachate. The results were analyzed by comparing means using Duncan's analysis (5%) and polynomial regression test using the WINSAT program (Machado and Conceição, 2002).

2.1 Ozonation Tests

The ozonation process was conducted in a 1-liter cylindrical glass column. An ozone generator (Panozon, 053308 P+70, Brazil) was used for ozone generation. The ozone gas entered the column through a pipeline. It passed through a cylindrical porous stone inserted at the base of the column to distribute the gas in small bubbles, thereby increasing the contact surface area between gas and liquid and enhancing the ozone reaction rate.

For each ozonation test, 250 mL of raw effluent was used. Different ozone dosages were applied for exposure times of 30, 60, 125, 180, 200, and 250 minutes, as shown in Table 1. The tests and physicochemical analyses were performed in duplicate under the same environmental conditions.

Table 1 – Ozone dosages applied in the tests and ozone exposure times

Ozonation time (min)	Ozone quantity (mg O_3)	Applied dosage ($\text{mg O}_3 \text{ L}^{-1}$)
0	0	0
30	21.9	87.6
60	43.7	174.8
120	65.5	262.0
180	131.1	524.4
200	145.7	582.8
250	182.1	728.4

Source: Authorship (2023)

An ozone generation test was conducted to verify the actual ozone generation capacity of the equipment. The iodometric method adapted from Apha (2005) and Rakness et al. (1996) was used for the test. This analysis determined an ozone generation rate of $43.7 \text{ mg O}_3 \text{ h}^{-1}$ or $174 \text{ mg O}_3 \text{ L}^{-1}$.

2.2 Physicochemical Characterization of Raw and Treated Leachate

The analyses of pH, true color, COD, BOD, TSS, and phosphorus were performed on the raw effluent immediately upon arrival at the laboratory and on the leachate treated at different ozone exposure times. The analyses were conducted according to the methodology described in the Manual of Water and Effluent Analysis (Quadro et al., 2016). The pH was measured using a benchtop pH meter (MS Tecnopon). The COD analysis was performed using the closed reflux titrimetric method.

2.3 Toxicity Bioassays

Phytotoxicity assays were conducted in triplicate using cucumber seeds (*Cucumis sativus*) and lettuce seeds (*Lactuca sativa*) as bioindicators, following the methodology adapted from Zucconi et al. (1981). Raw leachate and leachate samples treated with different ozone dosages were tested and compared to a control sample.

The assays were conducted in triplicate, and they involved adding 10 cucumber seeds or 20 lettuce seeds and 5 mL of leachate sample to Petri dishes. The control was prepared with 5 mL of distilled water. The dishes were covered with Parafilm to facilitate gas exchange and minimize moisture loss. Subsequently, the plates were incubated at 25°C for 48 hours.

After incubation, the number of germinated seeds was recorded, and the root length was measured using a digital caliper. The Germination Index (GI) was calculated using Equation 1 (Zucconi et al., 1981)

$$GI = G \frac{Lm}{Lc} \quad (1)$$

Where:

GI – Germination Index;

G – Number of germinated seeds in the sample divided by the number of germinated seeds in the control;

Lm - Average root length of the germinated seeds in the sample (mm);

Lc - Average root length of the germinated seeds in the control (mm);

3 RESULTS AND DISCUSSION

Table 2 presents the characterization of the raw leachate for the parameters of pH, true color, COD, BOD, BOD₅/COD ratio, phosphorus (P), and total suspended solids (TSS). These same characterizations were performed at different ozone exposure times (30, 60, 125, 180, 200, and 250 minutes) to evaluate the effect of different ozone dosages on each parameter individually.

Table 2 – Physicochemical characteristics of the raw leachate sample

pH	True color (UC)	COD (mg L ⁻¹)	BOD (mg L ⁻¹)	BOD ₅ /COD (mg L ⁻¹)	P (mg L ⁻¹)	TSS (mg L ⁻¹)
8.35	16,198.40	5,011.40	392.00	31.74	31.74	123.46

Source: Authorship (2023)

The physicochemical characterization of the raw leachate showed a basic pH and high true color value, indicating that it is a mature effluent with intense pigmentation and high concentrations of phosphorus and solids. The pH indicates leachate maturation, being basic for mature landfills and acidic for new landfills (Renou et al., 2008). Additionally, pH significantly influences the removal of refractory organic compounds present in leachate (Wu et al., 2021). Similarly, the strong coloration of the leachate can be attributed to humic and fulvic acids, mainly present in mature leachates (Renou et al., 2008).

