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Case Report

Application of a Fenton process after a biological nitrification treatment: A successful case for leachate treatment

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ABSTRACT

Leachate is one of the most complex wastewater due to the variable composition depending on landfill age, time, climate, waste composition, etc. Usually, conventional treatments are not enough to satisfy the environmental regulations for discharge. The incorporation of an advanced oxidation process, such as a Fenton procedure, into the treatment sequence can be a suitable solution to improve the discharge conditions of the effluent. The efficiency of applying the Fenton process to a real leachate from a sanitary landfill, previously treated by biological nitrification, has been evaluated at a preindustrial scale (1000 L), an aspect not very much found in the recent literature. The raw and the biologically treated leachate have been characterized, and a Fenton process was incorporated into the treatment sequence of the leachate in the effluent coming from the biological treatment. Optimal operational conditions ($[H_2O_2] = 0.2$ M, $[Fe^{2+}] = 4$ mM, pH 3 (25% sulfuric acid), 0.25% antifoam addition, and agitation) were used. Removal of about 90% of COD and BOD, and significant reductions of other parameters, such as concentration of surfactants and color, confirm that the Fenton process is an outstanding candidate to improve the conventional leachate treatment at a preindustrial scale.

1. Introduction

A municipal solid waste (MSW) landfill generates a leachate containing diverse pollutants: organic matter (e.g., humic and fulvic acids, phenols, aromatic hydrocarbons, pesticides, etc.), inorganic metallic components (Ca, Mg), chlorine (Cl), ammonia (NH_4^+), heavy metals (Cd, Cr, Pb), etc. [1]. A landfill leachate has a high contamination potential, especially dangerous for people living close to the release of the effluent [2] and has to be treated before discharge or reuse [3]. The composition of the leachate depends on the MSW composition, landfilling technology, and, mainly, the leachate age [1–5]. Generally, a conventional treatment is insufficient to achieve the decontamination degree required by the environmental regulations, and additional stages are necessary [6]. The UE Agency proposed multistage processes as the best available technology for treating landfill leachates [7].

Advanced Oxidation Processes (AOPs) such as Fenton processes can be combined to improve conventional processes as a pre- or post-treatment of a biological step. In the Appendix, Section A1, a simplified mechanism of the Fenton reaction Eqs. (S1)-(S13)) is presented, showing that H_2O_2 is activated by Fe^{2+} to generate mainly highly energetic $HO^•$ radicals that degrade recalcitrant compounds [8–10]. The Fenton and photo-Fenton processes, combined with a biological step, have been successfully used to treat landfill leachates [2,11–19]. In most papers, the Fenton process is applied before the biological treatment to convert recalcitrant compounds into biodegradable ones, allowing their removal by a further economic biological process; however, this generally leads to a high consumption of reagents and/or energy. On the other hand, when the wastewater contains some biodegradable fractions, the Fenton step can be used as a post-treatment (polishing step) [20]. As the variation in the leachate characteristics hinders the establishment of universal treatment conditions for landfill leachates [21], several laboratory experiments must be first performed to find the optimal conditions for a specific leachate.

On the other hand, the scaling of laboratory experiments to pilot-,

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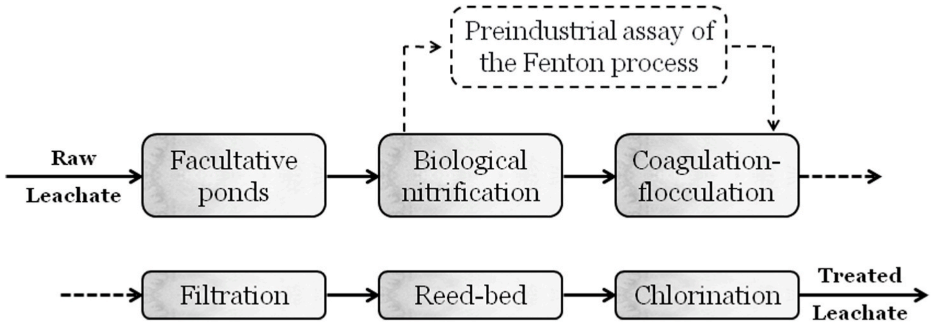

Fig. 1. Scheme of treatment sequence of the leachate indicating the point where the Fenton process is intended to be coupled.

preindustrial- or industrial-scale must be carefully carried out to characterize the technical and economic feasibility of the application of a Fenton process at full-scale and only some studies investigated the leachate treatment by Fenton at preindustrial or industrial scale [13–15,17,18,22–24].

