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Article

Acidification of recalcitrant effluent: Optimization of time and reagent volume

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Abstract

Hydric bodies suffer from the discharge of recalcitrant effluents, such as landfill leachate, which requires advanced treatment, such as photo-Fenton. Though, it requires a acidification step of effluent to adjust it to pH≈3. The objective of this work was to apply sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) in different concentrations in the gross leached, and decide which of one would be better in relation to time optimization and volume of acid spent. Potentiometric curves and trend lines were formulated by the polynomial regression degree 5 and the hypothesis test was used to determine the volumes to be added, to achieve the desired pH (pH 3). The H_2SO_4 (6 N) presented better results in the average time control of 6 min 35 s – while spending 27 min 50 s without the potentiometric curves. From hypothesis test, sulfuric acid achieved the best result when compared to the final, theoretical and real volume used in the acidification of the leachate.

Keywords leached; pH; hypothesis test; potentiometric curve; reagent concentration.

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1 Introduction

Society has intensified over the last decades its consumption pattern, culminating in the giant production of solid waste. As a final disposal, we have sanitary landfill, an adequate and viable site for management of solid urban waste (Santadellaet et al., 2014). In landfills, leachate is generated from the combination of waste moisture, rainwater and decomposition occurring inside the grounding cells. In this effluent compounds with high pollutant potential such as humic substances and heavy metals, high concentrations of ammoniacal nitrogen, chlorides and recalcitrant organic compounds are present (Kawahigashi, 2012; Mannanino, 2013; Santadella et al., 2014; Su et al., 2014; Huda et al., 2017; Bansal, 2019).

The leachate final destination occurs often in water bodies (surface or underground) or in the soil, causing contamination and affecting the biota of these environments, as well as public health because potentially toxic and carcinogenic compounds are present in this efluentesuch as phenols, toluene, benzene, metals, drugs, hormones, pesticides and dioxins (Ribeiro, 2001; Koh et al., 2004; Silva et al., 2004; Rocha, 2010; Zhang et al., 2011; Zhang, 2018).

Therefore, it is necessary to have adequate leachate treatment in order to avoid irreversible damages to environment, especially to aquatic and terrestrial ecosystems, and it is necessary that the leachate's discharge standards are in accordance with CONAMA RESOLUTION 430 of 2011 (Brazil, 2011).

In order to meet the BOD (Biochemical Oxygen Demand) limit, COD (Chemical Oxygen Demand), sedimentable materials, inorganic compounds, pH, among other parameters, there are several processes that aimed at decontaminating of these liquids, either by biological, physical or chemical processes. The Advanced Oxidative Processes (AOP) are considered the most effective pollutants degradation process, especially those of high recalcitrance through the use of species of high oxidant grade and, therefore, they present themselves as an alternative to leachate decontamination (Brazil, 2011; Silva et al., 2013). In the last decades, the AOPs have received prominence in the research and development of effluent treatment technologies, proving efficient in the degradation of pollutants and consequent environmental impacts reduction (Araújo et al., 2016).

Among POAs, we highlight photo-Fenton process that is sensitive to pH which has a great influence on treatment efficiency, whose acceptable range varies among authors; for Singh and Tang (2013) and Deng and Zhao (2015) this range is between pH 2.5 and 4.5, whereas for Clarizia et al. (2017) pH range should be between 2.8 and 3.5. However, despite the advantages offered by photo-Fenton, costs with acids or even bases to adjust to the desired pH due to its restricted range, the viability of the process is countered. However, in literature, a pH value where a great process efficiency occurs to many researchers is at pH 3.0 ± 0.1 (Martins et al., 2011; Klamerth et al., 2013; Silva et al., 2013; Singh and Tang, 2013; Gonçalves et al., 2014; Amor et al., 2015; Paulino et al., 2015; Oliveira et al., 2016).

According to Rocha (2010), in the solar photo-Fenton process, which uses sunlight to increase pollutant removal potential, the leachate acidification step requires a long time to reach the desired pH in order to interfere in duration of the reaction to the sun, as well as in sample loss and reagents, when the ideal pH value has been exceeded. Therefore, the optimization of this step was essential for a greater efficiency in the photo-Fenton process and consequently reagents reduction, often toxics that are released into the environment. In the absence of works that seek to minimize these setbacks, a methodology was elaborated for a later reproduction of this stage.

