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Influence of recirculation over COD and N-NH_4 removals from landfill leachate by horizontal flow constructed treatment wetland

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ABSTRACT

Treatment of landfill leachate is a challenge due to its complex chemical composition and high recalcitrance and because of high costs for conventional wastewater treatment. In our study, leachate from the *Quitaúna* Landfill, Sao Paulo Metropolitan Region, Brazil, was treated at a laboratory scale with a horizontal subsurface flow constructed treatment wetland (HF-CTW) operating under a recirculation regime. Two units planted with *Heliconia psittacorum* (HP) and *Cyperus papyrus* (CP), and one unplanted control unit were assessed. With a recirculation regime over 21 days, the planted units removed 40% of chemical oxygen demand (COD) while the control unit removed only 29%. True color removal efficiencies were 2, 22, and 23% for the control, HP, and CP HF-CTWs, respectively. The ammonium nitrogen removal efficiencies for a 21-day hydraulic retention time (HRT) were 63–81% for planted units and 72% for the control. The increase of the HRT from 7 to 21 days led to the enhancement of ammonium nitrogen removal but did not affect the COD and total nitrogen removals. This phenomenon is a consequence of leachate's low biodegradability. The present study shows the importance of the HRT and plant presence for landfill leachate treatment using HF-CTWs.

KEYWORDS

Cyperus papyrus; *Heliconia psittacorum*; horizontal flow constructed treatment wetland; hydraulic retention time; nitrification; recirculation

Introduction

Landfill leachate is an important issue related to landfills due to its high potential for environmental impact. Landfill leachate quantity and chemical composition are site-specific and vary in response to waste composition, waste age, landfilling technology, and climate (Bulc 2006; Naveen *et al.* 2017). It also varies throughout the operational life of a landfill (Abbas *et al.* 2009). Leachate contains high concentrations of organic and inorganic compounds, including humic and fulvic acids, ammonium nitrogen, and xenobiotic compounds (Wiszniowski *et al.* 2006; Zhang *et al.* 2016). Several studies have shown high leachate toxicity in terms of both chronic and acute toxic effects on receiving waters (Nivala *et al.* 2007; El-Gohary and Kamel 2016).

Thus, the Brazilian National Environmental Council (CONAMA) Regulation No. 430/2011 establishes that the landfill leachate must be treated to comply with the discharge standard limits in order to prevent degradation of the natural characteristics of receiving water bodies (CONAMA 2011)

Conventional biological wastewater treatment systems traditionally used for municipal wastewater do not satisfactorily remove recalcitrant organic matter and nitrogen from landfill leachates. They also incur high costs making full-scale applications unfeasible at many sites (Abbas *et al.* 2009; El-Gohary and Kamel 2016). A sustainable low-cost solution for on-site treatment is the constructed treatment wetland system (CTW). This technology has been used to treat, with varying degrees of success, various types of wastewater such as domestic sewage, agricultural wastewater, industrial effluents, mine drainage, polluted river waters, and urban run-off (Vymazal 2009).

CTWs are engineered systems designed to simulate physical, chemical, and biological removal mechanisms present in natural wetlands. They consist of vegetation, substrate, microorganisms, and wastewater with construction, operation, and energy consumption costs considerably lower than conventional aerobic wastewater treatment technologies, such as activated sludge (Zhu *et al.* 2014)

The high concentration of organic matter and nitrogen, mainly in ammonium form (NH_4^+-N), in landfill leachates causes toxicological effects (Nivala *et al.* 2007; Zhang *et al.* 2016). Biological transformation and biodegradation are the main pathways for organic matter and nitrogen removal in CTW, including aerobic and anaerobic degradation, ammonification, nitrification, and denitrification (Vymazal and Kröpfelová 2009). The removal routes of organics and nitrogen in wetland systems are critically dependent on environmental and operational factors such as pH, dissolved oxygen

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Table 1. Characterization of raw and prepared leachate of the Quitaúna landfill used in this study.

