Ozonation as a pre-treatment of landfill leachate

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ABSTRACT

Ozone is an oxidizing agent with a potential for removing complex molecules, including those of difficult microbiological decomposition, such as the molecules found in landfill leachates. This effluent presents high organic content, including recalcitrant molecules. Therefore, this study evaluated the efficiency of ozonation in the treatment of raw leachate at the Primavera landfill, located in Mato Grosso, Brazil. The experiments were carried out using the batch system. The leachate pH value was set at 7 and 10, and the contact times between the gas and the leachate were 20, 40, 60, 80, and 100 minutes. Throughout the study, we analyzed color, turbidity, pH value, chemical oxygen demand (COD), and ultraviolet absorbance at 254 nm (UV abs) of the leachate, both before and after ozonation. Results show that ozonation presented high removal of color, COD and UV abs when pH was 7. In contrast, turbidity removal was higher when the pH value was 10. Regarding contact time, we observed a high removal of color (between 80% and 90%) and UV abs (between 60% and 70%) at 40 minutes, depending on the pH value. Removal was also high for turbidity (approximately 75%) starting at 20 minutes with the pH value at 10, and at 70 minutes with pH at 7. The removal relation within all analyzed parameters (due to ozone consumption) decreased throughout the reaction time. Thus, we concluded that ozonation as a pre-treatment of leachate is indeed satisfactory because of its great capacity for organic material removal.

Keywords: degradation, leachate, ozone.

Ozonização como pré-tratamento de lixiviado de aterro sanitário

RESUMO

O ozônio é um agente oxidante com potencial para remoção de moléculas complexas, de difícil decomposição microbiológica, como as moléculas presentes em lixiviados. Este efluente apresenta alta carga orgânica, sendo parte desta recalcitrante. Assim, neste trabalho avaliou-se a eficiência da ozonização no tratamento do lixiviado bruto do aterro sanitário de Primavera, localizado no município de Sorriso – MT. Os experimentos foram realizados em batelada, com o valor de pH do lixiviado ajustado em 7 e 10, e tempos de contato entre o gás e o lixiviado iguais a 20, 40, 60, 80 e 100 min. Foram analisados cor, turbidez, pH, DQO e absorbância ultravioleta a 254 nm (abs UV) do efluente antes e após a ozonização. Os resultados indicaram

que a ozonização apresentou maiores remoções de cor, DQO e abs UV em pH 7, ao passo que a remoção de turbidez foi maior em pH 10. Em relação ao tempo de contato, foram observadas altas remoções de cor (entre 80 e 90%) e abs UV (entre 60 e 70%) em 40 minutos, dependendo do valor de pH; e turbidez (aproximadamente 75%) a partir do tempo 20 minutos, em valor de pH 10, e em 70 minutos, em pH 7. A relação de remoção entre todos os parâmetros analisados em função do consumo de ozônio foi decrescente ao longo do tempo de reação. Por fim, conclui-se que o emprego da ozonização como pré-tratamento de lixiviado é satisfatório, pois apresenta boa remoção de material orgânico.

Palavras-chave: chorume, degradação, ozônio.

1. INTRODUCTION

Population growth results in a disorderly growth of cities, mainly in large urban areas, which causes serious problems, especially in regard to the generation of urban waste and its final destination. Concern for residue disposal is studied both in a national (Agostinho *et al*., 2013) and an international scale (Lombardi *et al*., 2017), due to the awareness surrounding environmental protection.

The main worry regarding the release of residues is the liquids produced by the landfill, commonly known as leachate. Landfill leachate has high concentrations of organic matter, chlorides, ammoniacal nitrogen, as well as organic compounds of difficult degradation, such as humic substances and metals, depending on the properties of the residues disposed of in the landfill (Renou *et al*., 2008; Kawahigashi *et al*., 2014). If discarded inadequately, leachate can cause serious environmental harm.

When one considers efficient techniques for the decomposition of complex compounds, the ozonation technique stands out, either alone or associated with other processes (advanced oxidation processes/AOPs). Thus, it was the technique of choice in this study, and it appears to yield great results in the degradation of recalcitrant molecules (Araújo *et al*., 2016), as well as in the oxidation and mineralization of organic and inorganic species (Dezotti, 2008). The mentioned technique also showed high efficiency in the reduction of biochemical and chemical oxygen demands (BOD and COD, respectively) (Carvalho *et al*., 2018; Gomes and Schoenell, 2018; Brito and Silva, 2012) and turbidity (Mondardo *et al*., 2006).