The leachate used in this study came from a lagoon system, specifically, from the facultative lagoon of the Metropolitan Sanitary Landfill of João Pessoa (ASMJP). Stabilization ponds are widely used in Brazil because of their low cost for biological treatment where organic and inorganic matter from the percolated liquid occurs through bacteria and phytoplankton action (Frascari et al., 2004; Castilhos Junior et al., 2009).

Thus, the objective of this work was to analyze and optimize the acidification step of the ASMJP leachate from the studied acids volume (sulfuric and hydrochloric), as well as its concentrations and time spent in the desired pH adjustment, in order to suit effluent and subsequent application in oxidative processes, in particular to the solar photo-Fenton process.

2 Materials and Methods

2.1 Acids seletion and their concentrations

The acids selection used was initiated with a survey of the application of POA in the treatment of leachate, in particular, in the photo-Fenton process and finally focused on Ferreira (2012) and Silva et al. (2015), which used H_2SO_4 at 36 N and HCl at 18 N, respectively, as well as at Porto (2016), which used H_2SO_4 at 2 N and HCl at 6 N and 18 N.The following concentrations were then used: H_2SO_4 at 2 N, 6 N and 36 N; HCl in the 6 N concentration, according to Porto (2016), and to be justified as an intermediate concentration between the concentrations adopted for H_2SO_4.

2.2 Determination of potentiometric curves

The pH potentiometric curve for each acid and its respective concentration was elaborated in order to analyze the leachate behavior from the ASMJP during the acidification stage.

The 0.1 L leachate volume used by Oliveira (2016) was adopted as reference. This volume was collected through a 100 ± 0.08 mL volumetric pipette and then added to the 250 mL beaker for later acidification.

The first acidification step occurred by incorporating the acid gradually and, with each added volume, the corresponding pH was annotated using the pH meter to define potentiometric curve. Leachate was homogenized during acidification by means of a magnetic stirrer at 105 rpm rotation for 10 minutes. The process was terminated when sample reached the desired pH (pH 3.0 ± 0.1). This procedure was performed in quadruplicate for H_2SO_4 at 6 N concentration and in quintupleta for other concentrations of the acids studied.

Potentiometric curves were constructed and identified through a trend line, which generated a 5rd order polynomial equation. The statistical hypothesis test was performed, a procedure usually used for statistical inferences in which one seeks to determine the value of a given unknown parameter (Naghettiniet al., 2007). From the "Achieve Goal" function of the hypothesis test (Excel, 2010), the 5rd degree polynomial equation was used and a volume was adopted to efficiently reach the desired pH, unknown parameter being the volume spent and the known pH of the sample.

From volumes obtained by the hypothesis test, the second stage of acidification occurred in which the volume of acid added for each desired pH was maintained: pH 8, 7, 6, 5, 4 and 3, with the same leachate sample volume and the same homogenization procedure. Tests were performed in triplicate for each of the desired pH's in which acid was added to the sample, homogenized and then pH readout. Finally, pH average obtained was calculated in order to verify if the pH reached in acidification approached the desired pH. Thus, a new acidification curve was obtained for each acid in its respective concentration.

2.3 Acidification time control

An acidification test of the ASMJP leachate (pH 8.44 ± 0.23) was performed to compare time and acid volume with 5 different operators and to know how long it would take to reach the desired pH (3.0 ± 0.1). Each operator was instructed to acidify the leach sample with HCl or H_2SO_4, both at 6 N concentration, simultaneously with homogenization on a magnetic stirrer. Finally, they should note the final volume of acid used and the total time they took to acidify the sample to the desired pH (3.0 ± 0.1).