Parameter	Raw leachate*	Prepared leachate	
		Acclimation period**($n=9$)	Initial recirculation feed
COD, mg.L^{-1}	2,881	660 ± 44	640
Total organic carbon (TOC), mg.L^{-1}	1,122	253 ± 30	–
True color, PtCo	–	1.26 ± 0.31	1,330
Ammonium nitrogen (NH_4^+-N), mg.L^{-1}	2,178	142 ± 29	107
Total nitrogen, mg.L^{-1}	–	200 ± 64	175
Alkalinity, mg.L^{-1}	10,459	–	498
pH	8.08	7.5 ± 0.04	7.5
Conductivity, mS.cm^{-1}	26.5	4.12 ± 0.65	4.08

*based on a single sample; **concentration results are expressed as mean ± SD.

concentration, temperature, and hydraulic retention time (HRT) (Saeed and Sun 2012). Various studies for performance enhancements in CTW have been developed, including different types of configurations, the supply of electron donors, aeration, substrate materials, and operation strategies (Wu *et al.* 2014).

Conventional CTWs treating landfill leachate have reported, in most cases, low-organic matter removal efficiencies due to the recalcitrance and toxicity of organic compounds (Bulc 2006; Vymazal 2009). Recirculation is an operational procedure applied to wastewater treatment systems to increase the HRT and the contact period of pollutants with the microorganisms and plants of the wetland, potentially improving the removal efficiency. A higher HRT may enhance NH_4^+-N oxidation due to the increased period for oxygen diffusion and contact of nitrogen compounds with microorganisms inside the system, thus improving the performance of the CTW (Lavrova and Koumanova 2010; Akinbile *et al.* 2012; Saeed and Sun 2012). The objective of this study was, therefore, to evaluate the performance of a subsurface horizontal flow constructed wetland (HF-CTW) operating in a recirculation regime for the removal of organic matter (i.e. chemical oxygen demand – COD) and nitrogen (as NH_4^+-N and total nitrogen) in landfill leachate.

Materials and methods

Landfill leachate

The leachate was obtained from the *Quitaúna* landfill located in the Metropolitan Region of Sao Paulo, Brazil (-23°23'S and -46°33'W). The landfill encompasses an area of 413,000 m^2 and, since 2001, receives mainly municipal solid wastes. Following collection, the leachate was kept refrigerated at -18 °C until used. Before feeding the CTW, the leachate was kept for three days in a tank with forced aeration (using a small air pump) in order to remove, by stripping, volatile compounds such as fatty acids and toxic concentrations of ammonia nitrogen. Then, in order to offer an appropriate growth environment for the microorganisms and plants (NH_4^+-N under 300 mg/L), it was diluted with tap water (volume ratio 1:5) and its pH adjusted from 8.0 to 7.5 (HCl 1N). The characteristics of the leachate before and after the preparation steps are summarized in Table 1.

HF-CTW experimental set-up and operation

The experiment was carried out in laboratory scale HF-CTW systems consisting of three parallel cells and effluent tanks. The HF-CTWs consisted of polypropylene tanks (0.73 m in length, 0.30 m in width, and 0.14 m in height) totally filled with calcareous gravel (∅5 mm, 49% porosity, Figure 1). Two HF-CTWs were planted in monoculture, at a density of 14 seedlings/m^2, with *Heliconia psittacorum* L. f. (HP) in one chamber, and *Cyperus papyrus* L. (CP) in another chamber. The roots reached the entire gravel bed depth during plantation. One control unit was retained unplanted (Figure 2).

Prior to initiating recirculation, the units were fed continuously with the prepared leachate for 104 days (Table 1) for plant acclimation and biofilm growth. During this period, the dissolved oxygen (DO) concentration inside each HF-CTW was measured using a DO probe (Hanna HI9829) at the following sample points: inside the preparation tank (influent), P1, P2, and the effluent (Figure 2).

Then, a fixed volume of 20 liters as the initial feed of prepared leachate (as shown in Table 1) was pumped and recirculated within the HF-CTWs by continuously reintroducing the effluent into the inlet in a total recirculation regime without new leachate feeding for 21 days at room temperature (18 ± 3 °C). The flow was maintained at 2.25 L/d, corresponding to a hydraulic loading rate of 0.0103 m^3/m^2.d.

Analysis

The influent (initial feed) and effluents were analyzed immediately after sampling, in accordance with Standard Methods protocols (APHA 2008). COD was analyzed using the closed reflux method, alkalinity using the titration method, true color using the spectrophotometric method, NH_4^+-N using the salicylate method, and total nitrogen (TN) using the persulfate digestion method. The colorimetric measurements were done in a spectrophotometer (Hach-DR5000, USA). The pH was analyzed by a pH meter (OrionStar Thermo Fisher Scientific A211, USA).