The treatment process of landfill leachates through ozonation takes place by exposing the effluent to ozone gas. This gas is commonly generated by the Corona effect, which consists of passing atmospheric air between two electrodes. These two electrodes, due to their potential difference, will generate an electric discharge that breaks down the O_2, resulting in the formation of atomic oxygen, which will bond to another O_2 molecule and form ozone (Balakrishnan *et al*., 2002; Kunz *et al*., 1999; Almeida *et al*., 2004).

Ozone reacts with various organic compounds, chiefly amines and aromatic compounds rich in electrons, such as phenols and alkoxylated benzenes, which results in •OH in parallel reactions. Therefore, •OH is always formed when potable water and residual water are treated with ozone (von Sonntag and von Gunten, 2012).

Thereby, the key of the ozone treatment is based on the O_3 direct reaction or on its indirect form, by generating the OH radical. Ozone, due to its elevated standard reduction potential ($E^0 = 2.08$ V) reacts slowly and has high selectivity in regards to a great number of organic compounds (Almeida *et al*., 2004; Geppert, 2018). However, OH radicals also present a high standard reduction potential ($E^0 = 2.80$ V) and react quickly and without a selection for the majority of the compounds in an aqueous solution (Metcalf and Eddy, 2015; Li *et al*., 2018).

The most studied components that influence the ozonation treatment are ozone dosage (Jung *et al*., 2017), exposure time to the gas (Camilo Júnior *et al*., 2019), pH value (Buffle *et*

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al., 2006), effluent characteristics (Silva and Jardim, 2006; Xu *et al*., 2002), among others (Galdeano *et al*., 2018).

This paper therefore evaluated the reduction of the initial values of color, turbidity, chemical oxygen demand, UV abs and pH variance of the raw leachate sampled from a landfill in Sorriso, MT, by considering the influence of pH value and contact time with ozone.

2. MATERIALS AND METHODS

The raw leachate was sampled at a landfill in Primavera, in the city of Sorriso, Mato Grosso, Brazil. The facility has been in operation since 2011, and receives solid residues from Class IIA, which is non hazardous and non inert, and Class IIB, which is non hazardous and inert. It services more than twelve cities and processes twelve thousand tonnes of residue each month.

In order to treat landfill leachate, there is a system that combines biological and coagulation and flocculation processes (a waste stabilization pond and a physical-chemical treatment). Apart from being licensed by the regulating agencies in charge, the landfill possesses a system for monitoring physical and biotic factors.

The experiments were carried out in the Water and Wastewater Laboratory (LAR) at the Federal University of Mato Grosso (UFMT), in Sinop, Mato Grosso.

The elements that composed the ozonation system were an ozone generator; a bubble column reactor with a sparger on its base to defuse ozone into the landfill leachate; a series of three one-liter beakers, where the first one is responsible for reception and storage of the foam, an event that takes place in the reactor, and the other two are responsible for capturing the exceeding ozone (Figure 1). The seizing of ozone was accomplished by using a potassium iodide solution (KI), and its concentration was 20 g L^{-1}. Silicone hoses were also used to connect the beakers.

The ozone generator deployed was the model that uses the Corona effect with atmospheric air, and its nominal production capacity was 0.4 g O$_3$ h^{-1}.