The objective of this work is to present results of the treatment of a real leachate (previously treated by biological nitrification) by Fenton technology, carried out directly in a real sanitary landfill facility and using industrial quality reagents. The interest is to present preindustrial scale Fenton treatments, which are scarcely reported in the literature.

2. Experimental

2.1. Materials and methods

Industrial quality reagents were used: phosphoric acid (Glensol, 85%), caustic soda (Glensol, 25%), Glensol 5919 antifoam, $FeSO_4.7H_2O$ (Servinlab®), H_2O_2 (Interox®, 200 vol), H_2SO_4 (Servinlab®, 25%) and Na_2CO_3 (Biopack®, reagent grade). For pH measurements, a Hach HQ30d pH meter was used.

Ammonia, nitrate, and nitrite were determined using colorimetric methods with Hach TNT832, TNT836, and TNT840 reagents, respectively, and a Hach DR1900 spectrophotometer. Volatile suspended solids (VSS) in the reactor were periodically determined through standard methods [25].

2.2. Leachate treatment layout

This work was performed at the Fachinal sanitary landfill (Misiones province, Argentina, 27°38′25″ S, 55°48′0″ W), where the raw leachate is collected from 12 cells of different ages (1–20 years) containing MSW. The sequence used to treat the raw leachate is indicated in Fig. 1, i.e., a homogenization in six facultative ponds ($20 \times 60 \times 4.5$ m^3), followed by a biological nitrification, a coagulation-flocculation stage, a filtration of the solid residues, a reed-bed phytoremediation step with Vetiver grass (*Chrysopogon zizanioides* in a subsurface flow configuration), and a chlorination step. Fig. 1 shows the point where the Fenton process was intended to be coupled. A dashed line was used to indicate that the Fenton process is not a usual stage in the treatment sequence of the company.

The facultative ponds provide a stable leachate in terms of organic load, and with a reduced solid content. An example of the characteristic parameters of a real leachate of the facility that enters the biological step is presented in Table A1 of Section A2 of the Appendix. The leachates present a relatively low toxicity and some biodegradability degree that allow their efficient biological treatment. Therefore, in the present case, the Fenton process would be more efficiently applied as a post-treatment (Fig. 1). Moreover, this configuration avoids the consumption of a high amount of reagents, which represents 85% of the total cost of a Fenton process [19].

In Section A3 of the Appendix, the characteristics and equipment used in the biological treatment are indicated. The characteristic physicochemical parameters of a leachate sample taken before and after the biological nitrification are presented in Table A2. It is important to remark that the initial variables are not the same as indicated in Table A1 as a different sample was taken for this experiment. As Table A2 shows, the biological treatment did not reduce significantly the COD but achieved more than 90% of BOD reduction, with a final biodegradability (determined as (BOD/COD)*100) less than 2%. Ammonia was also significantly reduced (94%), and ABS was reduced to about 65%.

2.3. Fenton process

The Fenton process conditions used at the preindustrial scale were searched previously in bench-scale assays (1 L). The H_2O_2 theoretical concentration was set based on literature data ($[COD]/[H_2O_2] = 0.47$ [26]) and on the leachate COD (Table A1). The concentration of dissolved Fe in the sample of the leachate (measured before applying the Fenton treatment) was 31 mg/L. Since the minimal required Fe^{2+} concentration was 223 mg/L (calculated based on reported $[H_2O_2]/[Fe^{2+}]$ molar ratios [27]), $FeSO_4$ was added externally. To verify if the addition of ferrous salt improved the efficiency of the leachate treatment, a preliminary test assaying $[H_2O_2]$ *vs.* $[H_2O_2]/[Fe^{2+}]$ was performed at bench scale, finding an increase of about 50% in the efficiency by the addition of ferrous salt. An antifoam was added before the incorporation of H_2O_2, due to the presence of alkylbenzene sulfonate (ABS) coming from detergents and surfactants.