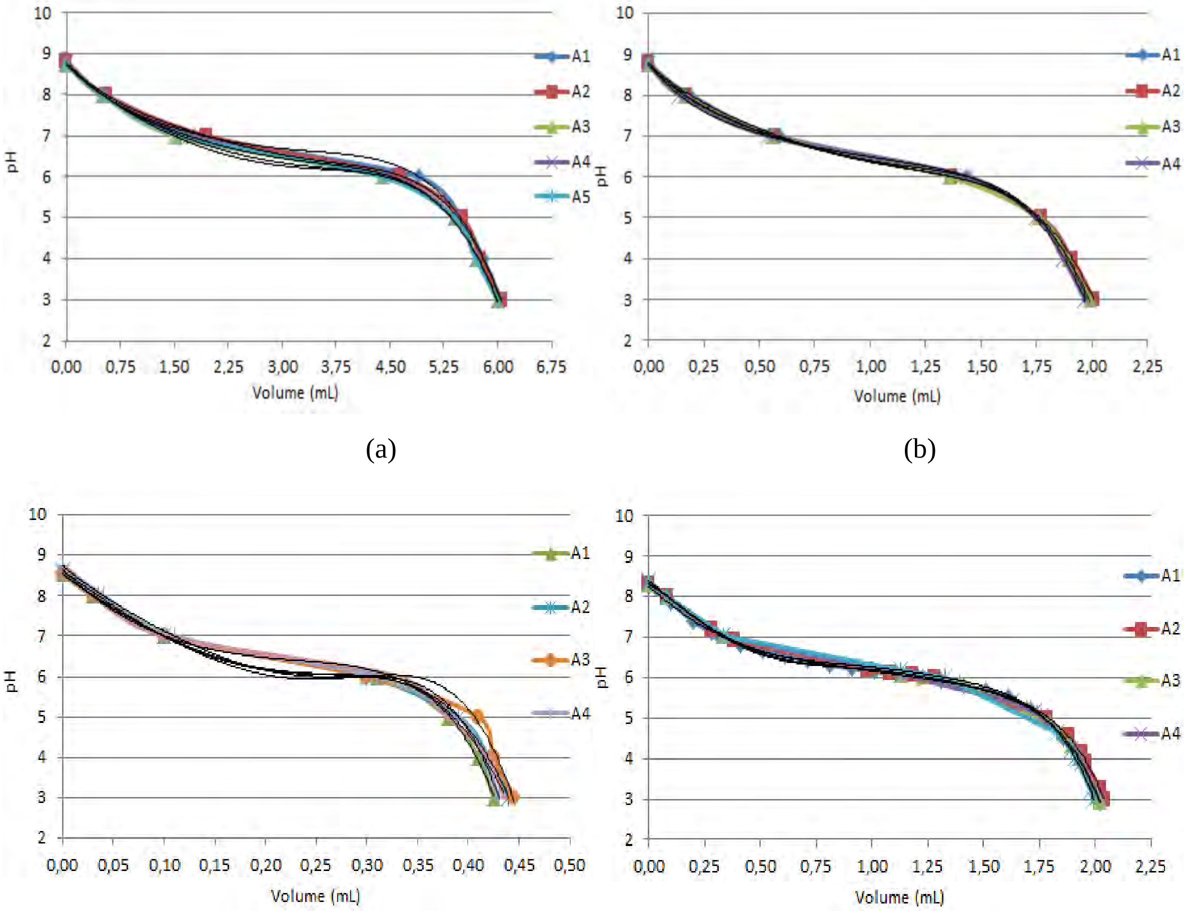
3 Results and Discution

3.1 Potenciometric curves and acid volume spent

In the formulation step of pontentiometric curves, it was possible to notice a similarity between them (Fig. 1) and verified the buffering effect, water capacity to maintain its stable pH as acids or bases are incorporated into the system (APHA, 2005). This effect was observed in the analyzed leachates from a resistance to pH change in acid medium, which may be related to its complex composition. This effect is evident in the passage from pH 7 to pH 6, since it required a greater amount of acid for this transition, proving the existence of buffer effect.

According to Ferreira (2010), the leachate buffer condition is controlled by the presence of volatile acids and it indicates that the organic matter transformation in the waste landfill cells is found in the methanogenic fermentation phase with precipitation occurrence and complexation of metals and increased gas production.

The most noticeable difference between the potentiometric curves generated consisted in the final volume of the samples to acidify them until pH 3, which was due to the different concentrations of the acids used.