After ozone application, characteristic changes were observed in the leachate, such as a decrease in pigmentation, a drastic reduction in phosphorus and solids concentrations, and an increase its biodegradability. Studies by Scandelai et al. (2021) and Miranda et al. (2021) suggest that ozone application promotes chemical changes favoring increased biodegradability as a pre-treatment. Additionally, according to Gomes and Schoenell (2018), increased biodegradability can be interpreted as the breakdown of recalcitrant compounds present in the leachate.

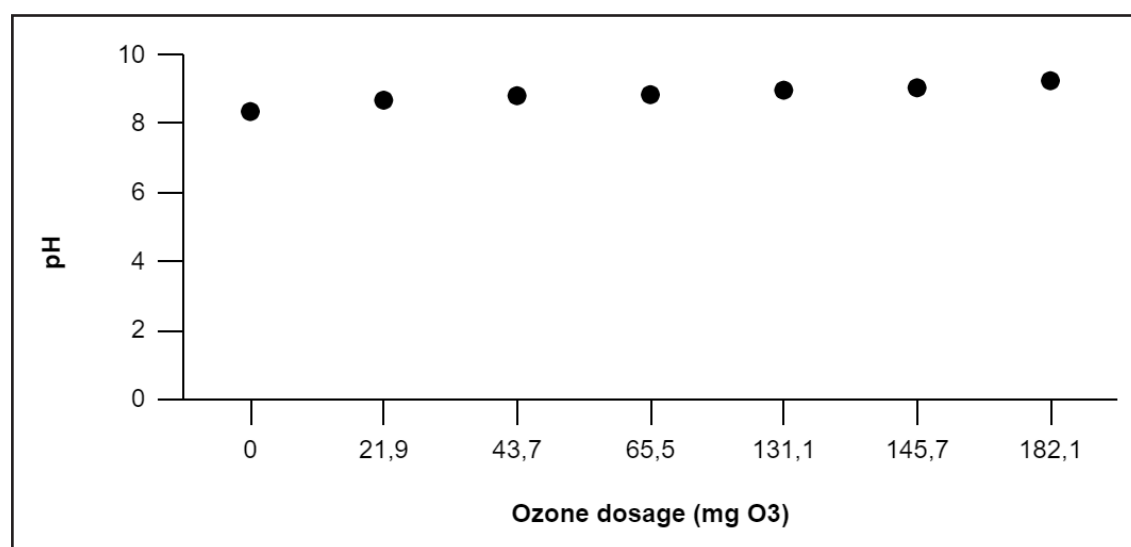
It is worth noting that biodegradability is one of the parameters used to classify leachates according to landfill age (Renou et al., 2008). This parameter is based on

the BOD₅/COD ratio, which ranges from 0.05 to 0.8. When this ratio is less than 0.1, it indicates that the effluent originates from a landfill with more than 10 years of operation (Renou et al., 2008). In this sense, the obtained value of 0.08 for the BOD₅/COD ratio of the raw leachate confirms that the studied leachate can be characterized as mature and originating from a landfill with more than 10 years of operation.

3.1 Effect of ozone dosages on pH

The effect of ozone dosages on the leachate's pH is presented in Figure 1, where a rapid increase from 8.3 to 9.2 can be observed. The fact that the ozonized leachate maintained a basic pH indicates that the ozone mechanism occurred indirectly through the hydroxyl radical, as this reaction is favored in alkaline media with a pH above 7.5, contributing catalytically to the oxidation of refractory organic matter (Hoffman et al., 2020; Webler et al., 2018; Gomes et al., 2020). Studies such as those by Scandelai et al. (2018) and Wu et al. (2004) observed that ozonation increased the natural pH of the leachate, possibly due to the removal of volatile fatty acids and the volatilization of CO₂ during ozone application, resulting in a reduction in H⁺ ion concentration.

