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Biological systems combined for the treatment of coffee processing wastewater: II - Removal of nutrients and phenolic compounds

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ABSTRACT. Three treatment systems consisting of anaerobic filters with upward flow followed by constructed wetland systems (CW) were evaluated for the removal of nutrients and phenolic compounds, in the treatment of coffee processing wastewater (WCP) in Viçosa, Minas Gerais State. Filters were made of PVC (1.5 m high and 0.35 m diameter) filled with gravel # 2 and CW were made of wooden boxes (1.5 m long, 0.4 m high and 0.5 m wide) sealed by HDPE geomembrane and filled with gravel 'zero'. The WCP had the pH adjusted with lime to values close to 7.0 and the nutrient concentration changed to obtain a BOD/N/P ratio of 100/5/1. As a result, hydraulic retention times longer than 160 h in all treatment systems during phase III had not promoted greater efficiencies for removal compared with the phase I, because the recovery time of the systems was inadequate. The F_1+CW_1 system that received the lowest organic load, showed a satisfactory performance with regard to the removal of nutrients (above 50%).

Keywords: anaerobic reactor, constructed wetland systems, phenols, nitrogen, phosphorus, coffee.

Sistemas biológicos conjugados para o tratamento das águas do processamento dos frutos do cafeeiro: II - Remoção de nutrientes e compostos fenólicos

RESUMO. Foram avaliados três sistemas de tratamento, compostos por filtros anaeróbios com escoamento ascendente, seguidos por sistemas alagados construídos (SACs), em relação à capacidade de remoção de nutrientes e compostos fenólicos, no tratamento da água residuária do processamento dos frutos do cafeeiro (ARC) em Viçosa, Estado de Minas Gerais. Os filtros foram confeccionados em PVC (1,5 m de altura e 0,35 m de diâmetro) e preenchidos com brita número 2 e os SACs foram constituídos por caixas de madeira (1,5 m de comprimento, 0,4 m de altura e 0,5 m de largura) impermeabilizados por geomembrana de PEAD e preenchidos com brita número 'zero'. A ARC teve o pH corrigido com cal até valores próximos a 7,0 e a concentração de nutrientes alterada de forma a se obter uma relação DBO/N/P igual a 100/5/1. Como resultado, observou-se que os tempos de detenção hidráulica superiores a 160 h, a que foram submetidos todos os sistemas de tratamento, durante a fase III, não proporcionaram maiores eficiências de remoção, quando comparadas à fase I, pois o tempo de recuperação dos sistemas mostrou-se insuficiente. O sistema F_1+SAC_1, que recebeu a menor carga orgânica, apresentou desempenho satisfatório no que se refere à remoção de nitrogênio e fósforo, sendo a eficiência maior que 50%.

Palavras-chave: reator anaeróbio, sistema alagado construído, fenóis, nitrogênio, fósforo, café.

Introduction

Brazil is the largest producer of coffee beans, but to obtain good returns with the activity is necessary to improve the quality of the beverage provided by the product, without raising the costs of processing and drying of beans. The wet processing of coffee cherries is an alternative, however generates a large amount of wastewater (WCP), rich in suspended organic matter, organic and inorganic compounds in solution, with high polluting potential (MATOS et al., 2007) which must be necessarily treated before its release into receiver water bodies. Biological systems have been studied for the treatment of these wastewaters. Nevertheless, the WCP has an unbalance between the amount of organic matter and nutrients (FIA et al., 2007), resembling the industrial wastewaters. In this way, for improving the performance of biological treatment systems, it is necessary to perform the nutritional correction of these waters prior to treatment (FERNANDEZ; FORSTER, 1993; METCALF & EDDY, 2003).

Besides these characteristics, the WCP has phenolic compounds (FIA et al., 2010b), considered resistant to biodegradation in the environment that can reduce the biological treatment efficiency. Part of these phenolic compounds can be removed by adding limestone, forming calcium precipitates (SARASA et al., 1998).

Even though, anerobic systems have proven to be able to degrade these compounds (BOLAÑOS et al., 2001). According to Henze and Harremöes (1983), among anaerobic reactors, those of fixed bed are usually more resistant to toxic effects of phenols by having a fixed the layer of support material, which gives them low susceptibility to biomass loss.

Given this, the present study aimed to evaluate the performance of anaerobic filters followed by constructed wetland systems (CW), subjected to different organic loads, in relation to the removal of nutrients and phenolic compounds of coffee processing wastewater in Viçosa, Minas Gerais State.