A single sample (500 mL) of the prepared leachate was collected from the feeding tank during the initial feeding. Single samples (250 mL) from each HF-CTW were collected in the outlet after 1, 7, 14, and 21 days of operation. Treatment efficiency calculations were based on the

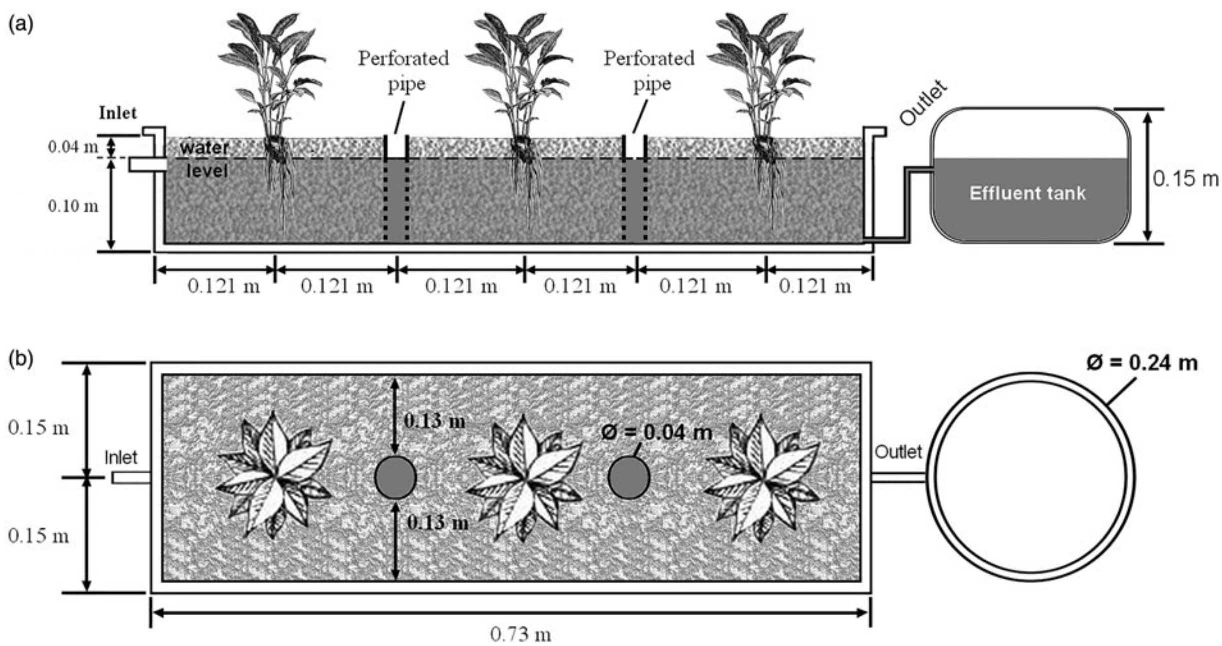

Figure 1. Schematic diagram of lab-scale subsurface horizontal flow constructed treatment wetland: (a) cross section and (b) plan view (1.5 column fitting image).

