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Evaluation of endoglucanase and β -glucosidase production by bacteria and yeasts isolated from an eucalyptus plantation in the cerrado of Minas Gerais

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ABSTRACT

The current global environmental and economic scenario is intrinsically related to the increase in fossil fuel consumption caused by technological development and world population growth. Thus, it is necessary to search for renewable sources of biofuel in an attempt to mitigate the effects of fossil fuels on the environment and the lack of these non-renewable fuels. The use of lignocellulosic biomass, an abundant and renewable resource in Brazilian regions, has contributed successfully to new research and technologies for second-generation ethanol production. The conversion of lignocellulosic biomass into fermentable sugars requires the use of cellulolytic enzymes produced by microorganisms found in the microbiota. This work evaluated the production of endoglucanase and β -glucosidase in a liquid medium containing carboxymethylcellulose by five microorganisms (bacteria and yeasts) from the bank of 348 isolates from eucalyptus soils in the Cerrado Mineiro. Microorganisms with the following enzymatic activity indexes were selected: IM1-74 (22), IM25-9 (5.33), IM32-90 (7.33), IM1-5 (10.33) and IM32-91 (5.44). The microorganisms with the highest enzymatic activity in the liquid medium were IM32-90 (endoglucanase = 0.214 U mL⁻¹) and IM32-91 (β -glucosidase = 0.067 U mL⁻¹).

Keywords: cellulase, enzymes, renewable sources.

Avaliação da produção de endoglucanase e β -glicosidase por bactérias e leveduras isoladas de plantações de eucalipto no cerrado de Minas Gerais

RESUMO

O atual cenário ambiental e econômico global tem uma relação intrínseca com o aumento do consumo de combustíveis fósseis causados pelo desenvolvimento de tecnologias e pelo crescimento da população mundial. Assim, é necessário procurar fontes renováveis de

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biocombustíveis na tentativa de mitigar os efeitos dos combustíveis fósseis no meio ambiente e a falta desses combustíveis não renováveis. O uso de biomassa lignocelulósica, um recurso abundante e renovável nas regiões brasileiras, contribuiu com sucesso para novas pesquisas e tecnologias para a produção de etanol de segunda geração. A conversão da biomassa lignocelulósica em açúcares fermentáveis requer o uso de enzimas celulolíticas produzidas por micro-organismos encontrados na microbiota. Este trabalho avaliou a produção de endoglucanase e β -glicosidase em meio líquido contendo carboximetilcelulose por cinco micro-organismos (bactérias e leveduras) do banco de 348 isolados de solos de eucalipto no Cerrado Mineiro. Foram selecionados micro-organismos com os seguintes índices de atividade enzimática: IM1-74 (22), IM25-9 (5.33), IM32-90 (7.33), IM1-5 (10.33) e IM32-91 (5.44). Os micro-organismos com maior atividade enzimática em meio líquido foram IM32-90 (endoglucanase = 0,214 U mL⁻¹) e IM32-91 (β -glicosidase = 0,067 U mL⁻¹).

Palavras-chave: celulase, enzimas, fontes renováveis.

1. INTRODUCTION

Global dependence on fossil fuels has caused unfavorable effects such as declining crude oil reserves, declining air quality and rising global temperatures. To promote sustainability and independence from fossil fuels, bioethanol is now considered to be a good substitute for a fossil fuel mixture or gasoline (Aditiya *et al.*, 2016; Toogood and Scrutton, 2018).

Lignocellulosic biomass represents a promising source of renewable energy, and can be used for the production of second-generation bioethanol (Squinca *et al.*, 2018). Currently, most of the lignocellulosic ethanol is produced by hydrolysis and fermentation. The enzymatic saccharification of plant biomass is the most efficient method for the breakdown of lignocellulose to sugars, although the cost of this operation is still one of the main bottlenecks in the commercialization of second-generation alcohol and has been reported as the most expensive point in the entire chain of bioethanol production, accounting for up to 40% of the total cost (Choudhary *et al.*, 2017; Cerda *et al.*, 2017). Decreasing the cost of cellulolytic enzymes remains one of the main goals of bioethanol research, as it can reduce the cost of these enzymes by up to 20% (Dodda *et al.*, 2018).

Cellulolytic enzymes include endo- and exoglucanases and beta-glucosidases that act synergistically and are necessary for the efficient hydrolysis of cellulose to soluble oligosaccharides (Lucarini *et al.*, 2017). The endoglucanases initiate hydrolysis by slicing the cellulose structure, uncovering reducing and non-reducing ends, whereas cellobiohydrolases (exoglucanases) act upon both ends to release cello-oligosaccharides and cellobiose units. Subsequently, β -glucosidases chop cellobiose to release glucose molecules. Further, during the hydrolysis reaction, the endoglucanases act on the amorphous region, which is the more soluble portion of the cellulose structure; meanwhile, cellobiohydrolases cleave β -1,4-glycosidic bonds from the chain ends (Srivastava *et al.*, 2018).

Cellulases have various kinds of industrial applications, including pulp and paper, laundry, food, animal feed, brewery and wine, textile, bioenergy industry (Idris *et al.*, 2017). Significant research has been developed focusing on the isolation, identification and optimization of these enzymes, thus leading to the marketing of cellulases. Commercially available cellulases are a formulation of endoglucanases, exoglucanases, and β -glucosidases (Obeng *et al.*, 2017).

The enzymatic process is pollution-free, economical and cost-effective (Li *et al.*, 2012). This gives a new horizon to finding cost-effective cellulose-producing microorganisms. Microorganisms such as aerobic bacteria, fungi, yeasts and actinomycetes produce cellulases that degrade cellulose by hydrolysis of β -1,4-glycosidic bonds of cellulose (Behera *et al.*, 2017).

The objective of this study was to evaluate the production of endoglucanase and β -

glucosidase by five microorganisms (bacteria and yeasts) isolated from the soil of a eucalyptus plantation in the cerrado region of the state of Minas Gerais, Brazil, with different periods of planting.

2. MATERIALS AND METHODS

For the evaluation of the production of endoglucanase and β -glucosidase, the 1-74, 25-9, 32-90, 1-5 and 32-91 microorganisms from the microorganisms bank of the Laboratory of Biotechnology and Genetics of the Department of Forest Engineering of the Federal University of the Jequitinhonha and Mucuri Valleys, Diamantina, Minas Gerais, Brazil, with previously calculated enzyme activity indices (EAI): EAI1-75 = 22, EAI25-9 = 5.33, EAI32-90 = 7.33, EAI1-5 = 10.33 and EAI32-91 = 5.44 were employed.

2.1. Culture medium and inoculum

To prepare the inoculants, the microorganisms assigned as described above were inoculated on a Petri plate with culture medium containing carboxymethylcellulose (CMC) as the main carbon source, according to the composition described in Table 1, and incubated in BOD (SL 200/334 SOLAB®) at 28°C for 96 hours.

Table 1. Composition of the culture medium used for initiation of inoculum growth.

Components	Amount (g)
MgSO₄ . 7H₂O	0.2
KH₂PO₄	0.4
K₂HPO₄	0.1
NaCl	0.1
Yeast extract	0.4
CMC	10
Agar	15
Distilled water	1000 mL

The culture medium was prepared from the mixture of the components described in Table 1. The pH was adjusted to 6.0, followed by sterilization in an autoclave at 121°C for 15 min.

After growth was verified, the microorganism was scraped with the aid of a platinum handle and inoculated in 12 mL of culture medium with a composition similar to that mentioned above, but without agar, contained in a 15-mL Falcon tube. The tube was incubated in a SHAKER (SL 222 SOLAB®) at 28°C and 150 rpm for 96 h.

