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Ultrafiltration, chemical and biological oxidation as process combination for the treatment of municipal landfill leachate

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ABSTRACT

A hybrid treatment process was evaluated to reduce the organic load of leachate generated in a former landfill. The leachate, regardless of the time the waste was deposited ("leachate A" has its source in young waste and "leachate B" has its source in old waste), presented high recalcitrance as evidenced by high pH values, salinity (chlorides), COD/BOD$_5$ ratio and concentration of metals. The fractionation of leachate through filtration using micro- and ultrafiltration membranes revealed that leachate B presented a higher proportion of soluble organic compounds, and its aging led to the decomposition of macromolecules. The ultrafiltration of leachate B with a 20 kDa cut-off membrane to a volume concentration factor of 3.2 and transmembrane pressure close to 1.0 bar resulted in flows of 15 to 20 L.m^{-2}.h^{-1} and removal efficiencies of 76% and 79% for COD and color, respectively. The concentrate, when treated with Fenton reagent at pH 4.0, 0.05 mol H$_2$O$_2$.L^{-1} and 200 mg Fe^{2+}.L^{-1}, presented its TOC content reduced by 79%. The aerobic biodegradation of the permeate and concentrate after chemical oxidation was evaluated, obtaining TOC removals in 24 h of 29% and 56%, respectively, indicating that even after the retention of a significant fraction of organic matter by the membrane, the leachate still presented compounds difficult to degrade and that the chemical oxidation contributed to the recalcitrance reduction.

Keywords: Landfill leachate treatment; Ultrafiltration; Fenton oxidation, Biodegradation; Hybrid process

1. Introduction

The accumulation of urban solid waste in municipal landfills, along with water from rain that percolates the garbage mass, produces a residual liquid which contains a complex mixture of organic and inorganic substances, dissolved or in colloidal state, in addition to many species of microorganisms. This residual liquid, called leachate, originates from three main sources: natural garbage moisture, which is significantly aggravated during rainy periods; water released during decomposition of various materials; and liquid from the solubilization of organic matter by the microbial population naturally present in the garbage mass [1].

Results from several studies indicate that high levels of organic pollutants predominate in active decomposition stages, gradually decreasing as the landfill stabilizes. Furthermore, minerals are still being lixiviated for several decades. The contamination of surface and underground waters by leachate in areas adjacent to the landfill is a potential problem in the design, operation and care of a landfill [1–4].

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In several studies on the treatability of leachate, researchers have investigated a number of different treatment options including chemical, physicochemical and biological treatment methods [5–10]. Leachates from old landfills have low biodegradability and biological treatment as the first stage of the treatment sequence is not recommended. In the case of physicochemical treatment, high doses of coagulant elevate the process cost significantly. New technologies are being studied with the objective of minimizing the toxic effects caused by recalcitrant and/or toxic components. The combination of different processes, called "hybrid processes", such as the combination of biological and chemical processes (oxidation) and those involving membrane separation processes, has shown to be promising in the treatment of high-complexity wastewaters [11–14].

Aerobic biological oxidation is limited when the feeding is recalcitrant to degradation and/or inhibitory and toxic to bioculture. This recalcitrance, which often can be attributed to the presence of compounds of high molecular weight, can be removed by means of ultrafiltration (UF), thus increasing the biodegradability of the effluent. In turn, in chemical oxidation processes, the reaction mechanisms change the structure and chemical properties of organic substances. The molecules are burst into smaller fragments and high percentages of oxygen appear in these molecules in the form of alcohol and carboxylic acids that, in most cases, are more readily biodegradable than the original compounds [12]. Among the chemical processes used in the treatment of wastewaters, the use of H_2O_2 plus Fe^{+2}, the Fenton reagent has gained strength [14–16]. The Fenton reagent has three characteristics that make its use highly attractive: $^{\bullet}OH$ radicals produced in the reaction of H_2O_2 with Fe^{+2} rapidly react with a wide variety of organic substances present in wastewaters; reagents are easy to handle and do not affect the environment, and H_2O_2 is already commonly used in the treatment of industrial wastes to minimize the chemical oxygen demand (COD) and ion Fe^{2+} presents low cost, reducing the treatment cost [15].