Figure 1 – pH values as a function of applied O₃ dosages

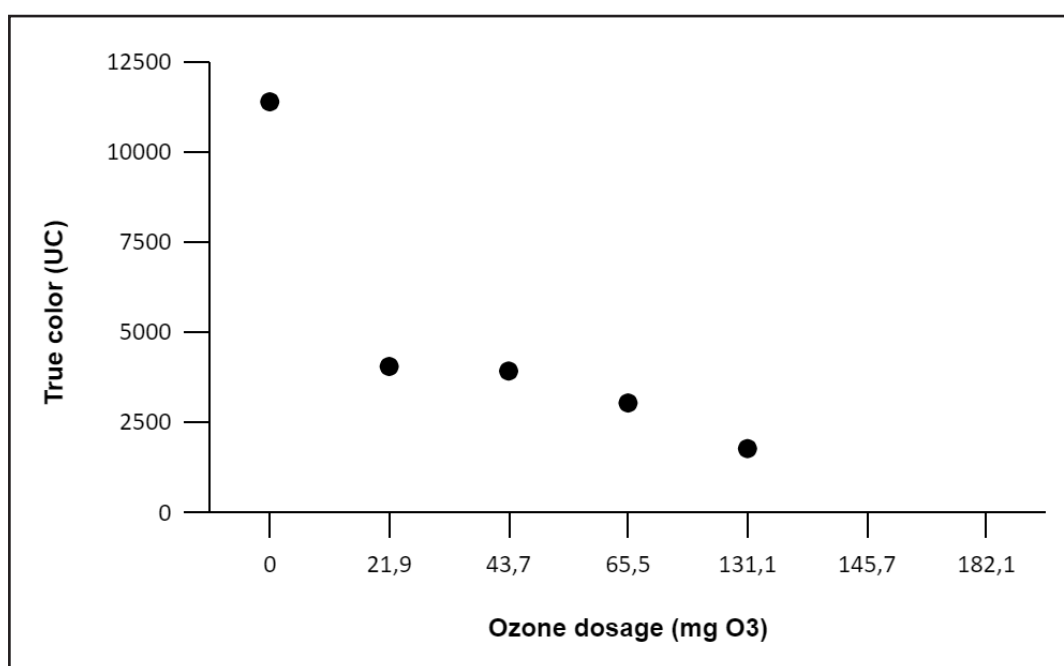


Source: Authorship (2023)

3.2 Effect of ozone dosages on true color

Figure 2 presents the effect of ozone dosage on the true color of the raw leachate. A clear decline in true color values can be observed at different ozone dosages. The gradual reduction of leachate color (from dark to yellowish) can be explained by the destruction of unsaturated organic compounds (chromophores) by the OH or O₃ radicals, thus reducing the true color of the leachate (Hoffman et al., 2020).

Figure 2 – True color concentration as a function of applied O₃ dosages



Source: Authorship (2023)

Table 3 – True color removal efficiency through applied O₃ dosages

Ozone exposure time (min)	Ozone dosage (mg O ₃)	Removal Efficiency (%)
0	0	0%
30	21.9	29.7%
60	43.7	75.0%
125	65.5	75.8%
180	131.1	81.2%
200	145.7	89.1%
250	182.1	93.4%

Source: Authorship (2023)