2.2. Culture of microorganisms for the production of endoglucanase and β -glucosidase

After 96 hours of growth, a 2-mL sample was withdrawn from the inoculum and the optical density was measured at 600 nm in a Lambda Bio PERKINELMER® spectrophotometer. The inoculum was then added to an Erlenmeyer flask containing 190 mL of the medium described above (without agar) and incubated in a SHAKER (SL 222 SOLAB®) at 28°C and 150 rpm for 240 hours. Two-mL aliquots were withdrawn at 24-hour intervals and centrifuged at 10,000 rpm for 10 min for evaluation of the production of endoglucanase and β -glucosidase.

2.3. Evaluation of endoglucanase production

Analysis of endo-1,4- β -glucanase activity was performed according the method modified from the International Union of Pure and Applied Chemistry (IUPAC) standard method, described by Ghose (1987). This method consists of hydrolysis of a solution of 1% CMC (Synth), followed by quantification of the soluble reducing sugars released over a given time

interval. The concentration of reducing sugars was determined according to the method described by Miller (1959) using 3,5-dinitrosalicylic acid (DNS) (Miller, 1959). The reaction was performed in Falcon tubes (15 mL) containing 1000 μL of 1% (w/v) CMC solution in 100 mmol.L⁻¹ acetate buffer, pH 4.8 and 1000 μL of the enzyme extract. The mixture was incubated at 50°C, aliquots of 200 μL were withdrawn at 0-, 15- and 30-minute intervals, and 200 μL of DNS was added. A reaction without the presence of the enzyme extract (blank) was prepared by the addition of 100 μL DNS to 100 μL of distilled water. Samples and blank were heated in a boiling water bath ($95 \pm 1^\circ\text{C}$) for 5 minutes, followed by cooling in an ice-water bath ($0 \pm 1^\circ\text{C}$) and the addition of 2 mL and 1 mL of distilled water to the sample and the blank, respectively. The absorbance was measured at 540 nm using an analytical glucose curve at concentrations of 0 to 1.6 mg/mL in 0.2 mg mL⁻¹ intervals as a reference (Figure 1).

Figure 1. Analytical glucose curve.

One unit of endoglucanase activity was defined as the amount of enzyme capable of releasing 1 μmol of reducing sugars, expressed as glucose, per minute per unit volume of enzyme extract. Data were submitted to analysis of variance considering a level of significance (α) of 0.05 using software ORIGIN PRO 8.

2.4. Evaluation of β -glucosidase production

The assay of β -glucosidase activity was implemented using the method modified from the International Union of Pure and Applied Chemistry (IUPAC) standard method, described by Ghose (1987), which involves hydrolysis of a 1% solution of cellobiose in 100 mmol L⁻¹ citrate buffer at pH 4.8 in the presence of the enzyme extract. The reaction medium was composed of 100 μL of the substrate solution and 100 μL of the enzyme extract in Eppendorf tubes. The tubes were incubated at 50°C in a water bath for 30 minutes, followed by boiling ($95 \pm 1^\circ\text{C}$) for 1 min and cooling in an ice bath. Aliquots of 15 μL were removed for quantification of glucose using the standard enzymatic procedure employing glucose oxidase/peroxidase (GOD-POD) (Lloyd and Whelan, 1969) with spectrophotometric measurement at 510 nm. For each enzymatic reaction, one was prepared without the presence of the enzyme extract (blank), following the same procedure described for the sample. One unit of β -glucosidase activity was defined as the amount of enzyme capable of releasing 1 μmol of glucose per minute of reaction, per unit volume (liter) of enzyme extract. Data were submitted to analysis of variance considering a level of significance (α) of 0.05 using software ORIGIN PRO 8.

3. RESULTS AND DISCUSSION

3.1. Profile of endoglucanase production

The five strains of microorganisms selected according to the enzyme activity index (EAI) were grown in medium containing CMC as the main source of carbon, and they were monitored for 240 h for evaluation of endoglucanase and β -glucosidase activities, as shown in Figures 2A, 2B, 3A and 3B.

Figure 2. A: Punctual activities of endoglucanase from the five strains evaluated during 240 hours of culture. B: Maximum endoglucanase activity.

There is a relatively similar behavior for endoglucanase activities in all the strains (Figures 2A and 2B). The highest endoglucanase activities (CMCase) were observed at 96 hours of fermentation with the microorganism IM32-90 (0.214 U mL⁻¹), 120 hours with IM25-9 (0.198 U mL⁻¹) and 168 hours with IM32-91 (0.210 U mL⁻¹).

In general, the enzymatic activities of endoglucanases in this work were similar to those of other organisms already studied, as in Yang *et al.* (2011), who observed 0.24 U mL⁻¹ in a study that demonstrated the presence of *Achromobacter xylosoxidans*, *Alcaligenes faecalis* and *Fusarium sporotrichioides*. A similar result was also obtained by Mohapatra *et al.* (2018) when evaluating cellulase production by *Aspergillus fumigatus* isolated from decomposing cellulosic waste, showing a higher endoglucanase activity (0.287 U mL⁻¹) obtained at 30°C, which shows that the cellulase activity is active in this temperature range. Carvalho (2013) obtained 0.164 U mL⁻¹ by cultivating *Cryptococcus laurentii* in medium supplemented with 2% wheat bran.

The analysis of variance showed that there was no significant difference at 0.05 between the endoglucanase production of the five microorganisms evaluated (Table 2).

Table 2. Analysis of variance (ANOVA) of the endoglucanase production by the five micro-organisms evaluated.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	4	0.00947	0.00237	1.51983	0.24653
Error	15	0.02338	0.00156		
Total	19	0.03285			

One Way ANOVA

Overall ANOVA

Null Hypothesis: The means of all levels are equal.

Alternative Hypothesis: The means of one or more levels are different.

At the 0.05 level, the population means are not significantly different.

3.2. Profile of β -glucosidase production

The production of β -glucosidase by the five selected strains was observed for 240 hours, as described in Figures 3A and 3B.

Figure 3. A: Punctual β -glucosidase activities of the five strains evaluated during 240 hours of culture. B: Maximum β -glucosidase activity.

The highest cellobiase activity (0.067 U mL⁻¹) was observed for the IM32-91 strain after 216 h of culture, followed by the IM1-5 strain (0.059 U mL⁻¹) at 144 h of culture. After 216 h of fermentation, the cellobiase activity of strain IM32-91 began to decrease. The IM1-74 strain with the highest enzymatic activity index (EAI = 22) had the lowest endoglucanase (0.161 U mL⁻¹) and β -glucosidase (0.039 U mL⁻¹) activities in liquid medium, as is shown in Table 3.

Table 3. Enzymatic activity indices expressed in solid medium and endoglucanase and β -glucosidase activity of the five strains evaluated in liquid medium.

Strain	EAI	Endoglucanase (U mL ⁻¹)	β -Glucosidase (U mL ⁻¹)
IM1-74	22.00	0.161	0.039
IM25-9	5.33	0.198	0.044
IM32-90	7.33	0.214	0.056
IM1-5	10.33	0.167	0.059
IM32-91	5.44	0.210	0.067

The analysis of variance showed that there was no significant difference at 0.05 between the β -glucosidase production of the five microorganisms evaluated (Table 4).

Table 4. Analysis of variance (ANOVA) of the β -glucosidase production by the five microorganisms evaluated.

	DF	Sum of Squares	Mean Square	F Value	Prob>F
Model	4	0.002	4.99157E-4	1.0913	0.41185
Error	10	0.00457	4.57398E-4		
Total	14	0.00657			

One Way ANOVA

Overall ANOVA

Null Hypothesis: The means of all levels are equal.

Alternative Hypothesis: The means of one or more levels are different.

At the 0.05 level, the population means are not significantly different.