Material and methods

The experiment was developed in the Department of Agricultural Engineering, Federal University of Viçosa, Viçosa, Minas Gerais State. The seat of the municipality is at 20° 45' S and 42° 52' W, and mean altitude of 650 m above sea level. According to Köppen classification, the climate is Cwa, altitude tropical, with rainy summer and dry winter.

Three anaerobic filters were constructed using segments of PVC pipe, 0.35 diameter and 1.5 long, with total capacity of 139.5 L. These units were filled with support medium (crushed granite-gnaisse # 2), forming 1.0m-columns on the false bottom, which was 0.2m from the bottom. As inoculum, we used 50 L of sludge from the anaerobic tank of swine wastewater treatment of the UFV.

The effluents of the three filters (F_1, F_2 and F_3) were respectively discharged into three CW (CW_1, CW_2 and CW_3) of horizontal subsurface flow, constructed on a pilot scale, made up by boxes (0.4 m high x 0.5 m wide x 1.5 m long) of pine wood, sealed by high density polyethylene (HDPE) membrane, positioned on the soil in 0.01 m m^{-1} slope. As a support medium it was used gravel 'zero' (diameter D-60 = 7.0 mm, and initial void volume of 0.491 m^3 m^{-3}). CW were filled with gravel up to the height of 0.35 m, allowing a 0.05 m free edge (unsaturated), once the water level was kept at 0.30 m. In each CW, we planted on the first 0.75 m of the bed, *Alternanthera phyloxeroides*, and on the last 0.75 m, *Typha* sp. (MATOS et al., 2010).

The system worked at room temperature and evaluated for 130 days, between June and October, divided into three phases of operation (42, 46 and 42 days, respectively). In the starting period of the system, filters were simultaneously fed with the same influent (diluted WCP, where the pH was corrected with hydrated lime to values close to 7.0).

From the second phase, there was a differentiated increase in organic load applied in the filters, using as reference the COD. The application of the WCP into F_1 and F_2 was done in diluted form, in the proportions of 50 and 75% (v v^{-1}), respectively, whereas the F_3 received undiluted WCP, holding constant the hydraulic retention time (HRT). Besides the pH correction, the WCP underwent a nutritional correction with urea and simple superphosphate, in order to achieve the ratio 100/5/1 between BOD, nitrogen and phosphorus (DBO/N/P) (METCALF & EDDY, 2003). In the third phase, similar proportions of WCP were maintained in the feeding the three filters, but increased about twice the HRT of filters and CW.

Mean values and standard deviations of operational characteristics of anaerobic filters and CW are listed in the Table 1.

Table 1. Operational characteristics of anaerobic filters and constructed wetland systems.

Treatment units	Q	HRT	OLR	OLR_A
		Phase I		
F_1	0.052 ± 0.018	33.5 ± 10.9	1.49 ± 0.61	-
CW_1	0.048 ± 0.005	58.7 ± 7.4	-	1058 ± 586
F_2	0.053 ± 0.020	32.3 ± 9.1	1.82 ± 0.73	-
CW_2	0.049 ± 0.004	57.6 ± 5.0	-	805 ± 280
F_3	0.049 ± 0.023	37.1 ± 12.2	1.77 ± 0.95	-
CW_3	0.049 ± 0.008	58.6 ± 9.9	-	798 ± 409
		Phase II		
F_1	0.050 ± 0.017	35.1 ± 12.8	5.60 ± 2.26	-
CW_1	0.056 ± 0.018	54.4 ± 13.7	-	3597 ± 1165
F_2	0.051 ± 0.016	34.2 ± 11.5	8.30 ± 2.55	-
CW_2	0.048 ±0.010	61.0 ± 13.4	-	6006 ± 1635
F_3	0.047 ± 0.015	37.2 ± 12.2	12.99 ± 6.84	-
CW_3	0.050 ± 0.012	59.5 ± 14.2	-	9092 ± 4559
		Fase III		
F_1	0.031 ± 0.011	56.4 ± 18.4	1.69±0.57	-
CW_1	0.027 ± 0.007	111.8 ± 27.0	-	1507 ± 213
F_2	0.029 ± 0.007	56.9 ± 13.7	3.24±0.88	-
CW_2	0.023 ± 0.005	126.7 ± 24.1	-	22579 ± 793
F_3	0.032 ± 0.011	54.1 ± 16.1	3.86±1.27	-
CW_3	0.025 ± 0.005	114.7 ± 22.5	-	3043 ± 1076

Q – flow (m^3 d^{-1}); HRT - hydraulic retention time (h); OLR – volumetric organic load rate (kg m^{-3} day^{-1} COD); OLR_A – organic loading rate based on the surface area (kg ha^{-1} day^{-1} COD). Five samples were collected in each phase to calculate the OLR and OLR_A, the Q and HRT were monitored daily.