There are reports on the application of the hybrid process for the treatment of leachate with recalcitrant characteristics in the recent literature. Pala and Erden [17] treated leachate with organic flocculants, chemical oxidation with Fenton reagent and adsorption with activated charcoal. Higher COD (79%) and color (98%) removal efficiencies were obtained with the Fenton reagent (with 2500 mg $H_2O_2.L^{-1}$ and 2500 mg $FeSO_4.7H_2O.L^{-1}$) and the addition of lime [17]. Gulsen and Turan [18] employed a fluidized bed anaerobic reactor (with sand as support) combined with oxidation with Fenton. The bioreactor treated the leachate by removing 80–90% of COD and the organic and inorganic biorefractory components were removed in a chemical oxidation stage with Fenton. Under optimum pH (2.5) and H_2O_2 (1200 $mg.L^{-1}$) conditions and at an organic load of 2.0 kg $COD.m^{-3}.d^{-1}$, a COD removal of 85% was observed in the oxidation step [18].

Therefore, this study aimed to evaluate a hybrid treatment process involving the leachate fractioning with different cut-off membranes, chemical oxidation of the concentrate generated on the permeation and the aerobic biological treatment of the permeate and concentrate after chemical oxidation. The results may serve as reference for the use of hybrid procedures in the treatment of other recalcitrant and also problematic wastewaters, such as those generated in the pulp and paper, textile, chemical and leather industries.

2. Material and methods

2.1. Leachate collection

The Gramacho metropolitan landfill is situated near the Sarapuí and Iguaçu Rivers at the margins of Guanabara Bay, in Duque de Caxias (Rio de Janeiro, Brazil). It occupies an area of more than 112 hectares and has been in operation since 1978, receiving solid domestic waste from the city of Rio de Janeiro and neighboring municipalities.

Leachate was collected from two different sites of the landfill: a collection site with waste deposited 6 months ago (called "leachate A") and a collection site with waste deposited 3 years ago (called "leachate B"). Both samples were stored in a freezer for characterization, fractionation and degradation tests.

2.2. Leachate fractioning with micro- and UF membranes

Preliminary filtration tests were conducted with Whatman 40 filter paper (pore size of 8 µm) and cellulose ester membranes of 0.22 and 0.45 µm of average pore size. These tests were conducted in Millipore filtration vacuum equipment (flat membrane, dead-end filtration). Membranes of 0.45 µm were used for the purpose of removing suspension material. Then, UF tests were conducted with polysulfone membranes of different cut-offs (50, 20 and 10 kDa). The cut-off of a membrane is defined as the molar mass of a standard solute which rejection rate is equal to or above 95%.

The fractioning was made in cascade; in other words, the leachate was initially filtered with a higher cut-off membrane, and the permeate was then filtered using a lower cut-off membrane and so on. The permeation unit—a laboratory-scale flat geometry module (crossflow filtration)—consisted basically of a feeding vessel, gear pump, permeation cell, rotameter and manometers. The conditions used in the cross-flow permeation system were pressure of 1.0 bar and circulation flow of 60 $L.h^{-1}$. In each test of microfiltration or UF a new membrane was used.

2.3. Leachate oxidation with Fenton reagent

Preliminary tests were conducted in Jar test equipment following the same procedure adopted for coagulation/flocculation tests. The determination of the best conditions in terms of pH, H_2O_2 and ferrous ion concentration was achieved using leachate B at room temperature (25±2°C) and a reaction time of 25 min.

To determine the optimum pH, beakers of 500 mL were filled with 300 mL of leachate and the pH adjusted with NaOH or H_2SO_4. Each beaker was filled with certain volume of a stock iron solution (ferrous sulfate) to obtain a final iron concentration of 500 $mg.L^{-1}$. Later, H_2O_2 50% (w/w) was added to a concentration of 0.05 $mol.L^{-1}$ and the oxidation process started. The coagulation stage was performed for 5 min under strong agitation (100 rpm). To continue the test, the agitation speed was reduced to 20 rpm over 20 min for the flocs formation, thus characterizing the flocculation step. Thereafter, the agitators were removed from beakers and the samples were kept in rest for 1 h to provide the decantation of the flocs formed. The supernatant from each sample was carefully removed and total organic carbon (TOC) and color analyses were conducted to evaluate the oxidative process.