The IM32-91 strain, which has one of the lowest levels of enzymatic activity among the selected microorganisms (EAI = 5.44), was the best producer of β -glucosidase (0.067 U mL^{-1}) and the second best producer of endoglucanase (0.210 U mL^{-1}). The highest endoglucanase production was observed for strain IM32-90 (0.214 U mL^{-1}). The values of β -glucosidase production were very satisfactory compared to those found by Vyas and Chhabra (2017), who cultivated the yeast *Cystobasidium oligophagum* in a medium containing CMC as carbon source under the same conditions and obtained 0.031 U mL^{-1} after 72 hours of incubation. Priyanka *et al.* (2017) obtained 0.077 U mL^{-1} endoglucanase as the maximum value produced by the fungus *Paecilomyces variotii* when testing different concentrations of CMC as a carbon source. This value was about three times less than the maximum value obtained in this work for endoglucanase (0.214 U mL^{-1}).

CMC is considered to be a better substrate for cellulase production due to its soluble nature compared to other carbon sources, demonstrating its effectiveness in this work (Sohail *et al.*; 2016).

The production of cellulolytic enzymes can be increased by changes in culture medium conditions, such as supplementation of the culture medium and the use of recombinant microorganisms. Anusree *et al.* (2016) cultured recombinant *Corynebacterium glutamicum* bacteria with *Xanthomonas campestris* genes to evaluate the production of endoglucanase and *Saccharophagus degradans* for the production of β -glucosidase in medium supplemented with 20 g.L^{-1} of CMC. They obtained $8 \pm 0.4 \text{ U mL}^{-1}$ of β -glucosidase and $4.5 \pm 0.2 \text{ U mL}^{-1}$ of endoglucanase. In medium supplemented with 20 g.L^{-1} of cellobiose, they obtained $9.0 \pm 0.5 \text{ U mL}^{-1}$ of β -glucosidase and $2 \pm 0.13 \text{ U mL}^{-1}$ of endoglucanase. The strains evaluated in this work will be submitted for molecular identification.

The analysis of variance (ANOVA) with significance level 0.05 (Tables 2 and 4) showed that there was no significant difference in the production of endoglucanase and β -glucosidase by the five cellulase-producing microorganisms evaluated. These results indicate that any of the organisms evaluated can be used for the production of these enzymes, and their production processes may be optimized in future works.

4. CONCLUSION

The five strains evaluated were effective for cellulase production, presenting hydrolytic activities in carboxymethylcellulose and cellobiose, and proving that they are promising tools for the production of these enzymes. Further studies should be performed to increase cellulolytic capacity and enzyme extraction for commercial application.

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Genesis of sandstone-derived soils in the Cerrado of the Piauí State, Brazil

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ABSTRACT

This study characterized the morphological, physical and chemical attributes of sandstone-derived soils at the Cerrado of the Piauí State, Brazil, in order to identify evolutionary standards. The study was carried out with five representative soil profiles identified as P1-RY (Typical Flavic Psychotic Neosol - Aquents), P2-PA (Typical Dystrophic Yellow Argisol - Alfisol), P3-RL (Fragmentary Litholic Dystrophic Neosol - Psammenit), P4-RQ (Typical Ortique Quartzenetic Neosol - Orthents) and P5-PV (Typical Dystrophic Red Argisol - Ultisol). Soil samples were submitted laboratory analysis described morphologically. In general, the soils presented high sand content, low pH, low content of exchangeable bases and low cation exchange capacity (CEC). Organic matter governed the CEC in most cases, suggesting dependence of organic matter in the supply of charges. These soils showed a low degree of weathering, but with iron of high crystallinity. Thus, the relief and the parent material are the major important soil-forming factors at the Cerrado of the Piauí State. Moreover, these soils are young, with the soils from the Piauí Formation being more evolved. However, the sandstones from the Canindé Group apparently are providing lithological secondary minerals for the soil.

Keywords: sand-loam soils, tropical soils, weathering indexes.

Gênese de solos derivados de arenitos no Cerrado Piauiense, Brasil

RESUMO

Objetivou-se com este trabalho caracterizar atributos morfológicos, físicos e químicos de solos formados a partir de arenitos no Cerrado Piauiense, na tentativa de encontrar padrões evolutivos. O estudo foi realizado em cinco perfis de solos representativos identificados como P1-RY (Neossolo Flúvico Psamítico típico - Aquents), P2-PA (Argissolo Amarelo Distrófico típico - Alfisol), P3-RL (Neossolo Litólico Distrófico fragmentário - Psammens), P4-RQ

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(Neossolo Quartzarênico Órtico típico - Orthents) e P5-PV (Argissolo Vermelho Distrófico Típico - Ultisol). As amostras dos solos foram submetidas à descrição morfológica e análises laboratoriais. Em geral, os solos desta região apresentaram altos teores da fração areia, baixo pH, baixo conteúdo de bases trocáveis e baixa capacidade de troca catiônica (CTC). A matéria orgânica governou a CTC na maioria dos horizontes, sugerindo uma dependência da matéria orgânica no fornecimento de cargas. Estes solos mostraram baixo grau de intemperismo, mas com ferro de alta cristalinidade. Assim, é possível afirmar que o relevo e o material de origem são os fatores de formação do solo que possuem maior influência nos atributos dos solos do Cerrado Piauiense, Brasil. Além disso, estes solos são jovens, sendo os mais evoluídos aqueles originados da Formação Piauí. Contudo, os arenitos do grupo Canindé aparentemente estão fornecendo minerais secundários diretamente da rocha ao solo.

Palavras-chave: índices de intemperismo, solos franco-arenosos, solos tropicais.

1. INTRODUCTION

The Cerrado biome is home to the main thrust of Brazilian agricultural expansion. Its climate and soil characteristics are very diverse. The soils of the Brazilian Cerrado evolve from rocks that go back from Pre-Cambrian to Cenozoic (Pfaltzgraff, 2010). The relief exhibits a very large range of morphological features distributed at different altitudes, constituting well-defined units, among which stand out plateaus, depressions, and plains. Soils consist of representative units of more than a dozen classes and are mostly dystrophic and acid (Pfaltzgraff, 2010).

In the state of Piauí, this biome occupies approximately 11.5 million hectares and is world-renowned for its economic importance as a reflection of its imminent agricultural expansion (Pragana *et al.*, 2012). There is also noticeable soil variability at different levels when compared with the Cerrado of other Brazilian regions. This variability, which occurs in response to the interaction between the soil formation factors, suggests major challenges for a conscious agricultural production.

The particularities of each site must be considered to facilitate the use of natural resources in agricultural activities. According to Buol *et al.* (2011), soil genesis and classification can permit better and more concise recommendations for soil use. Since weathering is directly related to the soil genesis, Kehrig (1949) reported that using weathering indexes such as Ki and Kr is a great option to explore soil genesis, considering the lack of more accurate information provided by x-ray diffractometry.

In the Cerrado of Piauí, most farmers consider general information about the soils for the adoption of management systems. Most of them assume that the soil belongs to the order of the Oxisols, with not only the same weathering level but also characteristics of soils from other places (i.e. soils in the south and southeast regions). This inevitably causes errors since not every soil in this region has the same level of weathering and homogeneity due to the landscape often observed in the Cerrado of Piauí State. Negative reflections of these deductions are amplified due to the sedimentary nature of the parent material of these soils, in addition to the climate intensity to which they are submitted each year, which demand more and more soil performance.

To correct this, the first steps involve soil characterization, recognizing that the evolution of each one soil is inherently tied to its formation environment. Thus, this study characterized the morphological, physical and chemical attributes of sandstone-derived soils at the Cerrado of the Piauí State in order to identify evolutionary standards.

2. MATERIAL AND METHODS

2.1. Study area

This research was carried out using five representative soils of the Upper Middle Gurguéia micro-region, at the Cerrado of the Piauí State, Brazil (Figure 1). The regional relief is craggy, with a warm and humid tropical climate, classified as Aw according to the Köppen classification. Historical data from the last 44 years, 1973 to 2017, show that the mean values of minimum and maximum temperatures are 20.3 and 33.6°C, respectively, and the mean annual precipitation is 939 mm (Inmet and Bdmnet, 2017).