To find the optimal conditions for the Fenton stage, several experiments at bench scale were performed trying different H_2O_2 concentrations (0.1–0.8 M), $[H_2O_2]/[Fe^{2+}]$ molar ratios (2.5–50), pH (1–4), temperature (10–40 °C), acid dosage (in one step or by pulses, using 10–98% H_2SO_4), antifoam addition (0.1–10%), different agitation procedures [28–31].

A further scale-up to pilot-scale (100 L) was applied in the optimal conditions: $[H_2O_2] = 0.2$ M and $[Fe^{2+}] = 4$ mM ($[H_2O_2]/[Fe^{2+}]$ molar ratio = 50), pH 3, room temperature (RT), acid dosage in one step with 25% H_2SO_4, antifoam = 0.25%/v and agitation with a stirring rod only during addition of reagents. The efficiency of the industrial quality reagents was similar to that of reagents used generally at laboratory scale [28,31].

For the preindustrial scale, a sample (1000 L) from the outlet of the biological step was loaded in a high-density polyethylene (HDPE) batch reactor of 1050 L ($1 \times 1 \times 1.05$ m) open to the atmosphere, and the optimal amount of reagents obtained from the pilot-scale tests was added. A neutralization step after the Fenton process, necessary for the leachate treatment sequence, was tested in a laboratory assay on a 500 mL sample of the Fenton treated leachate, adding 25% NaOH, as will be reported in section 3.2.

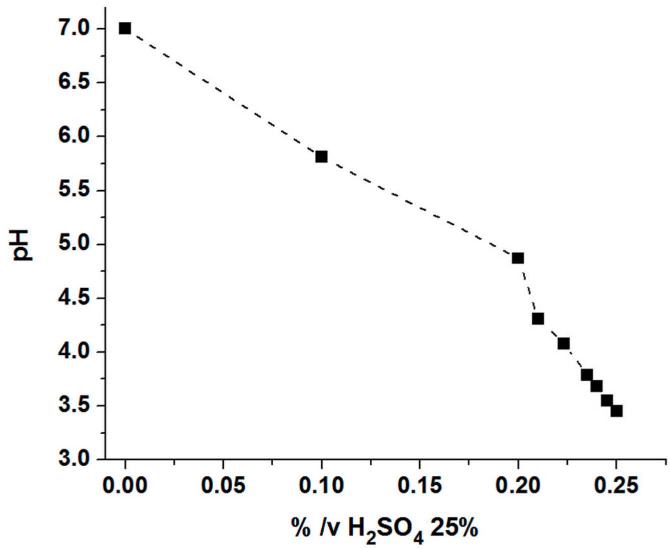

Fig. 2. Acidification curve of the biologically treated leachate with 25% H_2SO_4, at preindustrial scale.

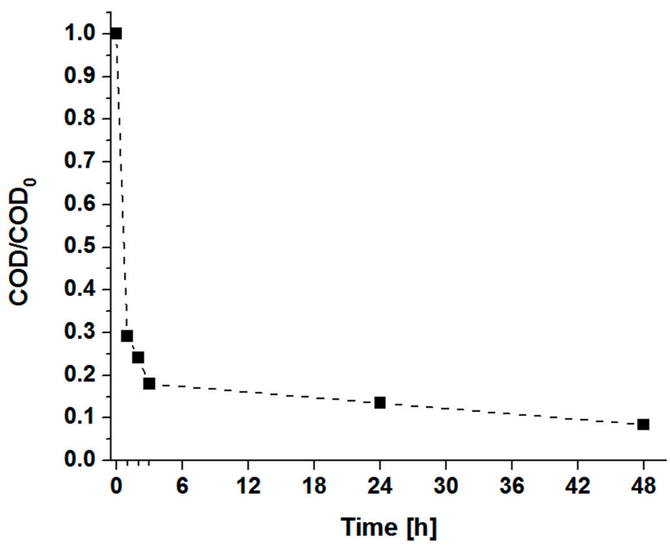

Fig. 3. COD/COD_0 evolution during the Fenton treatment of the leachate performed at the preindustrial scale.