After pH determination, which provided greater TOC and color reduction, a study was conducted to determine the optimal hydrogen peroxide concentration. 300 mL of leachate were added to each beaker, and the pH was adjusted to the optimum value indicated in the previous test. Different volumes of H_2O_2 50% (w/w) were added to each beaker to obtain different concentrations and certain volume of a stock ferrous sulfate solution, obtaining an iron concentration of 500 $mg.L^{-1}$. The coagulation, flocculation and decantation stages were conducted as described above. The supernatant of each sample was carefully removed and TOC and color analyses were conducted to evaluate the oxidative process.

To carry out the study on the iron concentration, each sample was submitted to several Fe^{2+} concentrations (100–1000 $mg.L^{-1}$), keeping pH and H_2O_2 concentration at previously determined optimal values. The tests were conducted under the same conditions indicated in the previous tests, and the supernatant of each sample was carefully removed for TOC and color analyses.

The best conditions identified in tests with leachate B were employed in the concentrate chemical oxidation resulting from the permeation of the leachate B with a 20 kDa cut-off membrane.

2.4. Leachate aerobic biodegradability

To assess the biodegradability of different leachate fractions after the process with membranes (permeate and concentrate), bench-scale bioreactors were employed. The bioreactors consist of aerated and agitated tanks in which a heterogeneous microbial population is introduced. The sludge has been adapted to the leachate permeate (20 kDa cut-off membrane), gradually increasing the permeate proportion in the feeding, consisting of raw domestic sewage and permeate. An initial volume of raw domestic sewage was inoculated with activated sludge (10% v/v) from a sewage treatment station and aeration and agitation were initiated. An aliquot of this mixed liquor was filtered (0.45 μm membrane) and stored at 5°C to TOC analyses. After 24 h, the aeration and agitation were switched off to allow the decanting of the biomass, an aliquot of the supernatant was sampled and filtered (0.45 μm membrane) to TOC analyses. If the TOC removal (calculated after analyses) was lower than 70%, the supernatant was removed and a new feed (raw domestic sewage) was introduced into bioreactor for more 24 h. If TOC removal was equal or higher than 70%, the feed composition was modified for 90% (v/v) of raw domestic sewage and 10% (v/v) of permeate. Then, aeration and agitation were switched on again and a new trial was conducted. This procedure was repeated until 100% of permeate was attained in the feed. A period of 2 weeks was necessary for the complete adaptation of the sludge to permeate. After adaptation period, the hydraulic retention time was kept constant at 24 h for all batches whereas the sludge retention time (SRT) was practically infinite, as almost no waste sludge removal took place during an operational period of 5 days. The initial concentration of total suspended solids in the bioreactor was 2000 mg/L. In an attempt to recover the sludge activity, the biomass concentration was doubled and the medium was then supplemented with nitrogen and phosphorus through the addition of urea and monobasic potassium phosphate in a proportion of TOC:N:P of 100:5:1. The sludge adaptation and the monitoring of biodegradability trials were followed by means of TOC analyses.

2.5. Analytical methods

Each fraction obtained was characterized in relation to color, pH, COD, TOC and fixed and volatile total solids. All these parameters, in addition to those employed in the leachate characterization were analyzed by standard methods [19]. Each sample was analyzed in triplicate and the error range of the analytical methods was of 5–10%.

3. Results and discussion

3.1. Leachate characterization

Table 1 presents the physicochemical composition obtained for leachate samples collected from the Gramacho landfill with different times of disposal. The

Table 1
Characteristics of the leachate from the Gramacho landfill collected in places with different disposal times

Parameter	Leachate A	Leachate B
pH	8.6	8.6
Color	2,454	4,860
Total solids	16,810	19,390
COD	2,520	5630
BOD_5	200	480
Total organic carbon	790	1,250
Total inorganic carbon	1,685	1,715
Chloride	32,420	36,530
Calcium	74.85	23.38
Sodium	>183.9	>183.9
Iron	9.16	7.87
Potassium	>185.1	>185.1
Magnesium	206.7	124.5
Aluminum	2.73	0.90
Zinc	0.75	0.23
Manganese	0.30	0.02
Barium	0.40	0.30
Nickel	0.28	0.52
Chromium	0.44	1.03
Strontium	11.07	12.34
Rubidium	1.70	4.35
Cobalt	0.07	0.13
Bromine	28.02	24.31
Iodine	0.57	1.09
Copper	1.78	0.46

All parameters are displayed in $mg.L^{-1}$, except for pH and color (Pt-Co units).

alkaline pH is due to advanced biodegradation stage in the landfill, indicating that the most easily assimilable organic material has probably been used by microorganisms present in the garbage mass. The high COD/BOD_5 ratio (around 12) also corroborates this fact.