In order to represent as much as possible the environments of the study area, the sampling sites were chosen based on its geodiversity map (Pfaltzgraff, 2010). At each site, a trench was opened to expose the profiles of the studied soils, a procedure described by Santos *et al.* (2015) in areas under native Cerrado. Soil horizons were collected and identified according to Santos *et al.* (2015).

The study was carried out in five representative soil profiles identified as P1-RY (Typical Flavic Psychotic Neosol - Aquents), P2-PA (Typical Dystrophic Yellow Argisol – Alfisol), P3-RL (Fragmentary Litholic Dystrophic Neosol - Psamments), P4-RQ (Typical Ortio Quartzenetic Neosol - Orthents) and P5-PV (Typical Dystrophic Red Argisol - Ultisol).

Figure 1. Study area location with indication of the sampling sites (profiles) on the parent material map.

The P1-RY is located at the flood plain and was developed under unconsolidated sediments (sand and gravel) of Cenozoic origin (Pfaltzgraff, 2010). The P2-PA and P5-PV were derived

from sandstone (Piauí Formation, Balsas Group) of Paleozoic origin. The P2-PA is located at the crest, whereas the soil P5-PV is on the shoulder (Pfaltzgraff, 2010).

The P3-RL was also derived from sandstone (Piauí Formation, Canindé Group), but it is positioned at the side slope (Pfaltzgraff, 2010). The P4-RQ is located at the toe slope and was derived from sandstone (Mesozoic origin, Urucuia Complex) (Pfaltzgraff, 2010).

2.2. Physical and chemical analyzes

The samples from each horizon or layer were air-dried and subsequently declotted, homogenized, sieved in a stainless steel sieve with a 2.00 mm mesh, and used for the physical and chemical analyses. The particle size distribution was performed using 0.1 mol L⁻¹ NaOH solution as a dispersant under slow stirring and the clay content was determined by the pipette method (Teixeira *et al.*, 2017). The clay dispersed in water (CDW) and the degree of flocculation were obtained according to Teixeira *et al.* (2017).

The pH in water and KCl, potential acidity (H+Al), calcium (Ca²⁺), magnesium (Mg²⁺), potassium (K⁺), and aluminum (Al³⁺) were obtained using the methodologies proposed by Raij *et al.* (2001). The P and organic carbon (C_{org}) contents were analyzed using the methodology recommended by Teixeira *et al.* (2017). The values equivalent to the sum of bases (SB), cation exchange capacity (CEC), base saturation (V), and aluminum saturation (m) were obtained by calculations proposed by Teixeira *et al.* (2017).

2.3. Sulfuric attack, weathering indexes and crystallinity of iron oxides

Topsoil samples of all profiles were used for analyses of sulfuric attack and crystallinity of iron oxides. Only the samples of the subsurface diagnostic horizons of the P2-PA (Bt1) and P5-PV (Bt) were used for that. In the total sulfuric attack, the samples were treated with H₂SO₄ to extract the total contents of iron (Fe₂O₃) and aluminum (Al₂O₃) oxides, followed by alkaline dissolution for silicon (SiO₂) and titanium (TiO₂) oxides according to Teixeira *et al.* (2017). These values were used to calculate the weathering indexes Ki and Kr as suggested by Kehrige (1949).

The free iron oxides (Fe_d) were extracted with citrate-dithionite-bicarbonate (Mehra and Jackson, 1960) and the poorly crystallized iron (Fe_o) was extracted with acid ammonium oxalate according to the methodology described by Camargo *et al.* (2009) adapted from Schwertmann (1973).

3. RESULTS AND DISCUSSION

3.1. Soil Morphology

The soil P1-RY presented the horizons A-2C1-3C2-4C3-5C4-6C5-7C6-8C7-9C8 formed by lithological discontinuity along a 2.0 m depth (Table 1). These layers show heterogeneity, among others, in the texture and color of the material that makes up the solid phase of the soil, denoting no or little relation to each other. This discontinuity is caused by the deposition of sediments in the Holocene period that occurs yearly during the wet season, which provides water presence on the surface. Darker shades in these layers are indicative of an erratic organic matter distribution (Demattê *et al.*, 2011). Silva *et al.* (2017) studied profiles of a landscape in the Brazilian Cerrado and also found in the flood plain a profile with color variability.

The P2-PA and P5-PV soil profiles showed the diagnostic horizon know as Bt, coherent with the Argissolos (Ultisol) class (Santos et al., 2018) (Table 1). The P2-PA has a yellowish hue (5Y), whereas the soil P5-PV has reddish hue (10R to 2.5YR), which probably reflects differences related to the goethite and hematite contents. Both P2-PA and P5-PV have differences along their depths, being P2-PA more developed. Apparently, the P5-PV evolved through soil-erosion processes often observed in soils located at the slopes of the landscape (Buol et al., 2011).

Table 1. Morphological description of the profiles studied.

Horizon	Depth (m)	Color (moisture)	Structure ^a	Consistency			Transition ^b
				Dry	Moisture	Wet ^c	
P1–RY							
A	0-0.15	5YR 4/3	S; Sm; e SubBlo	Slightly hard	Friable	Npl / St	Grad e Fla
2C1	0.15-0.35	5YR 6/6	Sg; T e Granu	Slightly hard	Friable	Npl / St	Grad e Fla
3C2	0.35-0.45	7.5YR 7/6	Sg; T e Granu	Slightly hard	Friable	Npl / St	Abr e Fla
4C3	0.45-0.80	7.5YR 7/6	Sg; T e Granu	Slightly hard	Friable	Spl / St	Grad e Fla
5C4	0.80-1.10	5YR 5/4	S; grande e SubBlo	Slightly hard	Friable	Spl / St	Abr e Fla
6C5	1.10-1.30	5YR 5/	Mo; Sm; e SubBlo	Slightly hard	Friable	Spl / Stk	Abr e Rip
7C6	1.30-1.40	5YR 5/4	Mo; T e SubBlo	Slightly hard	Friable	Spl / Stk	Grad e Fla
8C7	1.40-1.60	7.5YR 6/8	Sg; T e SubBlo	Slightly hard	Friable	Spl / St	Grad e Fla
9C8	1.60-2.00+	7.5YR 6/8	Sg; T e SubBlo	Slightly hard	Friable	Spl / St	-
P2–PA							
A	0-0.20	5Y 2.5/2	Mo; T e SubBlo	Slightly hard	Friable	Npl / Stk	Cle
AB	0.20-0.35	5Y 5/2	Mo; Sm; e SubBlo	Slightly hard	Friable	Npl / St	Grad
BA	0.35-0.60	5Y 7/4	Mo; Sm; e SubBlo	Slightly hard	Friable	Npl / St	Dif
Bt1	0.60-1.10	5Y 7/6	Mo; Sm; e Granu	Slightly hard	Friable	Spl / St	Dif
Bt2	1.10-2.10+	5Y 7/6	Mo; Sm; e Granu	Slightly hard	Friable	Spl / St	-
P3–RL							
A	0-0.10	7.5YR 3/1	W; Sm; e Granu	Slightly hard	Friable	Spl / St	Abr e Des
CR	0.10-0.40	7.5YR 3/1	W; Sm; e Granu	Slightly hard	Firm	Spl / St	-
P4–RQ							
A1	0-0.20	5YR 4/4	W; Sm; e SubBlo	Soft	Very friable	Npl / NSTk	Dif e Fla
A2	0.20-0.55	5YR 5/6	W; Sm; e SubBlo	Soft	Very friable	Npl / NSTk	Grad e Fla
C1	0.55-1.13	5YR 6/8	W; Sm; e SubBlo	Soft	Very friable	Npl / NSTk	Cle e Fla
C2	1.13-2.00+	5YR 6/8	W; Sm; e SubBlo	Soft	Very friable	Npl / NSTk	-
P5–PV							
A	0-0.20	2.5YR 3/6	S; Med e SubBlo	Slightly hard	Friable	Spl / St	Fla e Cle
BA	0-20-0.30	2.5YR 4/6	S; Med e SubBlo	Hard	Firm	Spl / St	Fla e Cle
Bt	0.30-0.85	10R 4/8	S; Med e SubBlo	Hard	Firm	Spl / St	Fla e Cle
BC	0.85-1.10	10R 4/8	S; Med e SubBlo	Hard	Extremely firm	Spl / St	Fla e Cle
CR	1.10-1.30	10R3/6	S; grande e SubBlo	Too Hard	Extremely firm	Spl / St	-

^aS, Strong; W, Weak; Mo, Moderate; Sg, Simple grains; T, Tiny; Sm, Small; Med, Medium; SubBlo, Subangular Blocks; Granu, Granular. ^bGrad, Gradual; Dif, Diffuse; Abr, Abrupt; Cle, Clear; Fla, Flat, Rip, Rippled; Dis, Discontinuous. ^cSt, Slightly tacky; Stk, Sticky; NStk, Not Sticky; Spl, Slightly plastic; Npl, Not plastic.