(a) (b)

(c) (d)

Fig. 1 (a) Potentiometric curve for H_2SO_4 (2 N) with 100 mL crude leachate sample (initial pH: A = 8.75, A2 = 8.81, A3 = 8.75, A4 = 8.76, A5 = 8.75); (b) potentiometric curve for H_2SO_4 (6 N) with 100 mL crude leachate sample (initial pH: A1 = 8.75, A2 = 8.81, A3 = 8.75, A4 = 8.76); (c) potentiometric curve for H_2SO_4 (36 N) with 100 mL crude leachate sample (initial pH: A1 = 8.53, A2 = 8.62, A3 = 8.55, A4 = 8.74, A5 = 8.64); (d) potentiometric curve for HCl (6 N) with 100 mL crude leachate (initial pH: A1 = 8.23, A2 = 8.30, A3 = 8.32, A4 = 8.37, A5 = 8.38).

Figs 1b and 1d, which show H_2SO_4 (6 N) and HCl (6 N) respectively, show that the volume spent to acidify leachate to the desired pH (pH 3) was practically the same, using 2 ± 0.02 mL acid. These values were satisfactory because they represent 2% volume of the leachate sample used, which would have good efficiency in real scale. It is also possible to realize that the Figs 1B and 1D trend lines presented a little more consistency than those from Figures 1A and 1C, which were further distanced from each other.

By analyzing step 1 individually, we see the difference in the amount of acid used to achieve the same pH due to the variation of acid concentration; while 2 N sulfuric acid (Fig. 1a) required around 6.02 mL to acidify the sample. Concentrated sulfuric acid (Fig. 1c) required an average of 0.44 mL to achieve the same pH. In relation to the volume used, the H_2SO_4 at 36 N would be a good alternative in terms of total volume however, it is necessary to analyze more precisely its stability in the next step.

Trend lines were close to the potentiometric curves (Fig. 1) with the determination coefficient (R^2) from each function near or equal to 1 (Eq. 1 to 4, referring to H_2SO_4 to 6 N). This confirms the similarity of the behavior of the potentiometric curves to the 5rd order polynomial equation.

$$y = -1.1619x^5 + 4.173x^4 - 5.9321x^3 + 5.3479x^2 - 4.7731x + 8.7498;\ R^2 = 1 \qquad (1)$$

$$y = -1.312x^5 + 5.5041x^4 - 9.3154x^3 + 8.673x^2 - 5.9755x + 8.8089;\ R^2 = 0.999 \qquad (2)$$

$$y = -1.4058x^5 + 5.7528x^4 - 9.3988x^3 + 8.4512x^2 - 5.7855x + 8.7481\ ;\ R^2 = 0.999 \qquad (3)$$

$$y = -1.4421x^5 + 6.1846x^4 - 11.083x^3 + 10.765x^2 - 6.6939x + 8{,}758\ \ ; R^2 = 0.9997 \qquad (4)$$

Table 1 shows the mean volumes and their respective standard deviations obtained from the hypothesis test using equations 1, 2 and 4, as well as their respective standard deviations from each acid investigated. Equation 3 for H_2SO_4 at 6 N was omitted from the calculations because the volumes calculated by the Hypothesis Test differed too much from the others.

Table 1 Volumes of theoretical acids obtained from the hypothesis test (Acid: mL±DP)

Desired pH	**H_2SO_4 – 2 N**	**H_2SO_4 – 6 N**	**H_2SO_4 – 36 N**	**HCl – 6 N**
pH 8	0.50 ± 0.02	0.17 ± 0.02	0.031 ± 0.005	0.08 ± 0.004
pH 7	1.69 ± 0.19	0.57 ± 0.03	0.100 ± 0.004	0.34 ± 0.01
pH 6	4.59 ± 0.14	1.42 ± 0.04	0.306 ± 0.019	1.25 ± 0.06
pH 5	5.33 ± 0.08	1.76 ± 0.01	0.390 ± 0.009	1.77 ± 0.02
pH 4	5.71 ± 0.08	1.89 ± 0.02	0.416 ± 0.008	1.92 ± 0.02
pH 3	5.97 ± 0.07	1.99 ± 0.02	0.437 ± 0.010	2.01 ± 0.02

SD: Standard deviation.

After performing step 1 to obtain potentiometric curves and theoretical volumes (hypothesis test), acidification tests were performed (step 2) according to figures 2 and 3, using the same acids and their respective concentrations based on the theoretical volumes. In these figures, the X axis represents the added volume and the Y axis expresses the expected pH, as well as the points indicating the expected pH of the average value from pH's obtained.