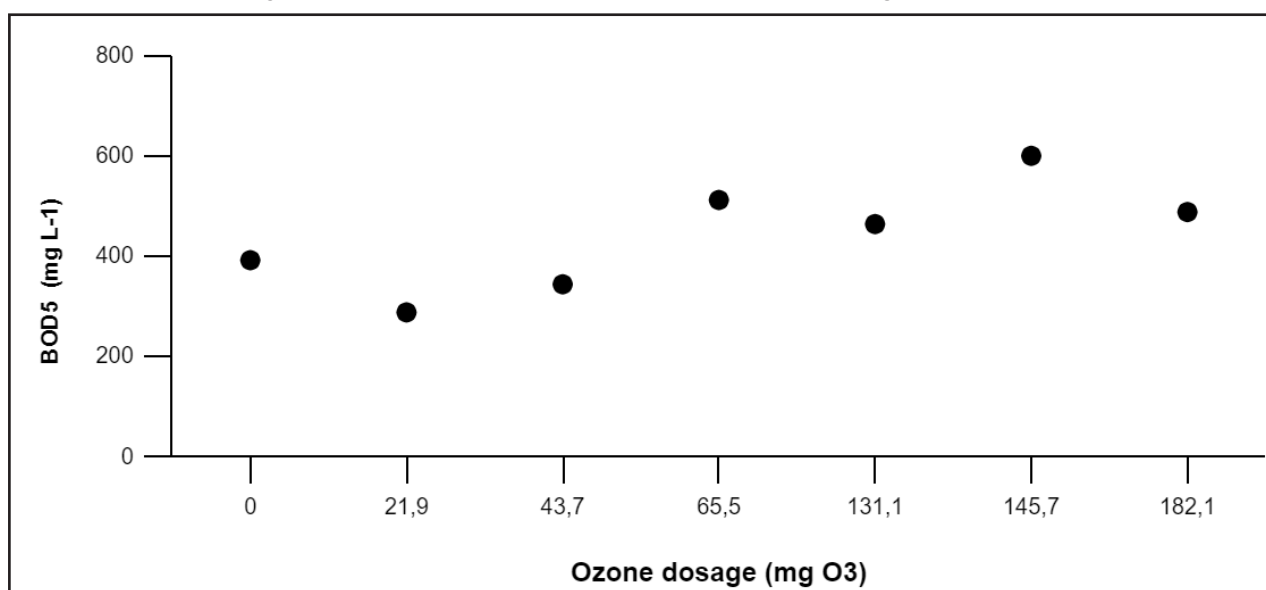
The changes in the true color of the leachate at different ozone dosages can also be expressed in removal efficiency, as shown in Table 3.

According to Table 3, at an ozone dosage of 43.7 mg O₃, the color removal efficiency reaches 75% at 60 minutes of treatment. This removal efficiency remains consistent until the ozone dosage reaches approximately 65 mg O₃. With an ozone dosage of around 145 mg O₃, the color removal efficiency approaches 90% at 200 minutes. These results demonstrate the efficiency of the process and indicate that color removal occurs quite rapidly. Similar results were reported by Webler et al. (2018), who applied combined techniques for the treatment of raw leachate, including an advanced oxidation process using ozone, and achieved an 85% color reduction with a consumption of 3,516 mg O₃ L⁻¹ and a 64% reduction with a consumption of 397 mg O₃ L⁻¹. Scandelai et al. (2019) achieved color removal of up to 96.33% by applying a 398 mg O₃ L⁻¹ dose.

3.3 Effect of ozone dosages on BOD₅, COD, and BOD₅/COD ratio

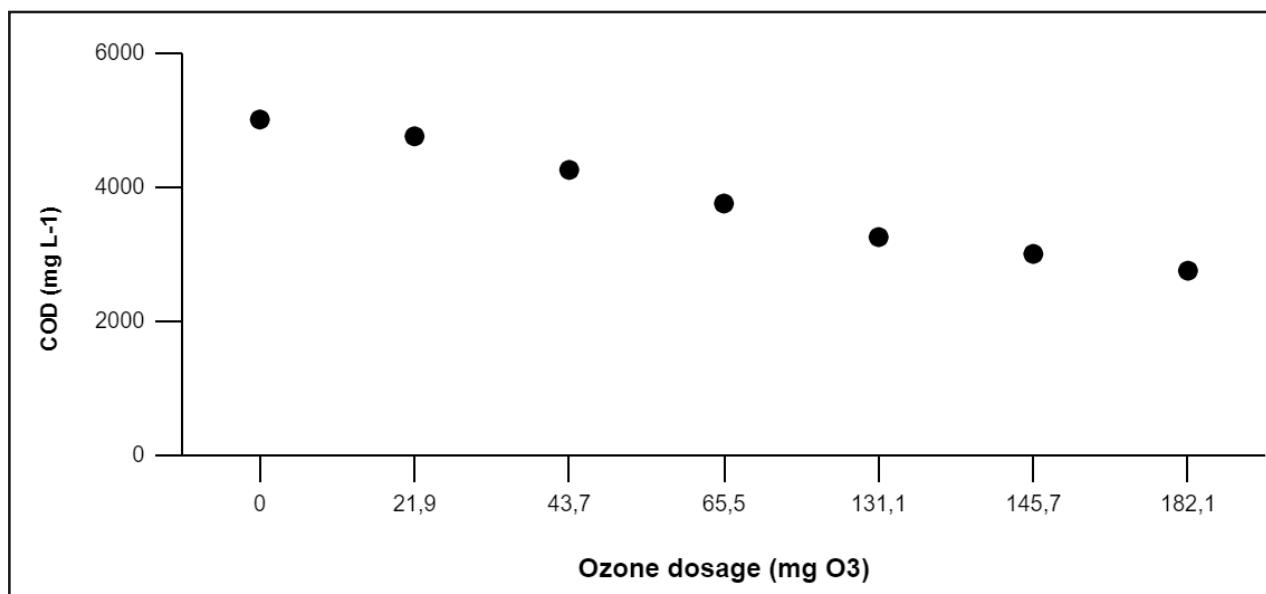
Figures 4(a) and 4(b) present the effect of different ozone dosages on BOD₅ and COD, respectively. Figure 4(c) shows the biodegradability ratio (BOD₅/COD) in landfill leachate treatment.