In P3-RL, an A horizon was found on a Cr layer, with lithic contact less than 50 cm deep (Table 1). This profile is located in a landscape position typically erosional, which prevents the formation of deeper soils. The A horizon had a dominance of very dark gray color (7.5YR 3/1) with a weak structure. The Cr layer consists of angular and subangular rock boulders, accounting for more than 90% of the volume of its mass, with the remaining part filled with a weathered material (A horizon). The current climate condition may promote higher advances in soil formation in the long term. Other factors, such as organisms and relief, do not give conditions to potentiate the use of these soils for food production.

The soil P4-RQ presented sequence of horizons A1-A2-C1-C2 with a depth higher than 2.0 m (Table 1). This profile is located in the foothill position on the slope. All horizons have colors predominantly with a hue of 5YR. Because of the high sand content, it presented weak aggregates with structure in subangular blocks and the humid and wet consistency were very friable and not sticky, respectively. Due to the poor performance of the pedogenetic processes

of soil formation, the characteristics of this profile are closely related to the parent material, predominantly composed of sandstone.

3.2. Physical Attributes

The soil P1-RY presented an erratic distribution of clay, silt, and sand content. This lack of uniformity may result from the variation in the sedimentary nature of the parent material (Table 2). Due to the formation by sedimentation of fluvial nature, the occurrence of irregularity in the granulometric contents along the profile is a characteristic of this soil class (Buol *et al.*, 2011). The observed sand contents were higher than 741 g kg^{-1} , occurring in most horizons and composed of a higher proportion of fine sand.

Table 2. Physical characterization of the horizons of the studied profiles.

Horizon	Depth (m)	VCS ^a	CS	MS	FS	VFS	TS	Clay (H ₂ O)	Clay (NaOH)	Silt	Flocculation %	Textural class ^b
g kg ⁻¹												
P1-RY												
A	0-0.15	13	14	52	410	252	741	51	204	55	75	SCL
2C1	0.15-0.35	0	1	57	671	236	965	25	25	10	0	FSA
3C2	0.35-0.45	0	1	135	728	90	954	25	25	21	0	FS
4C3	0.45-0.80	1	3	35	511	312	862	25	100	38	75	LFS
5C4	0.80-1.10	6	24	49	135	259	810	25	153	37	84	LFS
6C5	1.10-1.30	30	216	375	195	98	914	25	75	11	67	FSA
7C6	1.30-1.40	5	47	105	271	296	724	25	101	15	75	SCL
8C7	1.40-1.60	0	3	12	477	436	928	25	50	22	50	FS
9C8	1.60-2.00 ⁺	1	21	327	424	177	950	25	25	25	0	FS
P2-PA												
A	0-0.20	4	43	278	480	79	884	25	101	15	75	LFS
AB	0.20-0.35	2	36	241	492	108	879	25	100	21	75	LFS
BA	0.35-0.60	1	31	223	478	67	800	0	176	25	100	FSL
Bt1	0.60-1.10	2	36	223	398	99	758	0	226	16	100	SCL
Bt2	1.10-2.10	1	36	213	373	90	713	0	251	35	100	SCL
P3-RL												
A	0-0.10	15	41	179	366	140	741	126	202	58	38	SCL
CR	0.10-0.40	18	48	210	313	163	752	100	190	58	38	SCL
P4-RQ												
A1	0-0.20	5	2	88	453	398	946	0	25	29	100	FSA
A2	0.20-0.55	1	2	109	437	361	910	0	75	15	100	FSA
C1	0.55-1.13	1	1	105	422	398	927	0	50	23	100	FSA
C2	1.13-2.00 ⁺	0	1	97	404	398	900	25	75	25	67	FSA
P5-PV												
A	0-0.10	5	18	203	468	135	829	75	125	45	40	LS
BA	0.10-0.20	4	20	196	322	82	624	0	201	175	100	SCL
Bt	0.20-0.85	5	18	180	307	84	594	0	251	155	100	SCL
BC	0.85-1.10	6	18	177	312	109	622	0	226	152	100	SCL
CR	1.10-1.30	4	21	209	338	91	663	25	175	161	86	SCL

^aVCS, Very coarse sand; CS, Coarse sand; MS, Medium sand; FS, Fine sand; VFS, Very fine sand. ^bSCL, Sand clay loam; FSL, Fine sand loam; LFS, Loamy fine sand; FSA, Fine sand; LS, Loamy sand.

The P2-PA and P5-PV soil profiles showed highest clay concentration at the B horizons than A horizons, with texture ratio B/A more than 1.8 (P2-PA = 2.4, P5-PV = 2.0), but with a predominance of sand (Table 2). The soil P2-PA varied from 101 to 251 g kg⁻¹ between A and Bt2 horizons, whereas the soil P5-PV presented a variation in clay content between 125 and

251 g kg⁻¹, the both in accordance with the criteria to classify it as a B textural, as observed by Santos *et al.* (2018).

Besides the difference of their position on the landscape, the soils P2-PA and P5-PV also had differences related to their parent material (i.e., sandstone). These differences may be responsible for granulometric variation and morphologic attributes showed in Table 1. However, both P2-PA and P5-PV showed flocculation equal to 100% at the Bt horizon that reflect the importance of the oxide content in the function of these soils.

In P4-RQ, the predominance of sand was significant in all horizons, with values ranging from 900 to 946 g kg⁻¹, while the clay content was between 25 and 75 g kg⁻¹. The parent material of this profile is characterized by sandstone and Cretaceous conglomerates, with a predominance of sandy sediments of continental, lacustrine, fluvial or wind deposition (Pfaltzgraff, 2010), which provided a higher amount of the sand fraction in this profile due to the little pedogenetic alteration.

3.3. Chemical Attributes

The values of pH in water of all profiles were higher in relation to the pH values in KCl, indicating a predominance of a negative net load for all the studied profiles (Table 3). All profiles, except for some layers in P1-RY, had low pH values, probably due to the low amount of exchangeable bases in these soils. The distribution of pH values presented a low variation in the horizons of each profile, except for P1-RY, presenting a pH in KCl with values between 4.3 and 5.6. The chemical characteristics of each layer of P1-RY are closely related to the parent material characteristic of this soil class, which is formed by sediment transport and deposition.

A low variation in the pH values was also observed in the soil P2-PA, with slightly lower values in the surface horizons. The lower values in the surface are possibly associated with the masking of the positive surface charge and creation of a negative charge due to the presence of organic matter. In addition, the increase of pH in the subsurface horizons of soils may be associated with the presence of iron oxides and the adsorption of H⁺ ions at the soil colloids (Buol *et al.*, 2011). The pH values in KCl show a similar tendency in the profile, but with a unit lower than that of the pH in water, indicating a predominance of acid cations (Al³⁺ and H⁺) at exchange sites (Tawornpruek *et al.*, 2006).