2.4. Analytical determinations

During the Fenton treatment, samples were periodically withdrawn, and COD, BOD, N–NH_3, ABS, and soluble Fe were determined by standard methods [25]. COD was used to follow the behavior of the process, as a global decontamination parameter. A Na_2CO_3 saturated solution (390 g/L) was used as an inhibitor of the remaining H_2O_2 on the COD determination in samples withdrawn at 1, 2, and 3 h, adding 3 mL to a 10 mL sample at RT, and heating at 90 °C for 10 min [32]. For experiments lasting 24 and 48 h, Na_2CO_3 addition was not needed.

For color measurements, a sample was filtered through a 0.45 μm Xinxing® filter, and the absorption of the solution was measured at 455 nm with the Hach spectrophotometer, reporting the values in the platinum–cobalt (PtCo) scale [25].

Table 1
Percentages of reduction of N–NH_3, BOD, and ABS after 24 and 48 h of Fenton treatment.

Reaction time (h)	N–NH_3	BOD	ABS
24	28%	48%	70%
48	44%	88%	71%

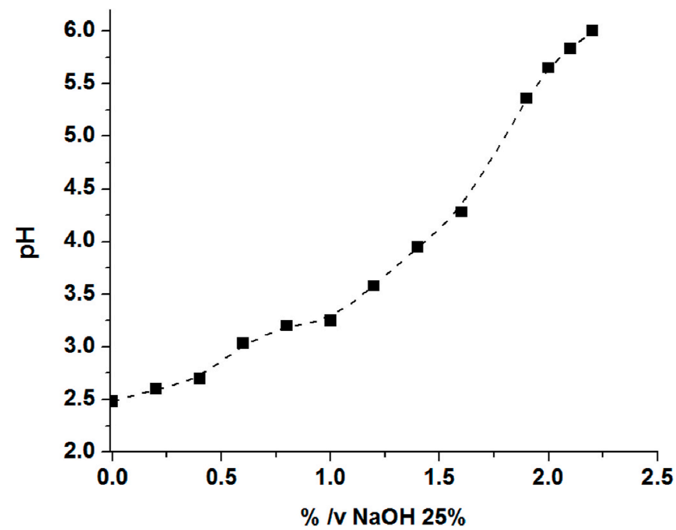

Fig. 4. Neutralization (with 25% NaOH) curve of a 500 mL sample of the leachate treated by Fenton (taken from the reactor at preindustrial scale).

3. Results and discussion

3.1. Preindustrial Fenton treatment

The optimal $FeSO_4$ and antifoam amounts were added to a 1000 L sample of the biologically treated leachate into the preindustrial-scale reactor. Before H_2O_2 addition, 2.5 L of 25% H_2SO_4 were added, acidifying the mixture up to pH 3.4. The acidification curve of the leachate is presented in Fig. 2, and it indicates that the addition of 0.20%/v H_2SO_4 reduces pH only in two units (from 7 to 5) and that an extra addition of 0.05%/v of H_2SO_4 reduces pH from 5 to a pH near 3; pH dropped to 3 after the H_2O_2 addition, to 2.59 at 3 h and 2.47 at 72 h. The evolution of COD in the leachate during the Fenton treatment is presented in Fig. 3.

After 3 h of Fenton treatment, a COD reduction higher than 80% was achieved (from 3275 to 589 mg/L, Fig. 3). Considering the time required for loading and unloading the reactor (0.5 h to load, 3 h for Fenton treatment, and 0.5 h to unload), each 1000 L batch took 4 h to be treated. This means that it is possible to treat 6 m^3 of leachate per day, reducing 80% COD. Moreover, if a hydraulic retention time of 48 h is applied, a COD reduction superior to 90% could be achieved (from 3275 to 275 mg/L, Fig. 3). Table 1 shows the values of N–NH_3, BOD, and ABS after 24 and 48 h of Fenton treatment. Reductions of 44, 88, and 71% were obtained, respectively, after 48 h of Fenton treatment, values similar to those achieved with other technologies (nanofiltration or adsorption) [6].