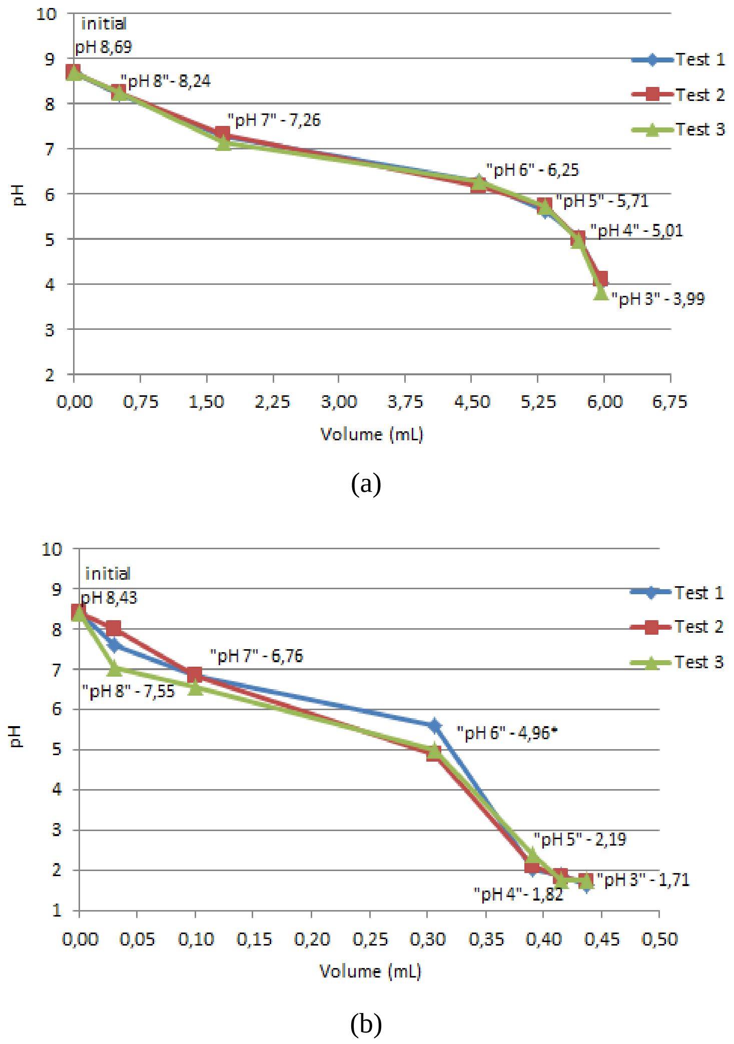
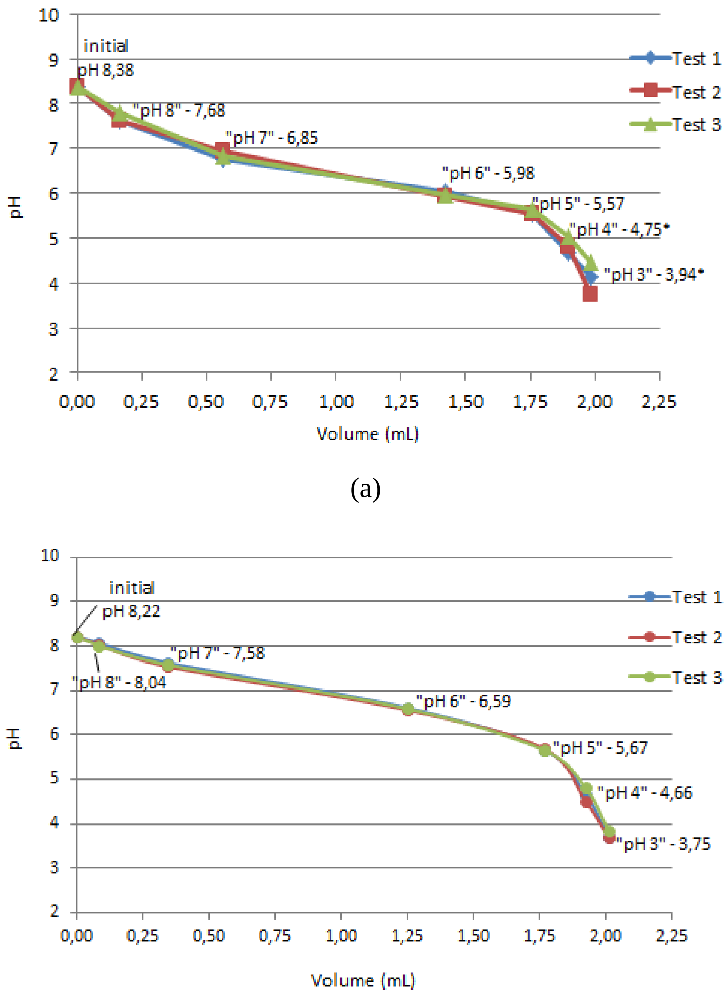