The experimental tests were performed following the methodology cited by Guimarães *et al*. (2010).
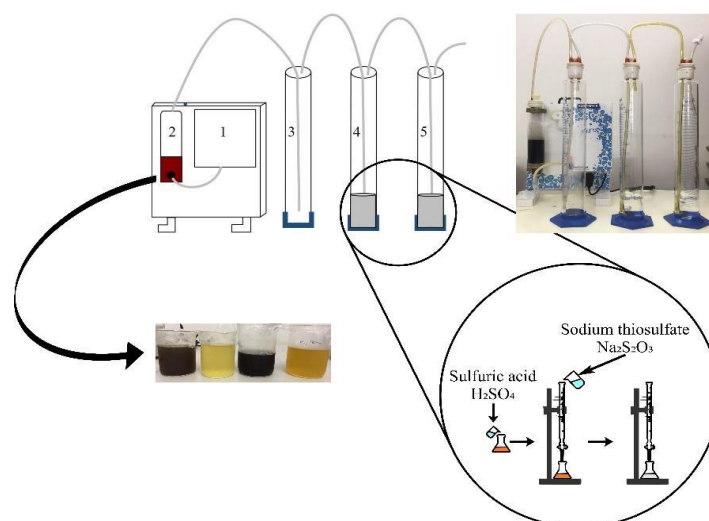

Figure 1. Flowchart of the landfill leachate treatment by ozone [an ozone generator (1); a bubble column reactor to defuse the gas into the leachate (2); a beaker to retain foam (3); and beakers to capture ozone (4 and 5) in a potassium iodide solution].

Samples of distilled water (reagent blank) or of landfill leachate had their pH values set at 7 (or adjusted to 7.3 for water) and 10. Portions (0.2 L) of water or effluents were added into the reactor. We added 0.2 L of potassium iodide (KI), its concentration set at 20 g L-1, into the beakers used to capture ozone. The reactor and beakers were then sealed, and the ozonation tests were initiated using the predetermined times of 20, 40, 60, 80 and 100 minutes

After each ozonation time, samples of water or landfill leachate were removed from the reactor, and, for the landfill leachate, we carried out the determination of pH value, color, turbidity, UV absorbance at 254 nm (UV abs), and chemical oxygen demand (COD). Afterwards, the values of pH, color, turbidity, and UV abs were obtained using the following pieces of equipment: a pH meter, a colorimeter, a turbidimeter, and a spectrophotometer, respectively. COD was determined through the heated chemical digestion method.

Then, after the tests, the potassium iodide on the beakers was used to quantify the exceeding ozone (non reactive) through an iodometric titration.

The consumed ozone was determined by the difference between the residual gas contained in the distilled water (reagent blank) and in the effluent.

The characterization of the landfill leachate in regard to the initial parameters is informed in Table 3. We verified that the effluent showed high values of color, turbidity, COD and humic substances (expressed in terms of UV abs), which indicates that the landfill leachate is heavy in pollutants.

The experimental design was entirely random in a 2×5 factorial design, with two pH values and five exposure times, resulting in ten samples with three repetitions. The analysis of variance was used, and, when we identified significant effects, we employed Tuckey's test in relation to pH value, and regression testing in relation to contact time.

3. RESULTS AND DISCUSSION

The results from the statistical analysis have shown that there was a significant effect ($p \leq 0.05$) from the initial pH value over the pH and COD parameters. Also, the interaction among the treatments showed a significant effect ($p \leq 0.05$) for the removal of UV abs, color and turbidity.

As for the pH at 7, we observed an increase in the pH value from the beginning of the experiment to its end (a negative variation). When the initial pH was 7, it is believed that a few intermediate decomposition reactions release OH^- ions, which causes the pH to increase. For the pH at 10, we observed a decrease in the pH value from the beginning of the experiment to its end (a positive variation) (Table 1), which, despite being low, indicates the consumption of OH^- ions, probably due to its reaction with ozone chain reactions (von Gunten, 2003).

The average value for the COD parameter, as a function of the initial pH value (Table 1), showed that a higher removal rate was obtained when pH was 7. In neutral pH values there is, basically, ozone reacting with organic matter. When the pH value is elevated, there is a tendency to create a hydroxyl radical (a higher standard reduction potential in relation to the molecular ozone), but there is also a tendency for its elimination (Buffle *et al*., 2006), reducing its availability for indirect reaction.

Table 1. Effect of the pH treatment regarding the variation of the parameters: pH value and COD.

pH value	Variation of pH (%)*	COD removal (%)
7	-14.5	31.9
10	1.7	17.7

* Negative values point out an addition in pH variation, and positive values point out a decrease in pH variation, from the beginning to the end of the experiment.