Table A3 in Section A5 of the Appendix presents the values of some additional parameters of the leachate treated for 48 h, showing noticeable low values of BOD, COD, nitrates, phenolic substances, NH_4^+, total and fecal coliforms.

3.2. Neutralization

Neutralization is necessary after a Fenton process to continue the treatment or to discharge the effluent to a receiving water body to comply with the regulations. For this purpose, 500 mL of the Fenton

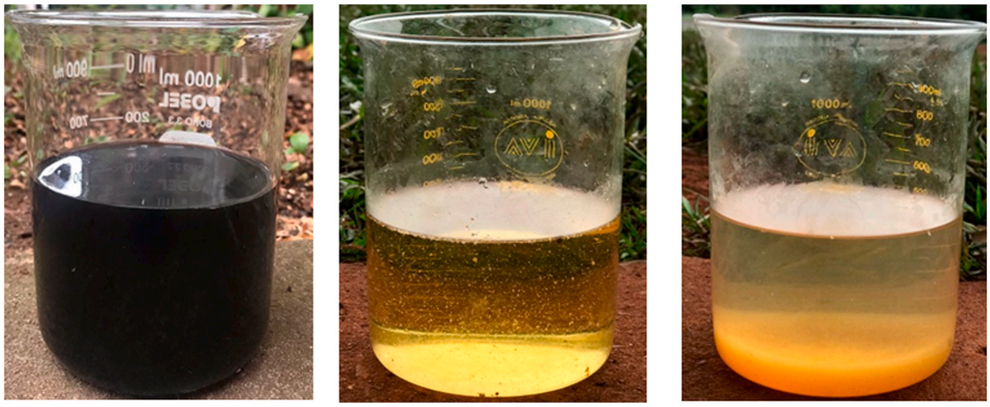

Fig. 5. Photographs of leachate samples before the Fenton process (left), after the Fenton process at pH 3 (center), and after neutralization at pH 6 (right).

treated leachate were taken from the preindustrial reactor and neutralized to pH 6 through the addition of 2.2%/v (11 mL) 25% NaOH (Fig. 4).

Moreover, the concentration of Fe in solution was reduced to 15 mg/L as a result of the precipitation of iron oxyhydroxides, which was clearly observable by the appearance of a brown precipitate [8,21] (see later, Fig. 5).

3.3. Improvement of the aesthetic features of the leachate and some economical aspects

A remarkable improvement in the aesthetic features of the leachate was achieved after the application of the Fenton process, as observed in experiments done in field at benchscale (Fig. 5). The initial dark and opaque untreated leachate (13200 [PtCo]) was transformed into a clear and light yellow solution (398 [PtCo]) after the biological + Fenton treatment of the leachate at pH 3, followed by a change to light brown (2640 [PtCo]) after neutralization to pH 6 [3].

The color of the leachate is mainly due to the presence of organic matter, associated suspended solids, and turbidity due to humic, fulvic acids, or other organic compounds [33]. The Fenton process can degrade these organic compounds. The solids settled after neutralization at pH 6 (Fig. 5, right) could be separated and integrated into the sludge treatment line existing in the landfill facility.

The cost of industrial-grade chemicals was ≈20 USD/m^3 leachate. The next step is to follow through with the scaling-up to the industrial scale, as a continuous system, to treat the total leachate flow rate. In that case, a further economic study must be necessary to evaluate the feasibility.

It is important to mention that the leachate treated by the Fenton process was incorporated into the treatment sequence of the company (Fig. 1), in order to complete its treatment for discharge according to the normative of the Misiones province in Argentina [34].

4. Conclusions

The successful application of a Fenton process to a real leachate from a sanitary landfill of the Misiones province in Argentina, previously treated by biological nitrification, has been evaluated at a preindustrial scale. Real operational parameters were determined, providing information on the treatment conditions at a preindustrial scale, in a real treatment facility, and using industrial quality reagents.

COD, N–NH_3, BOD, and ABS reductions after 48 h of Fenton treatment were very good (90, 44, 88, and 71%, respectively). Additionally, perceptual parameters of the leachate such as color and turbidity were significantly enhanced. Another advantage is that a photo-Fenton process is not needed, saving energy consumption.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.cscee.2022.100208.

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