Once a week, influent and effluent samples were taken from filters and effluents from CW to evaluate the pH, by potentiometric method, total phosphorus concentration (P) by spectrophotometry (APHA et al., 2005), exchangeable calcium (Ca) by titration (GRASSHOFF et al., 1983), total nitrogen (N) by the semi-micro Kjeldahl method with addition of salicylic acid, adapted from Kiehl (1985), and total phenolic compounds (F_T) according to Folin and Ciocalteu (1927).

For the statistical analysis we considered a completely randomized design with three systems (F+CW) and three phases and with the number of repetitions equal to the number of samplings. Then an analysis of variance and mean values of removal efficiencies were compared using a Tukey's test, at 5% probability. For this we used the statistical software SAEG® (RIBEIRO JÚNIOR, 2001).

Results and discussion

The concentrations of N have increased in phases II and II, and of P, in phase II, due to nutritional correction of WCP with commercial fertilizers based on urea and simple superphosphate (Table 2). In the phase II, even adding simple superphosphate, there was a reduction in P concentration. The greater removal efficiency of P was probably associated with the lime, added to correct the pH influent to the filter, with the P in the influent forming calcium phosphate, a low soluble salt, as observed by Tanaka et al. (2007). However part of the removal can also be attributed to the absorption by microorganisms, sedimentation, adsorption to the support material and precipitation occurring in the system (REDDY; DELAUNE, 2008).

Despite the different removal efficiencies on nitrogen, phosphorus and phenolic compounds, no significant difference ($p > 0.05$) was detected between treatment systems during the three phases (Table 3), except for nitrogen during the third phase.

Higher mean concentrations of N, P, and Ca in effluents, relative to the influents, during the phase I, can be justified by the high concentration of these elements in the sludge (pig farming) used as inoculum to start the filters. With the advance of the experimental period, satisfactory reductions were observed in the concentration of P, but the removal of N remained low. During the second phase, the F_2+CW_2 system presented, on average, a negative removal efficiency of nitrogen, i.e., during samplings we obtained effluent values higher than influent, which can be related to the sudden increase of flow and consequent washout of sludge. Moreover, according to Parawira et al. (2005), it has been mentioned low removal efficiency of nitrogen and phosphorus in the anaerobic digestion in anaerobic reactors, once this system does not produce large amounts of sludge.

Table 2. Mean values and standard deviation of pH, nitrogen (N), phosphorus (P), calcium (Ca) and total phenolic compounds (F_T) of influent (C_1, C_2 and C_3) and of effluent from anaerobic filters (F_1, F_2 and F_3) of constructed wetland systems (CW_1, CW_2 and CW_3).