High color levels are peculiar in leachate from old landfills, being associated with the presence of fulvic and humic substances formed in the anaerobic fermentation process. Calace and Petronio [20] determined that in leachate from old landfills, a considerable amount of macromolecular organic compounds are present. The characterization of these compounds showed that they are similar to humic and fulvic acids.

Data presented in Table 1 indicate that the leachate presented a high content of total solids, and that most of these salts were probably dissolved under the form of chlorides, since the content of this ion in the leachate was quite high. The total inorganic carbon content (68% of the total for leachate A and 58% for leachate B), associated with carbonates and bicarbonates, confirms the significant presence of these salts in the leachate. In relation to the organic matter, it has always been at levels below those of salts, but still, in appreciable levels.

It can be concluded that Gramacho's leachate, regardless of the time of waste disposal, presented high recalcitrance. This can be evidenced by the high pH values, salinity (chlorides) and COD/BOD_5 ratio, besides the presence of several metals.

3.2. Leachate fractioning with micro- and UF membranes

The results of the leachate fractionation with microfiltration membranes are presented in Table 2. The filtration on a filter paper contributed only for the removal of small portion of the total solids from leachate A (20%), hardly changing the total solids of leachate B and neither color, COD and TOC of both leachate. On the other hand, filtration with a 0.45 μm membrane contributed to a reduction of 49% on color, 23% on COD, 30% on TOC and 55% on the total solids from leachate A. The low additional removals obtained with filtration with a 0.22 μm membrane (from 0 to 16%) would not compensate for the decrease on the filtrate flow due to the smaller size of the membrane pores.

In filtration tests using leachate B, the best removal efficiencies were obtained with the 0.22 μm membrane (79% for color, 37% for COD and 45% for total solids), with the exception of TOC in which the best removal efficiency (30%) occurred with the membrane of 0.45 μm.

With respect to the analysis of fixed and volatile total solids, the values obtained indicated that much of the total solids present in the leachate are inorganic (from 77 to 88% of the total solids). The fractioning of these solids with different membranes indicated that a significant percentage of inorganic solids (around 73% for leachate A and 55% for leachate B) is retained by the 0.22 μm membrane. While organic solids (volatile) are retained in much smaller proportions, 34% and 51% of reduction for leachate A with 0.45 μm and 0.22 μm membranes, respectively, were obtained, and no reduction with leachate B. This result indicates that leachate B presents a greater proportion of soluble organic compounds, and the aging of the leachate leads to the decomposition of macromolecules, facilitating the permeation through membranes of smaller pore size.

Different fractions obtained from the A and B leachates through UF membranes were characterized in relation to color, pH, COD and TOC (Table 3). Analyzing the quality of permeate from each membrane, it may be concluded that in the fractioning of leachate B, better removal efficiencies were obtained with the 50 kDa cut-off membrane (66% for color and 72% for COD). The continuation of the fractionation with 20 and 10 kDa cut-off membranes did not increase the quality of the permeate significantly, especially with respect to parameter COD. With leachate A, the 20 kDa cut-off membrane presented good results in terms of color (44%) and COD (63%) removals, and

Table 2
Preliminary results for the leachate fractionation with microfiltration membranes