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For pH values lower than 7.5, the decomposition via direct oxidation (O_3) is highly efficient. For pH values higher than 7.5, the presence of hydroxide ions initiates the decomposition of ozone in radicals, causing an increase in organic decomposition through indirect oxidation (Takashina *et al*., 2018; Usepa, 1999). However, for pH values higher than 9, the ions found in it (normally bicarbonate and carbonate) act as scavengers of •OH, so they limit indirect oxidative action, reducing the oxidation process (Takeuchi *et al*., 1997).

Differently, as described by Jung *et al*. (2017) in their work evaluating saltwater ozonation, the increase of the sample's pH value (7 to 9) did not affect the decay of ozonation concentration, due to its quick reaction with bromide ions. The authors describe that the pH value alteration, however, changed the bromide species, which modified the reactions with ozone without changing its decomposition.

The removal behaviors of color, UV abs and turbidity (in addition to the interaction of pH value and exposure time) are presented in Figure 2. Higher color removals occurred for contact times above 40 min, with a better tendency of removal for the effluent in pH value set at 7 (Figure 2A).
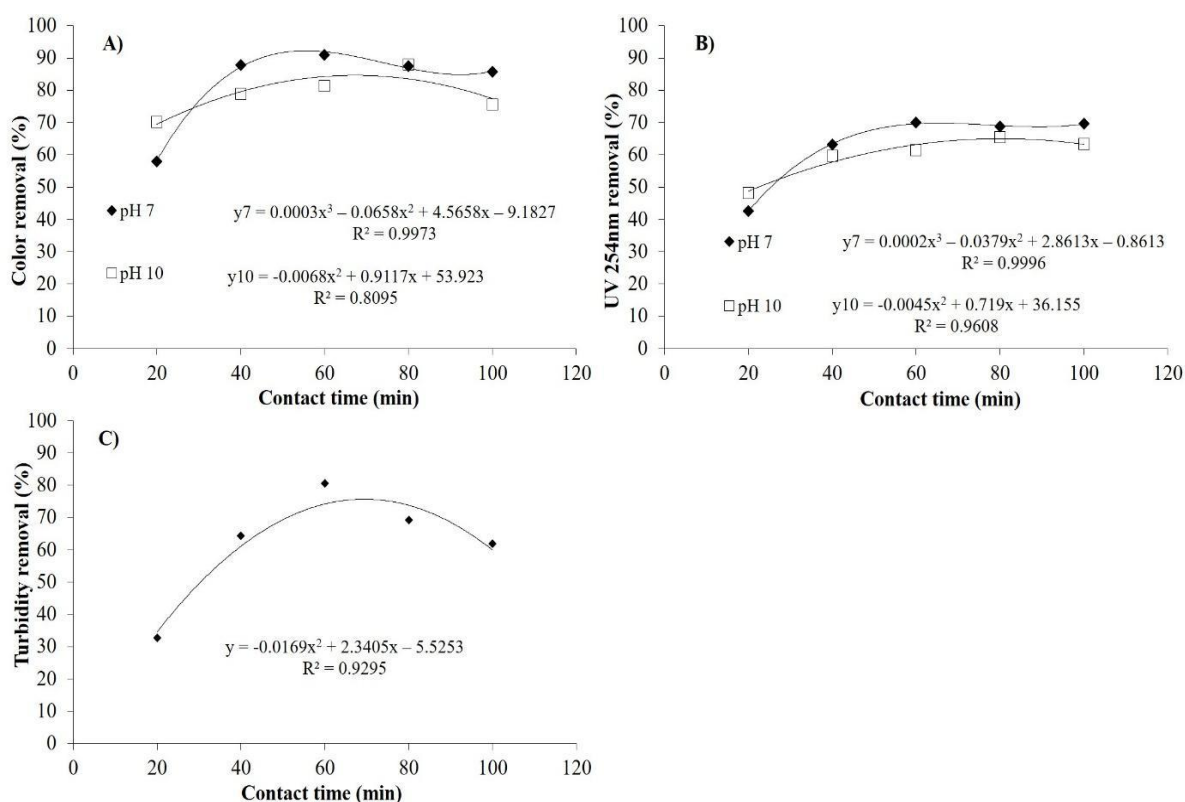

Figure 2. A) Effect of the contact time and the landfill leachate's initial pH value concerning color removal. B) Effect of the contact time and the leachate's initial pH value concerning UV_{254nm} removal. C) Effect of the contact time and the leachate's initial pH value set at 7 concerning turbidity.