Treatment units	pH	N	P	Ca	F_T
Phase I					
C_1	6.69 ± 0.43	24 ± 11	3.7 ± 0.6	565 ± 294	14.7 ± 2.2
F_1	7.05 ± 0.49	24 ± 15	4.9 ± 2.6	523 ± 186	8.6 ± 5.2
CW_1	7.21 ± 0.38	11 ± 4	1.5 ± 1.0	491 ± 178	3.3 ± 1.2
C_2	6.80 ± 0.45	24 ± 12	3.6 ± 0.7	577 ± 457	13.2 ± 4.5
F_2	7.07 ± 0.42	31 ± 21	5.3 ± 1.3	638 ± 310	7.0 ± 6.2
CW_2	7.25 ± 0.31	13 ± 6	2.4 ± 1.3	527 ± 204	4.3 ± 2.5
C_3	6.81 ± 0.59	23 ± 7	2.8 ± 1.4	512 ± 368	12.2 ± 3.8
F_3	6.98 ± 0.37	29 ± 15	4.6 ± 2.8	521 ± 317	5.0 ± 4.3
CW_3	7.36 ± 0.59	15 ± 5	1.8 ± 1.3	546 ± 256	4.2 ± 2.7
Phase II					
C_1	6.60 ± 0.55	296 ± 29	9.0 ± 6.4	1.322 ± 236	30.1 ± 26.6
F_1	6.73 ± 0.54	272 ± 26	5.9 ± 1.9	973 ± 319	27.1 ± 23.2
CW_1	7.39 ± 0.18	235 ± 15	3.9 ± 0.6	712 ± 243	5.6 ± 0.6
C_2	6.91 ± 0.56	365 ± 90	12.3 ± 7.1	1.516 ± 379	49.4 ± 41.5
F_2	6.38 ± 0.12	351 ± 59	6.6 ± 1.4	1.296 ± 258	44.8 ± 34.4
CW_2	6.95 ± 0.19	391 ± 91	4.3 ± 1.1	1.330 ± 538	14.1 ± 3.0
C_3	6.84 ± 0.62	539 ± 137	13.0 ± 5.5	2.296 ± 613	65.0 ± 42.1
F_3	6.30 ± 0.19	469 ± 55	11.1 ± 4.4	1.902 ± 783	65.2 ± 49.9
CW_3	6.49 ± 0.51	493 ± 159	6.0 ± 2.3	1.202 ± 557	23.0 ± 3.2
Phase III					
C_1	7.45 ± 0.61	333 ± 19	2.6 ± 1.1	746 ± 187	14.0 ± 4.6
F_1	7.74 ± 0.12	319 ± 18	1.6 ± 1.3	374 ± 161	9.6 ± 7.0
CW_1	7.71 ± 0.26	133 ± 75	1.5 ± 1.2	393 ± 120	9.2 ± 4.2
C_2	7.82 ± 0.17	428 ± 81	2.2 ± 1.5	1.748 ± 396	15.1 ± 5.5
F_2	7.35 ± 0.37	438 ± 54	1.3 ± 1.5	1.059 ± 201	13.2 ± 5.2
CW_2	7.84 ± 0.11	244 ± 132	1.6 ± 1.6	479 ± 137	12.5 ± 4.8
C_3	7.87 ± 0.22	536 ± 98	2.7 ± 1.7	1.890 ± 428	20.8 ± 7.4
F_3	7.13 ± 0.51	469 ± 54	2.7 ± 2.5	1.358 ± 142	17.3 ± 10.1
CW_3	7.50 ± 0.23	368 ± 200	2.6 ± 1.6	993 ± 288	13.4 ± 6.6

N, P, and Ca in mg L^{-1}; FT in mg L^{-1} of tannic acid.

Even though, Prado and Campos (2008) reached maximum removal efficiencies of N and P equal to 35 and 61% for concentrations ranging from 15 to 35 and from 38 to 351 mg L^{-1} of N and P in UASB reactor operated with HRT between 8 and 70 hours, used for treating coffee processing wastewater.

Table 3. Mean removal efficiency (%) of nitrogen (N), phosphorus (P) and phenolic compounds (F_T) by the treatment systems formed by anaerobic filters followed by constructed wetland systems (F+CW), in the three phases of system operation.

Variables	F_1+CW_1			F_2+CW_2			F_3+CW_3		
	I	II	III	I	II	III	I	II	III
N	34A	20A	50A	33A	-17A	33AB	31A	1A	23B
P	62A	38A	52A	30A	49A	37A	27A	43A	35A
F_T	77A	73A	37A	64A	57A	11A	63A	56A	27A

For the same variable, within the column of a same phase, mean values with the same upper case are not significantly different by Tukey's test at 5% probability.

In the treatment systems there was a downward trend for nitrogen removal efficiency with increased applied load. Nevertheless, there was increasing trend in removal efficiency in phase III, especially in the F_1 + CW_1, whose efficiency exceeded that obtained in the phase I. Once the wastewater passes through anaerobic filters, before being released into the CW, there is an upward trend in the degradation of organic molecules containing nitrogen, which were converted into ammonium, a form of easier degradation. This probably favored the nitrogen removal by plants in CW_1 and CW_2, and to a lesser extent in the CW_3, which had no vegetation given its senescence (FIA et al., 2008). The temperature may also have affected the nitrogen removal in CW, since the experiment occurred during the winter. With low temperatures, there is a reduction in plant and microbial activity. Kuschk et al. (2003) found efficiencies of 53 and 11% in removal of nitrogen from wastewater when worked in wetland systems in the summer and winter, respectively, regardless the organic load. A similar result was also registered by Poach et al. (2004).