Fig. 2 Acidification curve (a) for H_2SO_4 (2 N) with 100 mL crude leachate sample and (b) acidification curve for H_2SO_4 (36 N) with 100 mL crude leachate sample.

In relation to the pH obtained experimentally in the second stage of acidification, it is possible to see that in Fig. 2a, results were initially similar. However from pH 5 on, the value reached began to distance itself from the desired pH, coinciding with the curve range in which the buffering effect is broken. It is noteworthy that even with these leachate pH variations, it was possible to reach pH 3.99 by the average of the three tests performed, which represents an acceptable and feasible result for laboratory use aiming at reagent saving and above all, acidification time optimization and effluents waste because when desired pH is not reached, it is discarded in a random way, which may also be another pollution source of natural resources.

Fig. 2b shows a behavioral abnormality from the beginning of acidification where the pH 8 values varied considerably from one another and did not have the behavior expected by the test. Other points to be highlighted were the expected results from pH 4, in which there was a sudden fall from this one and pH 3. A possible reason for this was the fact that acid was well concentrated and, consequently, volumes added were very close between (Table 1), making the addition of a drop of acid in addition to the expected, present a significant difference in the result in question.



(a)

(b)

Fig. 3 Acidification curve (a) for H_2SO_4 (6 N) with 100 mL crude leachate sample and (b) acidification curve for HCl (6 N) with 100 mL crude leachate sample.

The acidification curve with 6N sulfuric acid (Fig. 3a) presented approximate results at the pre-established pH's up to pH 6, similar to that observed in Fig. 2. In the following points, curve behaved with small variations that can be explained by the leachate complexity, however presented better final results than those presented in the graph of Fig. 2a with the advantage that the final volume was reduced by 3 times for H_2SO_4 (6 N), compared to H_2SO_4 (2 N).

For the hydrochloric acid at 6 N (Fig. 3b), the beginning of the acidification had pH values of 7, 6 and 5 more distant than the ones from figure 3Aa however from pH 4 on, values obtained experimentally approximated what expected, showing better results regarding sulfuric acid at 6 N, especially when the pH 3 value was analyzed.

When considering the proximity in which the volumes of the added acids reached the desired pH's (8, 7, 6, 5, 4 and 3), it was observed that the 6N sulfuric and hydrochloric acid curves with 3.94 final pH (Fig. 3a) and 3.75 (Fig. 3b), respectively obtained the most satisfactory results in relation to those found in the graphs from Fig.l 2, so they were the concentrations used for the acidification time control step.

3.2 Time control of acidification

For the determination of the volumes to be added, independent term (sample initial pH) from the leachate trend lines equations collected in the first stage of the study was changed. The hypothesis test was performed again in order to find a new volume of H_2SO_4 and HCl both at 6 N to acidify the new samples at pH 3.0 ± 0.1 (Table 2).

Analyzing first the acidification carried out by "Researchers", leachate sample when HCl (6 N) was added reached 2.99 final pH, using 2.04 mL of said acid.The first acidification performed by it did not reach the desired pH, However, the 2.79 final pH is considered satisfactory for some authors, who state that 2.8 pH is the most efficient for the photo-Fenton process (Carra et al., 2014, Manenti, 2014, Romero et al., 2016). As this study sought greater precision at the desired pH, it was decided to repeat acidification with HCl until it reached pH 3.0 ± 0.1, which totaled 8 minutes and 29 seconds.

For the test with H_2SO_4 (6 N), still carried out by "Researchers", in the first attempt at acidification, pH exceeded the value 3.0 ± 0.1. This may be due to operational errors or lack of initial homogenization of the sample. In the second experiment, the initial volume added was reduced to pH 3.33, and with two additions of the acid, pH 3.07 was reached with 1.83 mL final volume and 6 minutes and 35 seconds total time (Table 2).