Figure 4(a) – BOD₅ concentration as a function of applied O₃ dosages



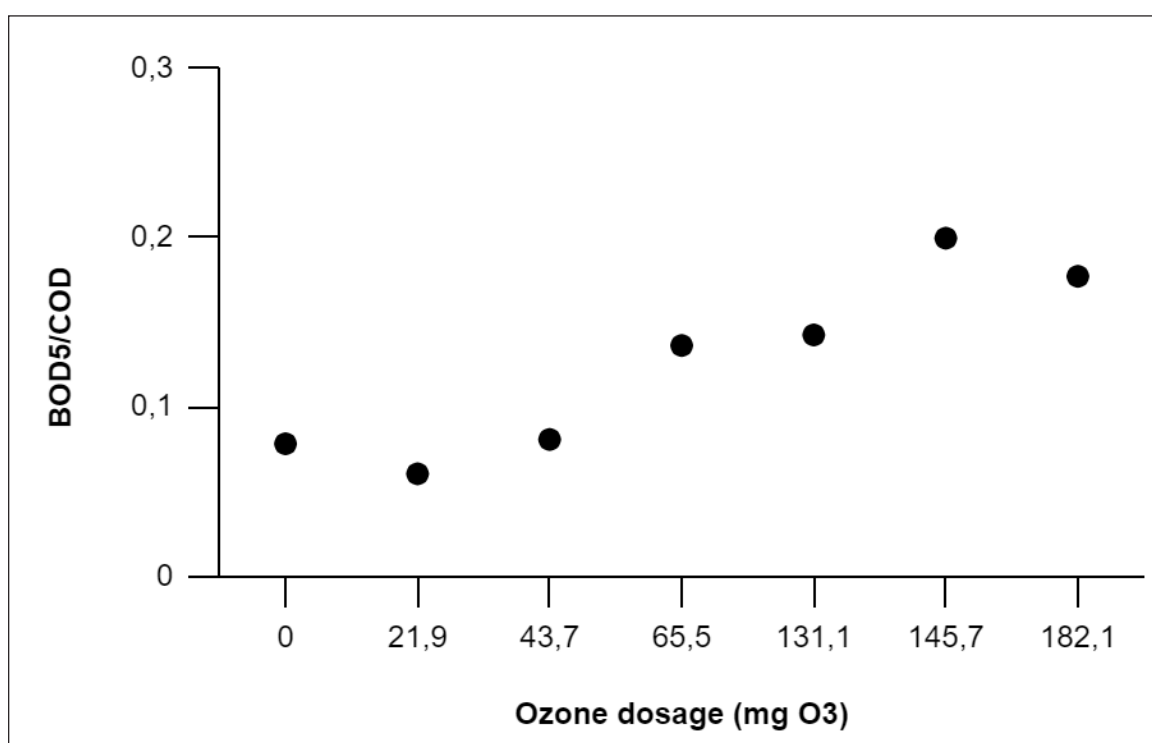
Source: Authorship (2023)