When evaluated the performance of CW working with sugar cane processing wastewater with HRT of 60 and 120 hours, Olguín et al. (2008) have obtained removal efficiencies of N between 73 and 76%, for the mean influent concentration of 26 mg L^{-1}. The same authors achieved a negative efficiency for P removal, with an influent mean concentration of 2 mg L^{-1}.

The mean removal efficiencies of nitrogen of the present study can be considered satisfactory, except for the phase II. In agreement with Von Sperling (2005), the mean efficiency of nitrogen removal in constructed wetland systems is below 60%. Also, according with the same author, the removal of phosphorus of CW used in domestic sewage treatment is less than 35%. In general, greater removals are related to lower loads applied (GOTTSCHALL et al., 2007), as observed herein.

During the phase II, an increase was verified in phenolic compounds, both in the influent and effluent from filters, due to the higher concentration of WCP used in the experiment, and to the greater organic load applied. In consequence, there was a reduction in the removal efficiencies of this compound. This is possibly associated with the removal of these compounds promote by increased pH in the WCP, which caused the settlement of particulate organic matter and consequent removal of phenolic compounds, similarly to results of Tsonis et al. (1989). In the presence of calcium ions, some intermediate products of phenol, including maleic acid, oxalic acid, and high molecular weight products, may bind to calcium ions and form insoluble compounds, which precipitate (HSU et al., 2007). Aktas et al. (2001) achieved an efficiency between 63 and 73% in the removal of polyphenols, when added lime to the wastewater of olive processing, rich in phenolic compounds.

Prado and Campos (2008) observed a maximum removal efficiency of phenolic compounds equal to 70%, for concentrations varying from 30 to 380 mg L^{-1}, in UASB operated with HRT between 8 and 70 hours, used in the treatment of WCP. Borges et al. (2009), treating WCP in horizontal fixed bed reactors with influent concentration at 7 mg L^{-1} of phenolic compounds and HRT of approximately 10 hours, have achieved around 25% efficiency (estimated) in removing these chemicals. Bruno and Oliveira (2008) verified mean efficiencies of 90% in the removal of total phenols, in treatment system of WCP made up by two stages UASB, at bench scale for a mean influent concentration of 97% mg L^{-1} and HRT of 223 hours. Grismer et al. (2003) treated wine production wastewater and registered removals from 48 to 78% of phenolic compounds, using constructed wetlands. The authors applied mean rates of 17 kg ha^{-1} day^{-1} (estimated) of phenolic compounds, quantified as tannins, being the HRT of 5.5 days.

During the phase III, removal efficiencies of phenolic compounds were relatively low. It is believed that there was no time to recuperate the system as for the recovery of microbiota degrading these compounds, probably reduced in the phase II.

Phenolic compounds can be aerobic- and anaerobically degraded. However during the first phase (Table 2) phenolic compounds were mostly degraded in anaerobic filters compared with CW.

Similar fact was observed by Bruno and Oliveira (2008), who observed that most of phenolic compounds (80%) were removed in the first stage of the UASB reactor with HRT of 149 hours. During the phase III, with increased concentrations, there was a higher efficiency of CW in relation to filters, which can be due to the toxicity presented by microorganisms to phenolic compounds, even under relatively low concentrations, such as 10 mg L^{-1} (BOLAÑOS et al., 2001).

Mean removal efficiencies of phenolic compounds obtained in systems that received the lowest organic loads were similar to obtained by Fia et al. (2010a). These authors registered about 70% of removal in anaerobic filters followed by CW cultivated with oats and ryegrass in the treatment of WCP. The WCP treated by Fia et al. (2010a) had lower concentrations of F_T, but treatment systems also presented lower HRT.

Comparing the three systems, the F_1+CW_1 had the best overall performance, in the three operational phases, highlighting the phase III, where the load of nutrients and phenolic compounds was lower and the biomass had already be adjusted to chemical conditions of the WCP. Nevertheless, it is not allowed the wastewater dilution to favor its treatment, and to maintain the load applied in the F_1+CW_1, it is required to extend the HRT.

Conclusion

The increase in organic loading rates and consequently of nutrients and phenolic compounds, applied to the systems has promoted a reduction in the removal efficiency of compounds evaluated in the three systems.

The hydraulic retention times exceeding 160 h in all treatment systems during the phase II have not promoted greater removal efficiencies compared with the phase I, because the recovery time of systems was insufficient.

The F_1+CW_1 system, which received the lowest organic and nutrients load, has presented a satisfactory performance as for the removal of nitrogen and phosphorus, on average higher than 50%.

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