For the UV absorbance parameter, the results were the same as for color removal. However, this parameter showed lower average values of removal (see Figure 2B), which shows that there is a correlation between the parameters. The elements of colors and UV abs are linked to the presence of chromophoric substances in the liquid solution, thus removals or modifications in the chromophoric molecules cause interferences which can be noticed in the visible spectrum (color) and in ultraviolet (UV abs). See Figures 2A and 2B.

Due to the ozonation process applied to the leachate, the sample's initial dark color tends to present a light-yellow color. This reduction of color happens because of the ozone's or the

OH radical's attack on the carbon double bond in the chromophore. The procedure results in the formation of the so-called "bleached" products, such as aliphatic acids, aldehydes, and ketones, which are by-products of low-molecular weight, normally biodegradable (Ntampou *et al*., 2006).

The ultraviolet radiation at 254 nm is easily absorbed by many organic, inorganic and colloidal compounds that integrate the effluents, which means it is associated with the presence of unsaturated and aromatic compounds (chromophores) in the liquid solution. Therefore, the reduction of UV abs, as well as color removal, is observed through the oxidation of such aromatic compounds (Cortez *et al*., 2010; Oloibiri *et al*., 2015).

Results have shown that, both for color (Figure 2A) and UV abs (Figure 2B), there is a tendency for higher removal rates when the timer is set for approximately 60 minutes when pH value is 7, reaching removal rates higher than 90% and around 70% for color and UV abs, respectively. Regarding the pH values set at 10, the tendency for a higher removal occurred at approximately around 70 and 80 minutes. We also observed average removals of 80% for color, and 60% for UV abs.

However, we observed that the removal rates for time spans shorter than 70-80 minutes are close to the values of maximum removal. In these cases, it becomes interesting to evaluate ozone production costs. It is necessary to observe production feasibility for longer periods of time, so the parameters can be reduced by 10%.

Then we observed that color and UV abs removal have an increasing rate in the initial times, but, as it advances, the oxidation rate from the organic compounds becomes stable. This same behavior was noticed by Cortez *et al*. (2010) regarding COD and UV abs, resulting in the hypothesis that the molecular ozone might have reacted with the unsaturated and aromatic compounds, and thus generated compounds that have a slower reaction and which are resistant to complementary oxidation, such as aliphatic acids and aldehydes.

These compounds produced by oxidation do not absorb the ultraviolet spectrum. However, they contribute to COD, which explains the higher removal of absorbance regarding the chemical oxygen demand. Moreover, the landfill leachate's high quantity of organic compounds (Table 3) limits COD removal, as well as possible concentrations of inorganic carbon, which are responsible for consuming OH radicals, therefore restricting the system's efficiency (Oloibiri *et al*., 2015).

As for turbidity (Figure 2C), the maximum removal occurred, approximately, at 70 min (75%) for the pH at 7. However, we observed that after 40 minutes removals exceeded 60%. Therefore, continuing the ozonation until it reaches 70 minutes may not be a viable alternative. As was suggested for the color and UV abs parameters, it is valid to consider the costs of ozone production.

The turbidity removal when pH was at 10 did not present any significant regression models ($p \leq 0.05$), reaching an average removal of around 74%.

The tendency of reduction for turbidity removal (according to the contact time) can have a correlation with precipitating metals, such as iron and manganese; although they were not quantified, these metals were probably part of the composition of the landfill leachate used in the study. The formation of precipitating particles was observed, mainly in higher ozonation times. The precipitated particles stood at the bottom of the reactor. At the end of the ozonation time, the precipitated particles were collected with the samples, which gave the samples turbidity (see Figure 2C).

Iron and manganese are soluble in their lower oxidation states (Fe^{+2} e Mn^{+2}), but they are relatively insoluble at their higher oxidation states (Fe^{+3} e Mn^{+4}). This is why these cations can precipitate in the forms of iron hydroxide and manganese hydroxide (Apha *et al.*, 1998).