Figure 4(b) – COD concentration as a function of applied O_3 dosages



Source: Authorship (2023)

Figure 4(c) – The leachate's biodegradability ratio (BOD_5/COD)



Source: Authorship (2023)

Figure 4(a) shows the effect of ozone dosages on BOD_5 , where a small reduction can be observed at 30 minutes when a dosage of 21.9 mg O_3 was applied. At this dosage, the

BOD₅ concentration, initially at 392.33 mg L⁻¹, was reduced to 286.0 mg L⁻¹. Subsequently, an increase in BOD₅ values was observed, reaching approximately 53% higher than the initial BOD₅ concentration determined for the raw leachate. As mentioned by Cortez et al. (2010) and Miranda et al. (2021), this can be explained by the conversion of large refractory compounds into smaller and more biodegradable products during the ozone process. In Cortez et al.'s (2010) study, for example, BOD₅ concentrations increased by 80% to 180% with an increase in ozone dosage from 63 mg O₃ L⁻¹ to 112 mg O₃ L⁻¹.

Regarding the behavior of COD presented in Figure 4(b), a progressive decrease in COD values can be observed as the applied ozone dosages to the leachate increase. The removal efficiency reached 45% at the highest ozone dosage applied (728.4 mg O₃ L⁻¹) and a time of 250 minutes. These results are consistent with those of Cortez et al. (2010), who applied a dosage of 112 mg O₃ L⁻¹ to leachate treated by biological processes, with a COD of 743 mg L⁻¹, and achieved a 30% COD removal efficiency in 60 minutes. Miranda et al. (2021) also conducted ozone treatment tests on leachate and wastewater. They obtained a 20.3% COD removal in 120 minutes of ozone treatment, but when the ozone contact time was extended to approximately 300 minutes, the COD removal increased to 46.2%. These comparisons between studies demonstrate that higher ozone dosages can improve COD removal efficiency.

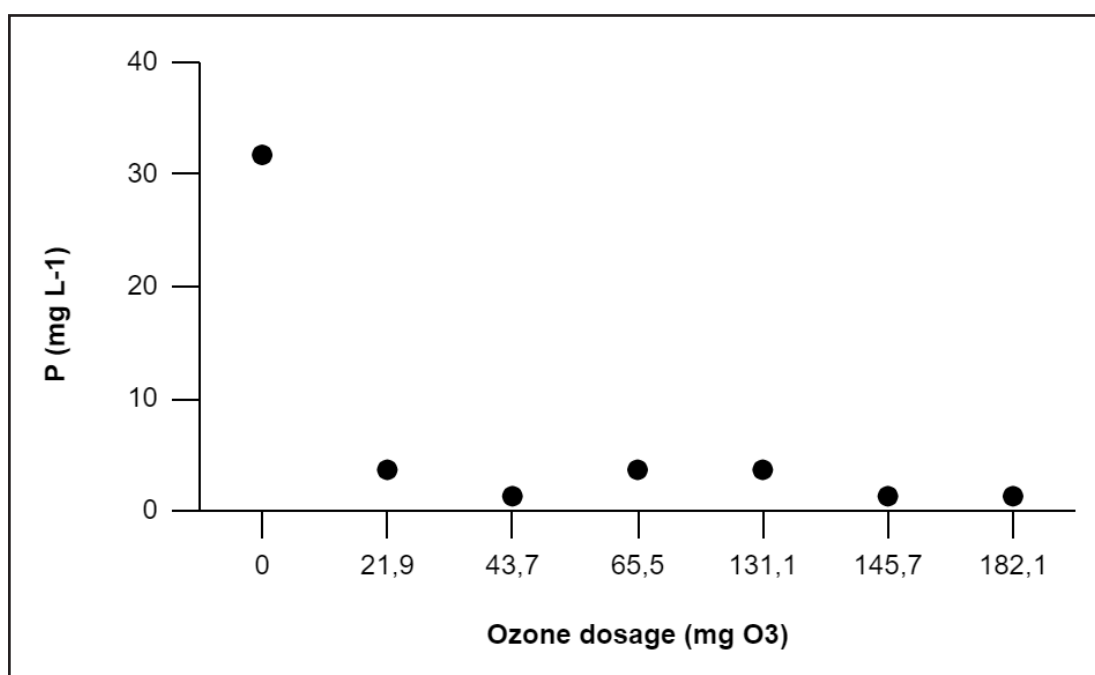
In Figure 4(c), the increase in leachate biodegradability at higher dosages is evident. The BOD₅/COD ratio shows a progressive increase throughout the process, reaching a value of 0.2 at ozone dosages above 140 mg O₃. The increase in the BOD₅/COD ratio indicates that ozone application enhances the biodegradability of landfill leachate. Studies such as Webler's (2018) also observed this increase in biodegradability. The ratio increased from 0.32 to an average of 0.49 after treating the raw effluent with different ozone concentrations.