Leachate	pH	True color (mgPt-Co.L^{-1})	COD (mg.L^{-1})	TOC (mg.L^{-1})	TS (mg.L^{-1})	TVS (mg.L^{-1})	TFS (mg.L^{-1})
Leachate A							
Raw	8.6	2,454	2,520	790	16,810	3,880	12,930
Filtrate, filter paper	8.6	2,625	2,650	720	13,430	3,230	10,200
Filtrate, 0.45 µm	8.6	1,242	1,940	553	7,620	2,570	5,050
Filtrate, 0.22 µm	8.6	860	1,700	615	5,380	1,910	3,470
Leachate B							
Raw	8.6	4,860	5,630	1250	19,390	2,350	17,040
Filtrate, filter paper	8.6	5,020	5,750	1132	18,760	2,560	16,200
Filtrate, 0.45 µm	8.5	3,140	5,535	874	15,660	—	12,610
Filtrate, 0.22 µm	8.6	1,025	3,525	927	10,720	—	7,720

TS, total solids; TVS, total volatile solids; TFS, total fixed solids. TVS values for filtrates 0.22 and 0.45 µm of leachate B are not shown due to high analytical error.

Table 3
Characterization of A and B leachates after fractionation through ultrafiltration

Leachate	VCF	pH	True color (mgPt-Co.L^{-1})	Color removal (%)	TOC (mg.L^{-1})	TOC removal (%)	COD (mg.L^{-1})	COD removal (%)
Leachate A								
Filtrate, 0.45 µm		8.6	1,244		558		1,940	
Concentrate, MW >50	3.0	8.6	2,030		1,250		2,065	
Permeate, MW <50		8.6	1,120	10.0	579	0.0	1,620	16.5
Concentrate, MW >20	2.0	8.6	1,590		985		1,650	
Permeate, MW <20		8.6	700	43.7	432	22.6	726	62.6
Concentrate, MW >10	1.2	8.6	880		651		934	
Permeate, MW <10		8.6	356	71.4	522	6.5	879	54.7
Leachate B								
Filtrate, 0.45 µm		8.6	3,140		874		5,535	
Concentrate, MW >50	3.2	8.6	6,360		1,640		5,860	
Permeate, MW <50		8.7	1,080	65.6	857	1.9	1,560	71.8
Concentrate, MW >20	2.0	8.6	3,640		1,102		3,275	
Permeate, MW <20		8.6	800	74.5	749	14.3	1,480	73.3
Concentrate, MW >10	1.1	8.6	900		834		2,050	
Permeate, MW <10		8.7	420	86.6	676	22.7	1,705	69.2

VCF, volume concentration factor = volume feed/volume concentrate; MW, molecular weight (kDa).

therefore, this membrane was selected for the permeation step in the treatment sequence. The results presented in Table 3 show that the filtration with membrane of cut-off of until 20 kDa still resulted in the separation of organic substances. The filtration with 10 kDa cut-off membrane resulted in an additional color removal. However, the separation of organic substances was not verified because the TOC and COD values in permeates were very similar (considering the analytical error).

Repeating the procedure for the UF of leachate B and keeping the same concentration factor (3.2) for each of the membranes tested separately generated permeates with similar quality. The 20 kDa cut-off membrane generated permeates with the best color (79%) and COD (76%) removals.

In order to obtain efficiency data of membranes with respect to the permeate flow, permeation tests were conducted with each membrane (Fig. 1) fed with raw leachate filtered only in a filter paper (Whatman 40). Generally, the flow of leachate A was always higher than leachate B. This probably is due to the larger amount of colloidal material in leachate B, which contributes to the phenomenon known as fouling, to be observed in shorter periods of time. As expected, the 50 kDa cut-off membrane presents the highest permeate flows, followed by 20 and 10 kDa cut-off membranes, due to the larger pore size.

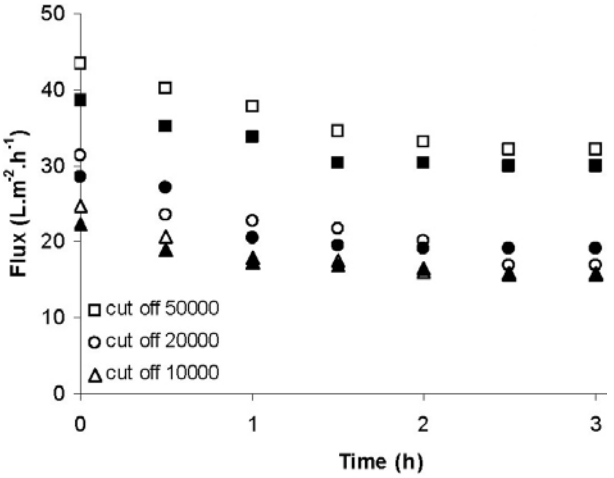

Fig. 1. Permeate flow over operating time of the ultrafiltration unit. (□, ○, Δ) = leachate A; (■, ●, ▲) = leachate B.