According to von Gunten (2003), the main oxidation for these inorganic compounds is via direct oxidation, using molecular ozone. As a result, it is possible that a partial oxidation of

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these elements via molecular O_3 happened. In addition, the precipitates formed are easily removed using sedimentation and filtration processes after ozonation (Moruzzi and Reali, 2012).

3.1. Ozone consumption

The relation of color, turbidity, UV abs, and COD removal, in function of ozone consumption, has shown significant changes ($p \leq 0.05$) regarding the isolated treatments, but no significant changes were detected regarding the interaction between them. The variation of pH value did not show any significant change for the evaluated variables either.

As for color, UV abs, and COD, the highest relation of removal due to ozone mass took place when pH was 7, independent of contact time. For turbidity, the highest removal occurred when pH was set at 10 (Table 2).

Table 2. Effect of the leachate's initial pH value regarding the removal of the parameters analyzed by milligram of ozone consumed.

pH value	Color (mg Pt-Co/mg O_3)	UV abs (abs/mg O_3)	COD (mg O_2/mg O_3)	Turbidity (NTU/mg O_3)
7	39.333	0.028	4.817	2.133
10	23.933	0.018	1.819	4.000

Considering the contact time regarding the removal of the parameters analyzed (Figure 3), we observed that the highest ozone mass removals consumed for the parameters color, turbidity, UV abs, and COD took place in the shorter reaction times. This behavior reflects the highest concentration of the elements in the beginning of the treatment, which favors their contact with the oxidizing agent. As the contact time increases, the concentration of the parameters reduces and, consequently, there is less use of the ozone that enters the system (Cortez *et al*., 2011; Zhang *et al*., 2018).
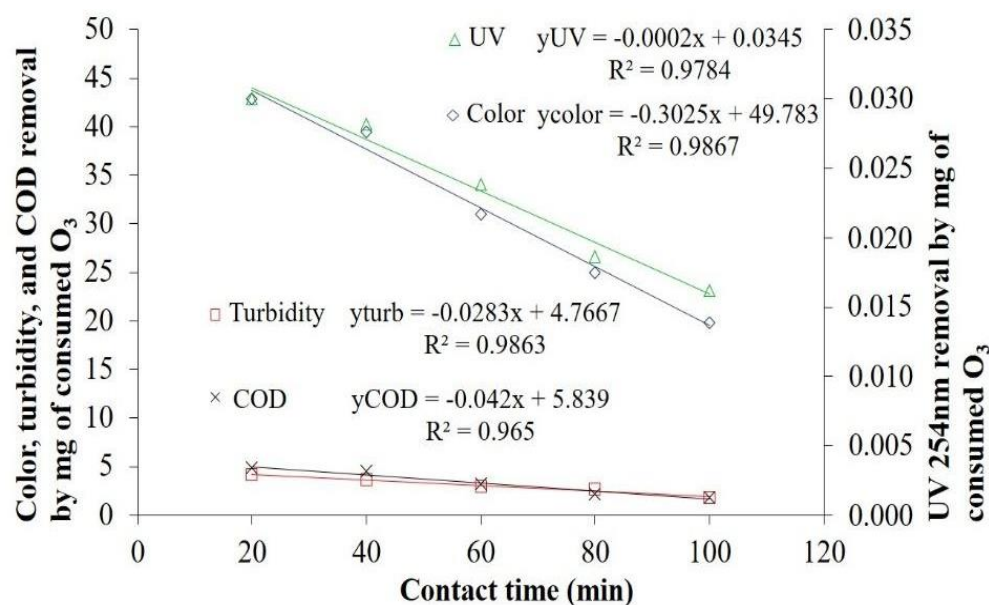

Figure 3. Effect of contact time concerning color, turbidity, UV abs, and COD removals by milligram of ozone consumed.

The residual values of color, turbidity, UV abs, and COD, determined after the treatment (Table 3), show that the landfill leachate presented high values regarding the parameters analyzed.

Table 3. Residual values obtained for the parameters of raw and treated landfill leachate analyzed, in addition to contact time and initial pH value.