The contents of P are similar for most of the profile horizons, except for P1-RY and the A horizons of P3-RL, P4-RQ, and P5-PV. The soil P1-RY presented the highest values of the sum of exchangeable bases (K⁺, Ca²⁺, and Mg²⁺), being predominantly composed of Ca²⁺ and Mg²⁺. However, the lowest values of the sum of exchangeable bases were observed in P4-RQ and P5-PV, which is related to an intense leaching of basic cations and favored by climate conditions and soil characteristics. In P2-PA, the value of SB varied from 0.6 to 0.7 cmol_c kg⁻¹, with the highest values in the subsurface horizons. The increase of SB in depth in this profile is possibly related to a higher clay content, which increases its adsorption potential by cations. The contents of Ca²⁺ were higher in comparison to Mg²⁺ in all horizons and its values increased in depth, as observed in SB values. The contents of K⁺ were low in all horizons, with values close to zero.

The dystrophic character was observed in most of the studied profiles, except for P1-RY, which showed a base saturation higher than 50% in the A horizon and in some of its layers. The low base saturation values observed in P2-PA, P3-RL, P4-RQ, and P5-PV reflect the low natural fertility of these soils. Regarding the C_{org} distribution, P1-RY presented an erratic distribution, while the other profiles showed higher values in the surface horizons, with decreases in depth. The same results were observed for the contents of Al³⁺ + H⁺, which presented higher values in the surface horizons. According to Inda Junior *et al.* (2007), the formation of organometallic complexes with Al³⁺ increases the resistance of organic matter and minimizes the losses of Al³⁺.

Table 3. Chemical characterization of the studied profiles.

Horizon	pH		Δ pH	P mg kg ⁻¹	K ⁺	Ca ²⁺	Mg ²⁺ cmol _c kg ⁻¹	Al ³⁺	SB	V (%)	m	C _{org} g kg ⁻¹	H+Al cmol _c kg ⁻¹	CEC
	H ₂ O	KCl												
P1-RY														
A	6.1	5.1	-1.0	31.1	0.6	3.9	1.7	0.2	6.2	70	3	1.7	2.6	8.8
2C1	6.2	4.5	-1.7	19.7	0.1	0.7	0.3	0.2	1.1	44	15	0.6	1.4	2.5
3C3	5.7	4.3	-1.4	9.6	0.1	0.5	0.2	0.1	0.8	44	11	0.1	1.0	1.8
4C3	6.1	4.4	-1.7	29.1	0.2	1.2	0.7	0.1	2.1	57	5	1.2	1.6	3.7
5C4	6.9	5.6	-1.3	9.5	0.4	3.7	3.9	0.1	8.0	92	1	2.9	0.7	8.7
6C5	7.2	5.2	-2.0	13.1	0.1	0.8	1.2	0.1	2.1	81	5	0.1	0.5	2.6
7C6	6.2	5.0	-1.2	5.5	0.2	1.1	2.0	0.1	3.3	79	3	0.6	0.9	4.2
8C7	6.7	5.0	-1.7	4.0	0.1	0.7	1.0	0.1	1.8	82	5	0.1	0.4	2.2
9C8	6.6	5.1	-1.5	3.2	0.1	0.4	0.1	0.2	0.6	55	25	0.1	0.6	1.1
P2-PA														
A	4.6	3.6	-1.0	1.7	0.1	0.4	0.1	0.1	0.6	15	14	5.2	3.5	3.9
AB	4.7	3.8	-0.9	1.7	0.1	0.4	0.1	0.6	0.6	23	50	2.3	2.2	2.6
BA	4.9	3.8	-1.1	1.7	0.1	0.4	0.1	0.5	0.6	28	45	2.3	1.7	2.1
Bt1	4.9	3.9	-1.0	1.7	0.1	0.5	0.1	0.5	0.7	33	42	1.7	1.6	2.1
Bt2	5.1	4.0	-1.1	1.7	0.1	0.5	0.1	0.3	0.7	39	30	0.6	1.3	1.8
P3-RL														
A	4.2	3.5	-0.7	4.0	0.1	0.1	0.1	1.1	0.3	7	79	10.5	4.1	4.4
CR	4.9	3.9	-1.0	1.4	0.1	0.2	0.2	0.2	0.5	31	29	4.2	1.3	1.6
P4-RQ														
A1	5.3	3.9	-1.4	2.0	0.1	0.1	0.1	0.5	0.3	17	63	2.9	1.7	1.8
A2	5.6	4.2	-1.4	1.7	0.1	0.1	0.1	0.4	0.3	18	57	1.2	1.7	1.7
C1	5.0	4.0	-1.0	1.7	0.1	0.1	0.1	0.4	0.3	17	57	1.2	1.8	1.8
C2	5.0	4.0	-1.0	1.7	0.1	0.1	0.1	0.4	0.3	19	57	0.1	1.6	1.6
P5-PV														
A	4.1	3.4	-0.8	3.0	0.1	0.1	0.1	0.8	0.3	9	73	7.6	3.1	3.3
BA	4.4	3.5	-0.8	1.7	0.1	0.1	0.1	0.9	0.3	20	75	1.2	1.5	1.5
Bi	3.6	3.3	-0.3	1.7	0.1	0.1	0.1	1.1	0.3	13	79	1.2	2.3	2.3
BC	3.6	3.2	-0.4	1.7	0.1	0.1	0.1	1.0	0.3	14	77	1.2	2.2	2.2
CR	3.8	3.2	-0.6	1.7	0.1	0.1	0.1	0.8	0.3	33	73	1.2	0.9	0.9

The highest CEC values were 8.8 and 8.7 cmol_c kg⁻¹ for the A horizon and layer 5C4 in P1-RY, respectively. Other studied soils showed CEC decrease with depth, as well as C_{org} content. This scenario can be related to the low clay content in these soils, as well as the probable predominance of minerals containing a small amount of permanent charge such as kaolinite, which provide high dependence of organic matter in the CEC. Tawornpruek *et al.* (2006) also observed a relationship between CEC variations in depth with soil organic carbon content.

3.4. Sulfuric attack, weathering indexes and crystallinity of iron oxides

In the clay fraction of the studied soils, the SiO₂ was the oxide of highest concentration (3.2 to 8.9%), followed by Al₂O₃ (1.5 to 8.5%), Fe₂O₃ (0.4 to 3.2%) and TiO₃ (0.2 to 0.9%) (Table 4). This behavior suggests a majority presence of phyllosilicates in this fraction (Buol *et al.*, 2011). The soils P2-PA and P5-PV presented the highest mean SiO₂ content, which is reflection of the content of the clay fraction in these soils. The behavior observed for Al₂O₃, and Fe₂O₃ suggests high content of aluminum octahedrons and iron oxides such as goethite and hematite, respectively.

Table 4. Oxides rates by sulfuric attack, weathering indices and crystalline and amorphous Fe.

Horizon	SiO ₂	Al ₂ O ₃ %	TiO ₃	Fe ₂ O ₃	Ki	Kr	Fe _d g kg ⁻¹	Fe _o	Fe _o /Fe _d
P1 – RY									
A	6.4	3.3	0.5	1.4	3.3	2.6	0.9	0.5	0.6
P2 – PA									
A	6.1	4.2	0.6	3.0	2.5	1.7	1.9	0.3	0.2
Bt1	8.9	8.5	0.9	3.2	1.8	1.4	1.5	0.2	0.1
P3 – RL									
A	7.7	6.7	0.3	0.9	2.0	1.8	1.0	0.2	0.2
P4 – RQ									
A1	3.2	1.5	0.2	0.4	3.6	3.1	0.6	0.2	0.3
P5 – PV									
A	9.8	6.7	0.4	1.7	2.5	2.1	1.8	0.1	0.1
Bt	8.7	8.0	0.5	1.6	1.8	1.6	1.0	0.1	0.1

The P1-RY and P4-RQ soil profiles presented the highest Ki values (Table 4). The P2-PA, P3-RL and P5-PV showed Ki values between 1.6 and 2.5 (Table 4). According Khegig (1949), as kaolinite has a Ki value of 2.0, soils with a Ki value near 2.0 are kaolinites. Whereas values much higher than 2.0 are considered slightly weathered and values much smaller than 2.0 are considered strongly weathered. Thus, it is possible to affirm that the soils derived from sandstone in the Cerrado of the Piauí State are newly evolved, and soils derived from sandstones of the Piauí Formation (P2-PA, P3-RL and P5-PV) are more evolved.