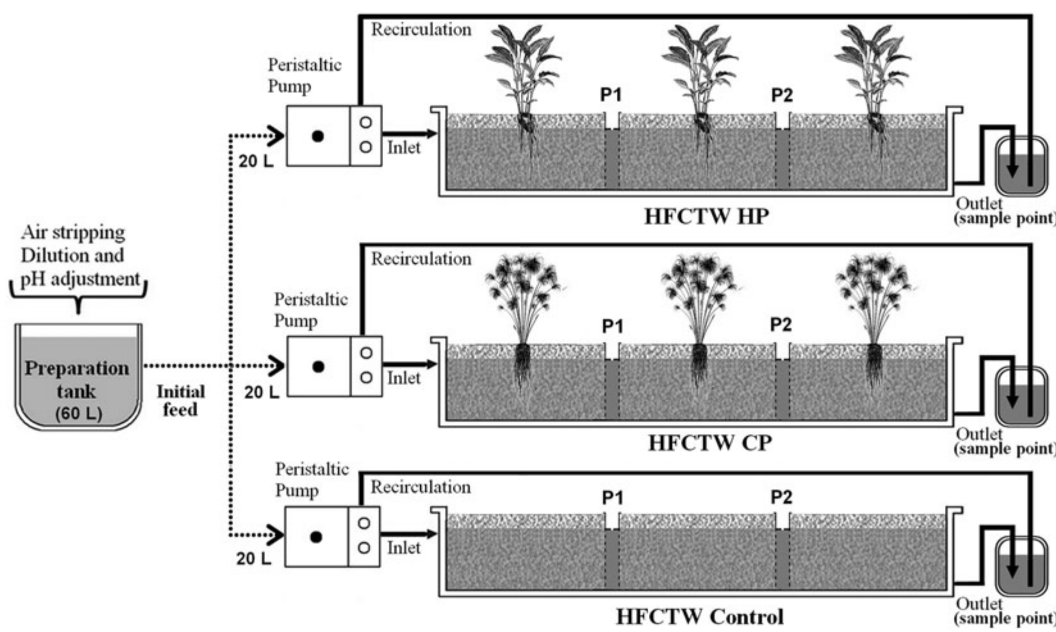

Figure 2. Lab-scale subsurface horizontal flow constructed treatment wetland system (1.5 column fitting image).

difference between the influent (initial feed) and effluent concentrations of the parameters evaluated.

Results and discussion

Organic matter removal

The COD values of the treated wastewater in HF-CTW during the 21 days of operation are presented in Figure 3. The data shows the influence of the recirculation and plant presence on the treatment performance of the system.

The COD effluent concentration decreased over time. After 21 days of operation, the COD concentration of the control wetland dropped from 641 to 458 mg/L, which corresponds to 29% removal efficiency. Both planted units had a COD removal efficiency of approximately 40%. All units showed a linear reduction of COD concentration after day 7 (R^2 ranging between 0.92 and 0.99), with higher removal rates for the planted units.

The increase in efficiency of the planted HF-CTWs might be a result of higher microbial activity in the plant's root zone (Ramamoorthy and Kalaivani 2011). However, in spite of an HRT of 21 days, the overall COD removal efficiencies were relatively low compared to HF-CTW treating other forms of biodegradable wastes, such as domestic wastewater. It has been reported that a significant proportion of recalcitrant material in landfill leachate is comprised of humic acids, phenolic, alicyclic compounds, and phosphate ester,

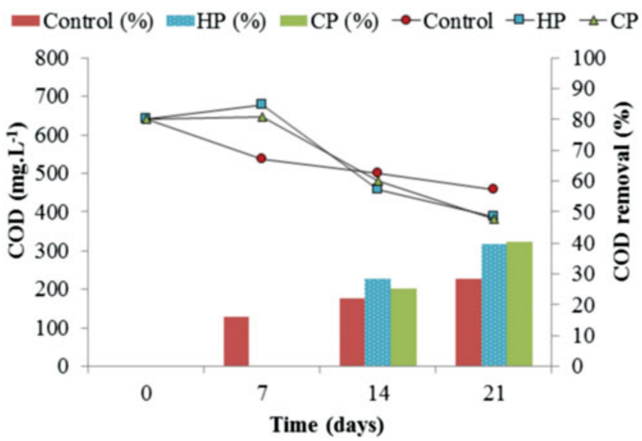

Figure 3. COD concentration and removal efficiency during the 21 days of recirculation in the HF-CTW for control and units planted with *Heliconia psittacorum* (HP) and *Cyperus papyrus* (CP) (single column fitting image/black and white in print).

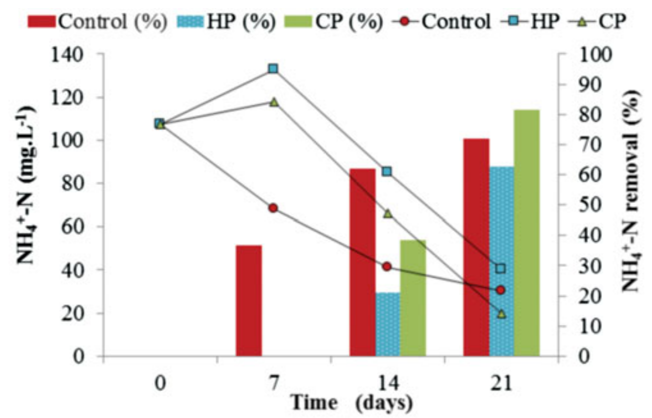

Figure 4. NH_4^+-N concentration and removal efficiency during the 21 days of recirculation in the HF-CTW for control and units planted with *Heliconia psittacorum* (HP) and *Cyperus papyrus* (CP) (single column fitting image/black and white in print).

which are resistant to biological treatment, thus resulting in low-organic matter removal efficiency (Vymazal 2009; El-Gohary and Kamel 2016).