Parameters	pH 7		pH 10	
	Raw effluent	Treated effluent	Raw effluent	Treated effluent
20 min				
Color (mg Pt-Co L^{-1})	14940.00	6215.00	11870.00	3512.00
Turbidity (NTU)	1104.00	743.00	1981.00	610.00
pH value	7.44	8.30	9.83	9.73
$UV_{254\,nm}$ (abs)	13.94	7.99	12.35	6.40
COD (mg O_2 L^{-1})	4321.00	3194.00	4158.00	3393.00
40 min				
Color (mg Pt-Co L^{-1})	15020.00	1845.00	13570.00	2858.00
Turbidity (NTU)	1104.00	400.00	2618.00	770.00
pH value	7.35	8.51	9.79	9.70
$UV_{254\,nm}$ (abs)	14.25	5.23	13.48	5.45
COD (mg O_2 L^{-1})	4318.00	2497.00	4045.00	3381.00
60 min				
Color (mg Pt-Co L^{-1})	15100.00	1346.00	13140.00	2478.00
Turbidity (NTU)	1104.00	248.00	2700.00	788.00
pH value	7.54	8.60	9.82	9.67
$UV_{254\,nm}$ (abs)	14.66	4.37	13.36	5.17
COD (mg O_2 L^{-1})	4335.00	2700.00	4042.00	3384.00
80 min				
Color (mg Pt-Co L^{-1})	14826.00	1836.00	11880.00	1427.00
Turbidity (NTU)	1104.00	366.00	2177.00	262.00
pH value	7.52	8.65	9.82	9.61
$UV_{254\,nm}$ (abs)	13.78	4.29	12.44	4.30
COD (mg O_2 L^{-1})	4691.00	3406.00	3868.00	3122.00
100 min				
Color (mg Pt-Co L^{-1})	14826.00	2060.00	10890.00	2656.00
Turbidity (NTU)	1104.00	408.00	2188.00	840.00
pH value	7.52	8.72	9.86	9.57
$UV_{254\,nm}$ (abs)	13.78	4.19	11.95	4.38
COD (mg O_2 L^{-1})	4691.00	3609.00	4370.00	3531.00

Thus, we identified that the values for color, COD and UV abs of the raw effluent were distinct among the pH values. Those with lower values of pH showed the highest values in the parameters. As for the turbidity values, we also observed some difference; however, the highest values were for the pH set at 10. Aziz *et al*. (2007) investigated the effect of the pH value regarding color reaction in the landfill leachate's treatment process; they also verified that its

black color became light-brown, as the effluent's pH value was reduced or increased, due to the chemical precipitation of humic substances. Therefore, the simple act of increasing or reducing the landfill leachate's pH value may have promoted a removal of the parameters analyzed.

It is important to highlight that, although the values obtained after the ozonation are high, the treatment did promote a significant reduction of the parameters studied, which can improve or reduce the landfill leachate's post-treatment time.

The ozone integration using other oxidation processes, such as electrochemical and chemical processes (Araújo *et al*., 2016), are alternatives to achieving the values established by the norm. Also, another interesting alternative is the association between the gas and adsorption processes. Poblete *et al*. (2017) observed good color and COD removals by using ozone and activated charcoal.

Bila *et al*. (2004) have shown that the sequence of coagulation-flocculation treatments + ozonation + biological treatment increases the efficiency of the biological treatment stage. Silva and Daniel (2015), by using ozone followed by chlorine disinfection, obtained good removals of biochemical oxygen demand.

4. CONCLUSION

Throughout the study, regarding the leachate's hydrogen ionic potential, we observed higher color, COD and UV abs removals when we used pH value at 7; and, for turbidity, when we set pH to 10.

When considering contact time, we verified that, in an exposure of 40 minutes, a good removal for color, turbidity and UV absorbance ($UV_{254\ nm}$) takes place.

The relation of removal due to ozone consumption decreases as the contact time increases.

Even though the ozonation process has presented good percentages of removal for the parameters analyzed, it must not be applied as the only stage of treatment, since it was not possible to achieve the quality required by the legislation for releasing it into bodies of water. Therefore, it is fundamental to associate it to the methodology of other treatments.

Some alternatives, such as varying the concentration of gas and associating the advanced oxidation processes (AOP) with other advanced or conventional processes, can be interesting from the point of view that it would be a reduction in the effluent load, which would make its treatment less complex.

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