While the Ki value of the soil P3-RL suggests a majority presence of kaolinite, this is a soil with many characteristics of a newly evolved soil, such as a deep horizon and the absence of the subsurface diagnostic horizons (Table 1). In addition, many of the Neossolos (Psamments) characteristics are provided directly from parent material, with smaller expression of the chemical weathering and neogenesis (Buol *et al.*, 2011). This suggests that part of these came directly from sandstones of the Canindé Group, given that is possible to notice the presence of kaolinite in sandstone (Shelton, 1964).

All soils presented Kr values higher than 0.75 (Table 4), suggesting that all of them have more kaolinite than oxides (Khegig, 1949). The soils P2-PA, P3-RL and P5-PV showed Fe_d expressively higher than other soils, which also suggests higher content of iron oxides such as hematite, goethite, lepidocrocite or maghemite (Buol *et al.*, 2011). The Fe_d values of the P3-RL reinforce the thesis that its mineralogical compound might be partially provided by parent material. Wu and Caetano-Chang (1992) reported that sandstone can provide lithological iron oxides improving cementation of sandstone. The Fe_o values are low for these soils (0.1 to 0.5), indicating a low occurrence of ferrihydrite and Fe from organic compounds in these soils (Buol *et al.*, 2011). The Fe_o/Fe_d ratio, except for P1-RY that have influence of fluvial sediments, show that studied soils have high content of crystalline iron oxyhydroxides, given that their values are close to 0 (0.1 to 0.3) (Buol *et al.*, 2011). This is a reflection of the pedogenical or lithological environment conditions with good drainage and aeration during the formation of these iron oxides, different from the soil P1-PY, which is influenced by river flooding annually.

4. CONCLUSIONS

The relief and the parent material are the most important soil-forming factors of Cerrado

of the Piauí State, Brazil. The relief is determinant in the thickness and horizon depth. In addition, river flooding has strong influences in soil on the floodplain, providing distinct characteristics. Sandstone is responsible for some pedogenetic standards in these soils, including the high sand content, as well as the low content of bases saturation and clay, which make the soils for this region extremely dependent on organic matter when it comes to soil charges. The soils from this region are young according to the weathering indexes. However, the sandstones from Canindé Group apparently are providing lithological secondary minerals for the soil.

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Deforestation impact on discharge regime in the Doce River Basin

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ABSTRACT

The construction of scenarios using hydrological models can evaluate the hydrological response in watersheds, due to changes in the soil use. In this context, this study analyzed the consequences of deforestation in the hydrological behavior of the Doce River Basin, which has a drainage area of approximately 86.715 km². The basin presents problems regarding water availability, floods, indiscriminate deforestation and inadequate soil management. The Model of Large Basins (MGB-IPH) was selected, using daily data from 1990 to 2014, 11 fluviometric, 81 rainfall and 12 meteorological stations, numerical model of the land, soil maps, and use and land cover. Hydrological modeling was performed in the following steps: calibration of parameters (1990 and 2005), validation (2006 to 2014) and simulation of deforestation scenarios (2000 to 2014). It was observed that the replacement of forests by pasture caused reductions in the average annual flows, indicating a decrease in average flows in deforestation scenarios. As for the behavior of floods, deforestation caused them to increase, while the annual minimum flows reduced with deforestation. The results demonstrate the worsening that the simulated scenarios can cause in the problems already found in the basin, such as floods and water shortages, to supply the uses for which the basin is intended.

Keywords: deforestation, Doce river basin, hydrological simulation.

Impacto do desmatamento no regime de vazões da bacia hidrográfica do rio Doce

RESUMO

A resposta hidrológica em bacias hidrográficas devido às mudanças do uso do solo pode ser avaliada por meio da construção de cenários, usando modelos hidrológicos. Nesse contexto, o objetivo desse trabalho foi analisar as consequências do desmatamento no comportamento hidrológico da bacia hidrográfica do rio Doce, que possui uma área de drenagem de 86.715 km². Alguns dos problemas enfrentados pela bacia estão relacionados à disponibilidade hídrica, inundações, desmatamento indiscriminado e manejo inadequado do solo. Sendo assim, foi selecionado o Modelo de Grandes Bacias (MGB-IPH), onde foram usados dados diários de 11 postos fluviométricos, 81 pluviométricos e 12 meteorológicos, modelo numérico do terreno, mapas de solos e uso e cobertura do solo (2000). A modelagem hidrológica foi realizada nas seguintes etapas: calibração dos parâmetros (1990 a 2005), validação (2006 a 2014) e simulação dos cenários de desmatamento (2000 a 2014). Foi constatado que a substituição de florestas por

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pastagens provocou reduções nas vazões médias anuais, indicando uma diminuição das vazões médias em cenários de desmatamento. Quanto ao comportamento das cheias, o desmatamento provocou o aumento das mesmas, enquanto que às vazões mínimas anuais reduziram com o desmatamento. Os resultados demonstram os agravamentos que os cenários simulados podem acarretar nos problemas já encontrados na bacia como inundações e escassez de água para suprimento dos usos para os quais a bacia se destina.

Palavras-chave: bacia do rio Doce, desmatamento, simulação hidrológica.

1. INTRODUCTION

Anthropogenic activities, such as high standards of living, demographic changes and intense land and water consumptions are pressuring natural resources, undermining sustainability and thereby environmental issues. Pressure on water resources at local, regional and national scales for human consumption, irrigation, energy production, and industrial uses, among others, are steadily increasing. All these pressures imposed on the soil-vegetation-water system impact the hydrological cycle, generating uncertainties in the sustainable maintenance of water resources in watersheds.

Due to difficulties in monitoring and data scarcity, water discharge in a river basin can not be constantly monitored. Thus, the impact of anthropic activity on hydrological variables in watersheds can be evaluated in qualitative and quantitative terms through the construction of scenarios by means of mathematical and physical models associated with geographic information systems (Prado, 2005). As a part of mathematical models, hydrological modeling emerges as a worldwide tool used for several applications, such as the analysis of hydrological response due to changes in land use and occupation (Abbaspour *et al.*, 2015).

The Large Basin Model (MGB-IPH) is a hydrological model that has been applied to determine the impacts of changes in land use on the hydrological regime in different types of climate and vegetation. Caram (2010), Bayer (2014) and Beserra (2016) conducted studies on the impacts of changes in land use in watersheds using MGB-IPH.

Land-use change processes encompass natural vegetation removal or degradation, both associated with significant ecological impacts on practically all scales. Globally, they contribute to greenhouse gas emissions that are associated with global climate change, while regionally and locally, they affect hydrological responses in the watershed (Ibarra-Montoya *et al.*, 2011).

The Doce River Watershed is an example of a basin that has issues related to indiscriminate natural vegetal removal and inadequate soil management. Consequently, the area constantly experiences extreme events (floods and droughts) and water availability and quality issues. This paper therefore estimated flow variations in the Doce River Basin over different deforestation scenarios, using the MGB-IPH.

2. METHODOLOGY

2.1. Study Area

The Doce River Watershed is located in the Southeast region of Brazil, covering part of states of Minas Gerais and Espírito Santo. The basin has a drainage area of approximately 86.715 km², of which 86% belongs to the State of Minas Gerais and the remaining 14% to the State of Espírito Santo (CBH-DOCE *et al.*, 2006). The main headwaters are located in the state of Minas Gerais, in the Mantiqueira and Espinhaço hills, and its water runs about 850 km until reaching the Atlantic Ocean, in the state of Espírito Santo (PIRH- DOCE, 2010).