3.4 Effect of ozone dosages on phosphorus concentration

Figure 5 presents the behavior of phosphorus concentration when applying different ozone dosages. With the first ozone dosage, a sharp decrease in phosphorus

concentration in the leachate can be observed, from 31.7 mg L⁻¹ to 3.7 mg L⁻¹, corresponding to a removal efficiency of 88%. It is interesting to note the speed of the phosphorus removal process through ozonation, as at 60 minutes with an ozone dosage of 43 mg O₃ h⁻¹ (or 174.8 mg O₃ L⁻¹), the phosphorus concentration was reduced to 1.3 mg per liter of leachate corresponding to a maximum removal efficiency of 96%.

Figure 5 – Phosphorus concentration as a function of applied O₃ dosages



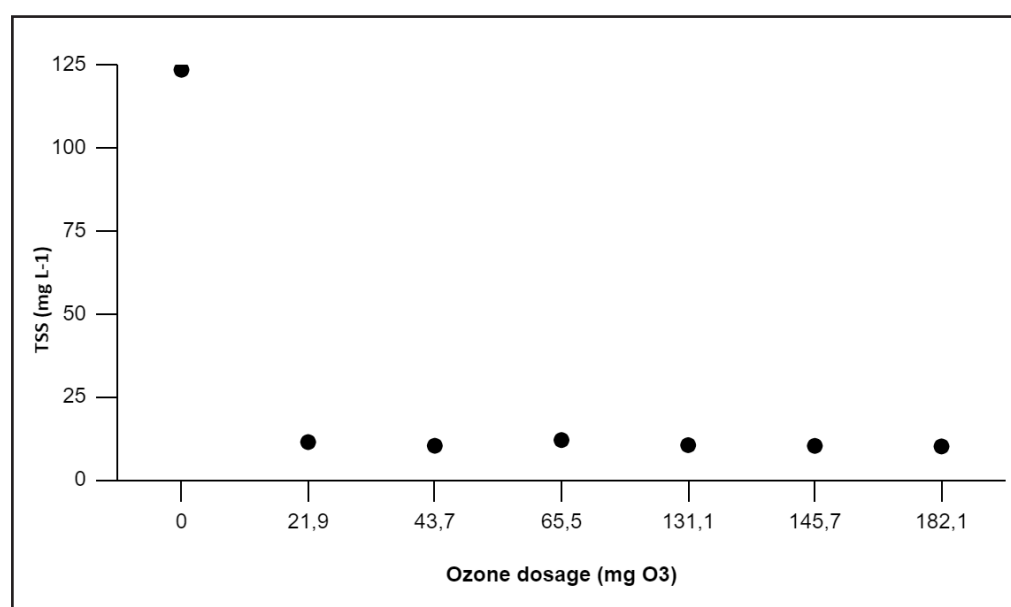
Source: Authorship (2023)

After the ozonation tests, the formation of a white-colored precipitate was observed. It is believed that this could be the formation of struvite (NH₄MgPO₄ • 6H₂O). This fact may explain the high phosphorus removal efficiency through the breakdown of organic matter molecules. Several studies mention the removal of phosphorus, nitrogen, and other nutrients through the chemical precipitation of struvite. For example, Xavier et al. (2014) evaluated the effective recovery of phosphorus from the raw supernatant of anaerobic digestion of sewage sludge. Struvite precipitation was achieved by adding Mg(OH)₂ or MgCl₂ as sources of magnesium at pH values of 8, 9, and 10. As a result, they achieved 90.6% phosphate removal and 29% ammonium removal.