Later, acidification carried out by the "Operators" who were unaware of the volumes obtained from the use of potentiometric curves was analyzed and an average of 24 min and 26 s was spent on hydrochloric acid (6N) using one or more samples until reach the desired pH.

In relation to the sulfuric acid behavior, the average time spent by "Operators" was on average 29min07s for acidification of the leachate. It was observed that "Operator 4" required a considerably longer time than the others at this stage for acidification of an effluent in order to adapt it, for example to the photo-Fenton process (Table 2).

It is worth mentioting that in the literature there are few studies that relate the importance of the time spent in the acidification stage to the efficiency of advanced oxidative processes. In addition, when the effluent does not have the ideal pH to the process, they are discarded in the environment without any treatment.

Table 2 Analysis of time in the acidification stage.

Acid used	Operator	Initial pH	Final pH	Volume (ml)*	Total time(min)*
HCl (6N)	Reserachers	8.12	2.99	2.04	08:29
HCl (6N)	Operator1	8.19	2.98	1.92	27:05
HCl (6N)	Operator2	8.60	3.06	1.71	21:48
H_2SO_4 (6N)	Researcher	8.54	3.07	1.83	06:35
H_2SO_4 (6N)	Operator3	8.66	3.09	1.80	19:51
H_2SO_4 (6N)	Operator4	8.54	3.09	1.84	38:24

*****The time lost between the acidifications of samples was not considered, but the time from the beginning to the end of acidification.

Concurrent to acidification time control, volume spent according to ± 0.02 mL standard deviation for H_2SO_4 and HCl both at 6 N (Tabble 1) was evaluated. Thus, considering the acidifications performed by the operators for each acid, H_2SO_4 was the one with the highest consistency, since it obtained a ± 0.02 mL standard deviation, obeying the allowed deviation limit. However, the HCl standard deviation was ± 0.167 mL. Concerning final volume, acidification carried out by the "Researchers" with H_2SO_4 required a smaller volume of acid compared toHCl used, even the first one having the highest initial pH, which confirms the H_2SO_4 higher efficiency.

Another factor to be considered is that H_2SO_4 in this last stage of study was evaluated in a leachate sample, in which acid had not been previously tested through potentiometric curves, thus obtaining satisfactory results in relation to the feasibility of using the same potentiometric curve for leachate at different times of the year. According to Ferreira (2012), which used H_2SO_4 and HCl concentrated both together and separately in the photo-Fenton process, sulfuric acid presented higher mineralization rates when compared to hydrochloric acid in the acidification stage, proving preference in their use, in addition to optimization of time and volume spent in the present study.

Based on the above considerations, it was observed the importance of achieving the desired pH with speed and efficiency for optimization of the process. Araújo et al. (2016) state that the use of chemicals to maintain optimum pH for the reaction of the advanced oxidative processes generates costs and, therefore, suggest a reduction of the reagent concentration, as done in the present work, in which it reduced 6 times the sulfuric acid concentration, resulting in less expensive treatment, reducing corrosion effect of materials and consequently, reagent with lower concentration released into the environment, a relevant factor for its efficiency without harming aquatic environment.

4 Conclusions

Sulfuric acid had a higher consistency than hydrochloric acid. In relation to concentration, pH results obtained were close to the desired pH at 6 N, with a 1.83 mL final volume of sulfuric acid to reach pH 3.0 ± 0.1. This spent volume represented 1.83% from the sample volume used (100 mL), optimizing the amount of acid that would be used, for example, on a real scale in the landfill or other industrial process. It is worth mentioning that the added volume was due to the initial pH, that it can undergo alteration depending on leachate constitutuion, time of year among other interferents.

Therefore, the relevance of optimization of the acidification step of the photo-Fenton process in the treatment of landfill leachate, both for the efficiency of the process and for environmental quality of the receiving body, is emphasized. This is because, when using the 6N sulfuric acid and potentiometric curves for definition of its ideal volume, there is a significant reduction in the total treatment time and reagents cost from the diluted acid, thus minimizing the impacts harmful to aquatic and terrestrial biota.

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