The flow of permeate decreased along the time of operation, and a more accentuated decrease was observed with the 20 kDa cut-off membrane. With this membrane, the flow in stationary regime ranged from 15 to 20 L.m^{-2}.h^{-1}.

3.3. Leachate oxidation with Fenton reagent

The results of the leachate B oxidation test with Fenton reagent are found in Figs. 2(a–c). One can see that the best oxidation conditions were obtained at pH 4.0, hydrogen peroxide concentration of 0.05 mol.L^{-1} and ferrous ion concentration of 200 mg.L^{-1}.

The best result at pH 4.0 was already expected and is in agreement with data from literature. The amount of HO• formed in the Fenton process is affected by the solution pH and HO• can be effectively formed under acidic conditions. Kim et al. [16] reported that the photo-Fenton process could remove refractory and toxic organic compounds from the leachate efficiently at pH 3.0.

The degradation of the organic matter increased with H_2O_2 concentration up to 0.05 mol.L^{-1}, obtaining 86% of TOC and 93% of color removal in this concentration. Increased Fe^{2+} concentration, maintaining the H_2O_2 concentration at 0.05 mol.L^{-1}, led to a small increase on the TOC removal. Better TOC (90%) and color (94%) removals were obtained with a Fe^{2+} concentration of 200 mg.L^{-1}. However, higher Fe^{2+} concentrations decreased the TOC and color removal efficiency. This result can be attributed to the reaction between Fe^{2+} and H_2O_2 to produce several complexes such as $Fe(HO_2)^{2+}$ and $Fe(OH)(HO_2)^{2+}$, which decompose to produce the hydroperoxyl HO_2• radical and Fe^{2+}. Since the HO_2• radical is much less reactive with organic matter, the TOC removal efficiency is reduced [21].

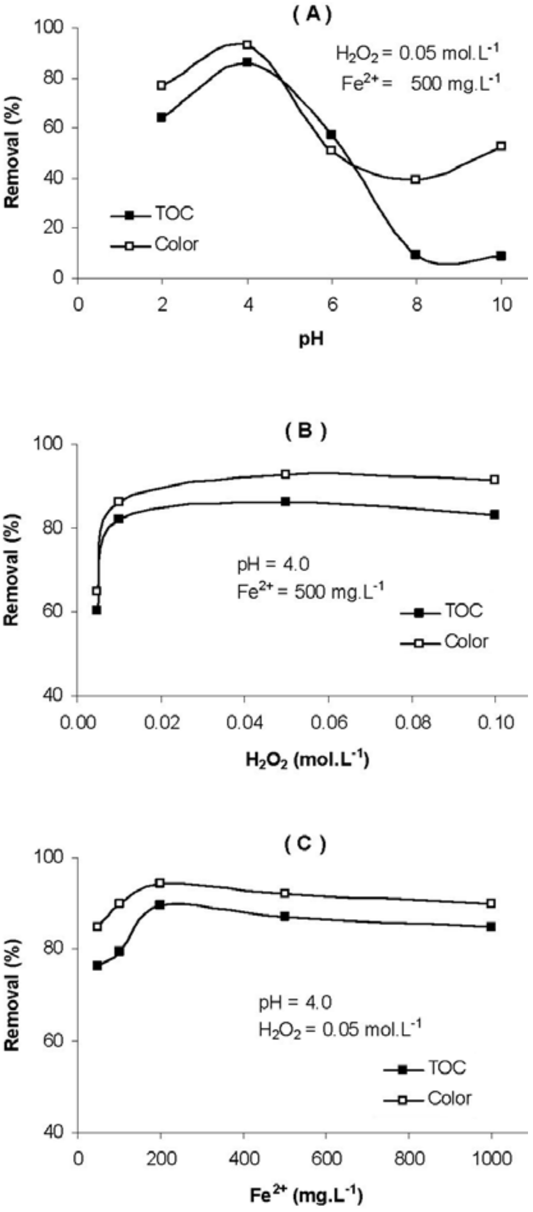

Fig. 2. TOC and color removal from leachate B after oxidation with Fenton changing pH (a), H_2O_2 (b) and (c) Fe^{2+} concentrations. Time of reaction, 25 min.