3.5 Effect of ozone dosages on total suspended solids (TSS) concentration

Figure 6 presents the total suspended solids concentration at different ozone dosages during the ozonation process. A reduction in TSS concentration can be observed with an ozone dosage of 21.9 mg O₃ h⁻¹ (or 87.6 mg O₃ L⁻¹). This reduction was from 123.5 mg L⁻¹ to 11.4 mg L⁻¹ in 30 minutes of treatment, corresponding to an efficiency of approximately 88%. It should be noted that the concentrations remained below 12 mg of solids per liter of leachate throughout the process.

Figure 6 – TSS concentration as a function of applied O₃ dosages



Source: Authorship (2023)

The notable results found in this study can be observed when comparing them to the study by Scandelai (2018), who combined ozone treatment with supercritical water, applying 1.0 g O₃ in 30 minutes and subsequently subjecting the effluent to 600°C and a pressure of 23 MPa. The TSS value in the raw effluent was reduced from 158 mg L⁻¹ to 10 mg L⁻¹, resulting in removal efficiency of 93.7%. In this context, it is evident that ozone treatment is highly efficient for landfill leachate treatment and can be combined with other techniques to enhance its efficiency further.

3.6 Effect of ozone dosages on leachate phytotoxicity

Cucumber seeds (*Cucumis sativus*) and lettuce seeds (*Lactuca sativa*) were used as bioindicators to evaluate the effect of different ozone dosages on leachate treatment. The plates containing cucumber seeds in the control group (distilled water) showed a germination index of 100%. Meanwhile, the plates containing lettuce seeds in the control group showed 83.3% germination. However, no root growth was observed in the plates with seeds subjected to tests with raw leachate and leachate treated at different dosages. Therefore, it can be inferred that the ozone process was inefficient in reducing the effluent's toxicity at the dosages applied in this study.

Poblete et al. (2019) used lettuce as a bioindicator in leachate samples with concentrations of 100% and dilutions (30%, 10%, 3%, and 1%) of ozonized effluent and found that germination occurred only in samples diluted below 10%. The non-germination of seeds at high concentrations may be associated with the presence of heavy metals that negatively affect seed germination. Additionally, sufficient dilution of the effluent with water may benefit plant germination. This study helps to understand that leachate concentration is a determining factor for seed germination and opens opportunities for future research. Another possibility would be to increase the ozone dosage applied to break the leachate's toxicity through increased ozone contact.

4 CONCLUSIONS

The physicochemical characterization of the raw leachate indicated that it is a mature effluent with intense pigmentation and high concentrations of phosphorus and solids. After applying ozonation, changes in these leachate characteristics were observed, such as the loss of pigmentation, a drastic reduction in phosphorus and solids concentrations, and an increase in biodegradability. The fact that the pH remained within the basic range allows for characterizing the leachate as mature. It suggests that the basic pH favored the indirect pathway for the decomposition of molecular ozone through the

hydroxyl radical. High values of true color (75%), phosphorus (96%), and total suspended solids (90%) removal from the leachate were observed within the first 60 minutes of ozonation. This period corresponded to applying a dosage of 174.8 mg O₃ L⁻¹. These results demonstrate that even with low ozone concentrations, highly satisfactory results can be achieved in the ozonation of landfill leachate.

Regarding the organic load and toxicity of the leachate, it was found that higher doses than 730 mg O₃ L⁻¹ of leachate are necessary to degrade all organic matter. The reduction in chemical oxygen demand (COD) was 45% at this dosage. The increase in biochemical oxygen demand (BOD) throughout the process may indicate the transformation of refractory compounds into smaller and more biodegradable products. This information is reinforced by the increase in the BOD₅/COD ratio, which was 0.08 for the raw leachate and 0.20 for the ozonated leachate. This confirms the increase in the biodegradability of the leachate during the ozonation treatment. Furthermore, the absence of root growth in cucumber and lettuce seeds demonstrated that the applied doses were insufficient to reduce the toxicity of landfill leachate. Based on the findings, it can be inferred that ozonation is a promising alternative for leachate treatment, and its biodegradability can be enhanced through biological treatment, which is already commonly employed in landfills.

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