In our study, after 21 days, color removal efficiencies of 2, 22, and 23% were achieved for the control, HP, and CP HF-CTW, respectively. The color of the landfill leachate is primarily caused by dissolved organic compounds in the form of recalcitrant compounds such as humic and fulvic acids (Akinbile *et al.* 2012). The poor color removal can thus be related to the low COD removal efficiencies. It has been reported that the recalcitrance of organic contaminants is an important issue limiting COD removal in CTW treating landfill leachate (Vymazal 2009).

Nitrogen removal

Nitrogen is an important component of landfill leachates due to its typical high concentration. NH_4^+-N in leachate increases during landfill age due to continuous protein degradation (Kjeldsen et al. 2002; Abbas *et al.* 2009; El-Gohary and Kamel 2016). Consequently, leachate must receive treatment to reduce nitrogen levels prior to discharge in order to comply with the Brazilian Regulations.

The NH_4^+-N influent concentration in the prepared leachate was 107 mg/L and all HF-CTW units performed some level of NH_4^+-N removal. The effluent concentrations of the control unit decreased linearly throughout the 21 days of operation (R^2 of 0.94). However, for the CP and HP units, a linear decrease started only after 7 days (R^2 of 0.99) for both units (Figure 4). For HRT of 7 days, only the control unit showed removal, i.e. 37%. The higher effluent concentrations in the HP and CP HF-CTW compared to control unit is most probably due to the more intense ammonification process turning the effluent more concentrated. Similarly, it was observed in a recirculating CTW that, within the first 24 hours, the degradation of organic compounds provided an additional source of NH_4 in the system thus affecting its removal efficiency (Bialowiec *et al.* 2012).

After 14 days, the control HF-CTW showed a removal efficiency of 62%, while the HP and CP HF-CTW units showed removal efficiencies of 39 and 21%, respectively. At the end of 21 days of operation, the NH_4^+-N removal efficiencies (relative to the initial influent concentration) were 72, 63, and 81% for the control, HP and CP HF-CTW, respectively (Figure 4).

In our study, all units showed a positive correlation between the decrease of NH_4^+-N and alkalinity concentrations (Figure 5). The decrease of ammonium concentration in wetlands is achieved through nitrification and root plant uptake. Nitrification is considered the main route of NH_4^+ removal (Saeed and Sun 2012) and 7.1 mg.$CaCO_3$/mgNH_4^+-N are consumed by both nitrifiers cell synthesis and ammonia oxidation (Ge *et al.* 2015). This observation suggests nitrification as the main process for NH_4^+-N removal in this study.

The results also showed an influence of the HRT over NH_4^+-N removal, increasing its efficiency when a higher HRT were applied to the wetlands. The increase in efficiency due to the increase in HRT is consistent with results from studies employing HF-CTWs.

In the treatment of synthetic wastewater, a high removal efficiency of NH_4^+-N was observed when the HRT was over 8 days with additional improvements after 20 days. At these HRTs, removal efficiencies ranged from 63 to 96%, respectively, have been reported (Akratos and Tsihrintzis 2007). The treatment of landfill leachate in a recirculating HF-CTW system achieved improved removal of NH_4^+-N from 30 to 54% by increasing the HRT from 1 to 21 days (Akinbile *et al.* 2012).

Taking into consideration the influent COD and NH_4^+-N concentrations and 21 days HRT, the theoretical total dissolved oxygen necessary for their oxidation is 22.5 g, representing a demand of 4.9 g/m^2.d. The principal oxygen supply mechanisms in HF-CTW are reported to be the oxygen diffusion from the atmosphere and radial oxygen loss by plants, with rates varying from 0.3 to 11.6 g/m^2.d and 0.1 to 2.2 g/m^2.d, respectively (Liu *et al.* 2016).