The best conditions identified in tests with leachate B were employed in the concentrate chemical oxidation resulting from the permeation of leachate B with a 20 kDa cut-off membrane, obtaining a TOC reduction of 79% (TOC concentrate = 3172 mg.L^{-1} and TOC after oxidation = 655 mg.L^{-1}).

3.4. Leachate aerobic biodegradability

The permeate obtained in the filtration of leachate B with the 20 kDa UF membrane was used in the adaptation

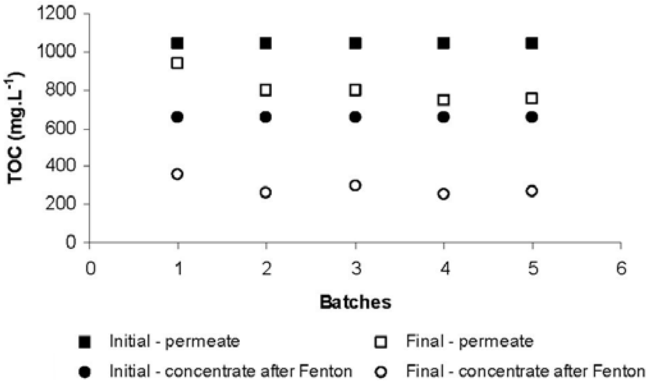

Fig. 3. TOC concentration along the aerobic biodegradation tests of permeate and concentrate (after oxidation with Fenton) of leachate B with a 20 kDa cut-off membrane. Initial and final = TOC concentration at the initial (T_0) and after 24 h (T_{24}) of the biodegradation tests.

process of the aerobic sludge in biodegradability trials. The adaptation of the sludge to the leachate permeated lasted about 15 days, obtaining final TOC removals (with 100% of permeated leachate as feeding source) of the order of 40%. With the continuation of feeding with 100% of permeated leachate, a drop in the TOC removal efficiency values to around 14% was observed, as obtained in biodegradation experiments with raw leachate reported in literature [22].

The results, obtained after the biomass concentration was doubled and the medium was supplemented with nitrogen and phosphorus, are shown in Fig. 3. It could be observed that these changes benefited the microbial activity, but the TOC removal levels (29%) remained below those achieved at the end of the adaptation period.

In a previous work with leachate from Gramacho, it was not possible to obtain COD removal in the aerobic biological treatment because the COD removal efficiency decreased with increasing leachate concentration in the reactor feeding medium [22]. This indicates that the leachate contains in its composition substances likely to inhibit the biological activity and that the retention of a significant fraction of the organic matter by the membrane (in the concentrate) contributes to reduced recalcitrance. However, part of this recalcitrance still persists in the permeate.

The concentrate after chemical oxidation with Fenton (according to the optimal conditions established) was also submitted to aerobic biodegradation (Fig. 3). It could be observed that the pre-chemical oxidation favored biodegradation since the average TOC removal efficiency values remained around 56%.

4. Conclusions

The use of a 20 kDa UF membrane followed by aerobic biological oxidation of the permeate and chemical/biological oxidation of the concentrate resulted in TOC removal efficiencies for leachate B of 29% and 56%, respectively. The low efficiency achieved is mainly due to the existence of recalcitrant compounds to the biodegradation, despite the retention of a significant fraction of the organic matter by the membrane (in the concentrate). This value is much lower than what could be obtained with the use of chemical oxidation of the leachate, but the costs with oxidants would be lower since volumes 10 times smaller would be treated through oxidation.

The results are only preliminary in relation to what could be done in terms of characterization and segregation of different classes of compounds (biodegradable or toxic/inhibitory, low or high molar mass) present in effluents through the use of processes involving membranes. The employment of the fractionation technique with different cut-off membranes can lead to a deeper knowledge on the wastewater constituents and thus one could define the most appropriate treatment choices more safely.

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