The average DO concentrations inside each unit are listed in Table 2. For the control, HP and CP, the values ranged

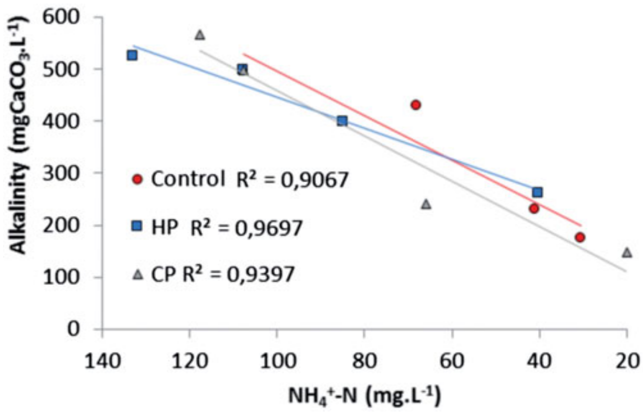

Figure 5. Correlation between NH_4^+-N concentration and alkalinity in the HF-CTW for control and units planted with *Heliconia psittacorum* (HP) and *Cyperus papyrus* (CP) (single column fitting image/black and white in print).

Table 2. Dissolved oxygen concentrations (mg/L) in the inlet, outlet and inside the units control, CP and HP.

	Dissolved oxygen (mg/L, $n = 16$)*			
HF-CTW	Influent	P1	P2	Effluent
Control	1.53 ± 0.92	1.78 ± 1.03	1.82 ± 1.05	1.5 ± 0.69
HP		1.53 ± 0.92	1.44 ± 0.95	1.46 ± 0.78
CP		1.59 ± 0.83	1.77 ± 1.07	1.47 ± 0.6

*concentration results are expressed as mean ± SD.

from 0.57 to 3.45 mg/L, 0.21 to 3.14 mg/L, and 0.3 to 3.92 mg/L, respectively. Although the mean values for all CTW were above 1.4 mg/L, the low DO levels (below 1 mg/L) most probably influenced the overall performance of HF-CTW.

The nitrification occurrence in conventional non-aerated HF-CTW is typically low. This, in part, is due to the competition between nitrifiers and heterotrophic microorganisms for DO and space (Bulc 2006; Vymazal and Kröpfelová 2009; Giustinianovich *et al.* 2016). The oxygen transfer rates are low in HF-CTWs (Tyroller *et al.* 2010) so, due to the slow specific growth rate inherent to nitrifiers, the presence of a carbon source results in a dominance of heterotrophs, causing the depletion of available oxygen and ultimately inhibiting the nitrification process (Wang *et al.* 2016).

Several studies reported artificial mechanisms to improve oxygen transport, such as the artificial aeration aiming at NH_4^+-N removal in CTW (Nivala *et al.* 2007; Wu *et al.* 2014). However, nitrification can also be achieved by the contribution of atmospheric oxygen diffusion, thus avoiding increased operational costs associated with externally forced aeration. It was observed in a recirculating CTW with no artificial aeration that the redox potential increased continuously during 14 days up to 67 mV, resulting in a more aerobically active biofilm in the system. (Bialowiec *et al.* 2012).

The removal of NH_4^+-N in the aerobic surface layer creates a gradient of the ammonia concentration, promoting an upward transport by NH_4^+-N diffusion from the lower layers (anaerobic) to the aerobic zone (Chandra and Kumar 2015). The limiting factor for nitrification in HF-CTWs is not only associated with DO in raw wastewater and plant transport but also with atmospheric O_2 diffusion at the surface of the CTW (Wu *et al.* 2001).

This is particularly important in CTWs with a shallow depth, as the one used in our study. It was found that water level is an important variable controlling the efficiency of subsurface CTWs. It was reported that an HF-CTW water depth of 0.27 m was more effective for the removal of NH_4^+-N (up to 66%) than HF-CTW with a 0.5 m water depth (Garcia *et al.* 2005). In tropical or subtropical regions, microbial communities from shallow CTWs are more related to aerobic microorganisms, such as *Nitrospira defluvii*, in accordance with the higher values of redox potential (Morató *et al.* 2014). Conversely, nitrifiers are highly sensitive to inhibition from a range of organic and inorganic compounds, such as anionic surfactants and chlorinated organic compounds (Rittmann and McCarty 2001). In our study, the electrical conductivity changed from 4000 to 5710 μS.cm^{-1} as expected for this type of wastewater. It was also observed that high-salinity levels result in the inhibition of nitrification (Rene *et al.* 2008). In high-salinity environments, bacteria need to regulate the osmolality of the cytoplasm. However, it is costly in terms of bioenergetics of their metabolism. This is particularly critical for nitrifiers because they gain small amounts of energy from their dissimilatory metabolisms compared to heterotrophs (Gonzalez-Silva *et al.* 2016).

In addition, the temperature is an important variable in nitrification reaction since it greatly affects the growth rates of both ammonia-oxidizing bacteria and nitrite-oxidizing bacteria (Okabe *et al.* 2011). Thus, nitrification treatment systems in tropical regions, such as in Brazil, have enhanced performance compared to regions of lower temperature.

Regarding TN, after 21 days, a slight decrease in concentration was observed from 175 to 167 mg/L and 172 mg/L for the CP and HP HF-CTW, respectively. For the control unit, there was no TN removal. It is well established that carbon availability and absence of oxygen play an important role in denitrification (Lavrova and Koumanova 2010). Denitrification takes place only in anoxic zones as dissolved oxygen suppresses the enzyme system required for this process (Chandra and Kumar 2015).

As mentioned, denitrification was not observed possibly due to the lack of readily biodegradable organic matter in the leachate, which is very much necessary for denitrifiers. Also, strictly anoxic zones could not be confirmed in the HF-CTWs. Denitrification is the major mechanism of TN removal in CTW, thus the lack of easily biodegradable organic matter and the presence of DO may hinder the TN removal efficiency (Saeed and Sun 2012).

It has been observed that denitrification rates in CTWs are significantly higher when sodium acetate is supplied as an external carbon source (Kozub and Liehr 1999), corroborating the effect of the availability of easily degradable organic matter on the reduction of nitrate. In this sense, higher BOD:N ratios may allow higher nitrate (NO_3) removal in CTWs, due to the presence of a carbon source supplement that acts as a major electron donor for denitrification (Wang *et al.* 2016).

In order to overcome recalcitrance, the integration of other types of technologies or processes, such as the advanced oxidation process (AOP) associated with biological treatment could be an alternative leading to improved COD and TN removals by increasing the biodegradability of the leachate prior to the biological treatment process (Abbas *et al.* 2009; Hilles *et al.* 2015). Ozone combined with H_2O_2 (Cortez *et al.* 2010) and electro-Fenton method assisted by chemical coagulation (Lin and Chang 2000) were found to be highly efficient in removing a large amount of recalcitrant organic and inorganic compounds in the leachate, improving the biological treatment efficiency. However, for each technology to be analyzed, besides performance, cost-benefits and trade-offs should also be taken into consideration in the decision-making process.

Thus, the combination of CTW technology with advanced treatment processes could provide new possibilities to increase nitrogen removal, as it may increase the biodegradability of the recalcitrant organic matter to be used as electron donors for denitrification or by post-treatment in order to alternatively remove nitrogen when biological treatment is inhibited (Hilles *et al.* 2015).

Conclusions

The influence of effluent recirculation in a lab-scale HF-CTW for landfill leachate treatment was assessed. The results underscored the influence of HRT on NH_4^+-N removal efficiency. In general, the literature has reported that HF-CTW is largely anaerobic and thus not commonly used for ammonium removal. Although in our study, nitrification was achieved in the total recirculation regime (HRT 21 days). The recalcitrance of organic matter affected the COD removal efficiency to levels below 40%, even at 21 days of HRT. The presence of plants, compared to the control without plants, increased the COD removal efficiency in the system. We conclude that HF-CTW is a promising sustainable technological alternative for landfill leachate treatment, as it can achieve up to 81% of NH_4^+-N removal for diluted leachate. Considering that there is no need to add pumps to supply O_2 as the conventional aerobic system requires, the overall costs are much lower. However, due to the high HRT (>14 days) necessary to achieve high treatment performance, it might require large areas. The system presents a significant pollutant removal capacity and simple operation, low-cost and esthetic potential. Yet, additional studies are needed to identify ways to increase organic matter biodegradability, oxygen transfer, and operational optimization for this type of wastewater in the CTW for a higher treatment performance. Future studies collecting DO measurements from depth-integrated points within the HF-CTW media bed are recommended for a better process control allowing deeper comprehension of nitrogen and organic matter oxidation enhancement.

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