Cupolillo *et al.* (2008) state that the predominant climate in the basin is classified as humid tropical. The rainy season starts in November and lasts until May with annual precipitation

exceeding 700 mm. Most of the entire basin has high average annual temperatures (above 18°C) during most of the year, even during winter. In general, the river water level follows the rainfall, characterized as perennial. The higher flows occur in the months of December, January, and March, while the lowers occur in August and September (PIRH-DOCE, 2010).

According to PIRH-DOCE (2010), in the Doce River Watershed, the higher specific flows, which mean the water flow per extension of an area, are not directly associated with the larger drainage areas, but with soil type and rainfall periodicity. The basins that are located closer to the headwaters present a higher water production than those in the state of Espírito Santo, indicating a diversity of water availability, which may require different actions for each specific sub-basin.

Floods, which are a major problem in the Doce River Basin, are mainly recorded in the months between December and February. In addition to the intense rainfall that causes flooding, since the begging of the 19th century, the Doce River Basin had its natural vegetation cover (Atlantic forest) removed due to the planting of coffee and sugarcane crops, logging and pasture formation. These actions modify soil infiltration and storage capacity, aggravating the effects of floods, especially in small basins. Along with to this, the disordered occupation of the floodplain areas, especially in urban areas, has aggravated the damages caused by floods (PIRH-DOCE, 2010).

2.2. MGB-IPH Model

The Large Basin Model (MGB-IPH), developed by Collischonn (2001), is a large-scale distributed hydrologic model that performs flow propagation using the Muskingum-Cunge method. The model uses conceptual and physical equations to simulate the terrestrial hydrological cycle: soil water balance; energy balance; evapotranspiration, interception, flow generation (surface, subsurface, and base flow); and flow propagation. In the present study, the MGB-IPH, Version 3.0, was used, which considers a discretization of the basin in irregular units (mini-basins) and Hydrologic Response Units (HRU's), both using the Digital Elevation Model (MDE), land-use map and according to similarity in hydrological behavior (Fan and Collischonn, 2014). The vertical water and energy balances are computed independently for each HRU in each mini basin. The balance is performed considering only one layer of soil, according to Equation 1:

$$\frac{dW}{dt} = P - ET - D_{sup} - D_{int} - D_{bas} + D_{cap} \quad (1)$$

Where: W (mm) is the water storage in the soil layer, P (mm dt⁻¹) is rain that reaches the ground, ET (mm dt⁻¹) is the evapotranspiration, D_{sup} (mm dt⁻¹) is the surface runoff, D_{int} (mm dt⁻¹) is the subsurface runoff, D_{bas} (mm dt⁻¹) is the percolation to the water table, and D_{cap} (mm dt⁻¹) is flow due capillary forces (Collischonn, 2001). The energy and evapotranspiration balance of soil and vegetation are estimated by the Penman-Monteith equation (Equation 2):

$$ET = \left(\frac{\Delta A + \rho_a c_p \frac{D}{r_a}}{\Delta + \gamma \left(1 + \frac{r_s}{r_a} \right)} \right) \frac{1}{\lambda m \cdot \rho_w} \quad (2)$$

Where: λ (MJ kg⁻¹) is the latent heat of vaporization, Δ (kPa °C⁻¹) is the function of saturation vapor pressure gradients, A (MJ m⁻² s⁻¹) is the available energy, ρ_a (kg m⁻³) is the air density, ρ_w (kg m⁻³) is the specific weight of water, c_p (MJ kg⁻¹ °C⁻¹) is the specific heat of humid air, D (kPa) is the vapor pressure deficit, γ (kPa °C⁻¹) is the psychrometric constant, r_s (s m⁻¹) is the surface resistance of the soil cover and r_a (s m⁻¹) is the aerodynamic resistance (Collischonn, 2001). The model parameters that are important to discriminate soil land use and

coverings are: albedo (α), leaf area index (IAF), aerodynamic resistance (r_a), surface resistance (r_s) and the interception coefficient (α), in addition to the parameter W_m , which determines the maximum storage in the soil and must be altered according to the land use and cover typologies (Collischonn, 2001).

The value of the albedo parameter defines the energy that is available for the evapotranspiration process. Deforestation tends to reduce evapotranspiration and increase runoff; thus, deforestation causes an increase in albedo (Collischonn, 2001). The albedo value was assigned from 0.12 to 0.16 for the forest and the albedo value from 0.18 to 0.24 for the pasture, which constitutes deforestation.

Regarding the IAF parameter, the higher the IAF, the greater the maximum interception blade, decreasing the flow. For the forest, the value was assigned from 4.0 to 9.0 and for the pasture, the value from 1.0 to 4.0.

The surface resistance represents the process of the flow of the water from the soil, through the interior of the plant, to the outside of the leaves, besides controlling the sweating process. The higher the surface resistance, the less sweating. In forests, the surface resistance is approximately 100 s / m in pasture, approximately 70 s / m (Shuttleworth, 1993).

As for the parameter of soil storage capacity, this is different for different vegetative cover, because the forests draw water from depths greater than the field vegetation, since the roots of the forest trees reach greater depths. This difference is fundamental in analyzing the hydrological consequences of land use change, especially during periods of drought, because while the volume of water available to the field vegetation is already depleted, the forest vegetation continues to perspire at a normal rate (Collischonn, 2001).

It is likely that parameter b has a strong relation with infiltration capacity; however, it is not possible to directly estimate the values of b based on the characteristics of the vegetative cover. Sensitivity analysis revealed that the higher the value of b , the larger the peaks of small and medium floods; however, b does not profoundly alter the peaks of the highest floods. Basins with deforestation should be represented by the model with higher values of b (Collischonn, 2001).

Both the maximum storage parameter (W_m) and parameter b are used as calibration parameters of the MGB model.

2.3. Input database

The first database required for modeling in MGB-IPH is composed of slope, pedology and land use/cover. The slope data used comes from an SRTM (Shuttle Radar Topography Mission), produced by NASA (National Aeronautics and Space Administration), with a spatial resolution of 90 meters (CGIAR, 2017). For pedology, data was extracted from the Brazilian soil map (EMBRAPA, 2001) in the scale of 1:5,000,000. Finally, for land use and occupation data, a map available from the Brazilian Institute of Geography and Statistics (IBGE, 2000) was used, in a 1: 1,000,000 scale, corresponding to the calibration and validation periods. The agricultural class predominates in the basin, occupying 67.60%, followed by the Atlantic forest class, covering 18.54%.

The other data required for modeling refer to pluviometric, fluviometric and climatological variables. Data from 81 rainfall stations was used for these, and even though there were 60 fluviometric stations inside the basin, only 11 of them were used due to reliability. The historical series of these stations were taken from January 1, 1990, to December 31, 2014, and the data were publically available through the National Water Agency (ANA, 2017). The climatological data (temperature, relative air humidity, wind speed, atmospheric pressure and insolation) were made available by the National Institute of Meteorology (INMET, 2017), totaling 12 meteorological stations. Figure 1 shows the location of the river basin and the stations used.

Figure 1. Location of the fluviometric, pluviometric and meteorological stations used in the simulation.

2.4. Hydrological Modeling

Hydrological simulation began with the spatial data discretization in ArcGIS. The drainage network was defined, and then divided into parts, which allowed the delimitation of the contributed areas for each part (mini-basins). The sub-basins were delimited according to the locations of the fluviometric stations and the basin outlet. As a result, 581 mini-basins and 12 sub-basins were identified.

Soil typologies were grouped in terms of two subjective classes of potential generation of runoff: 1) low potential or deep soils; 2) high potential or shallow soils. The first class comprises soils with low- and medium potential generation for runoff and high potential for water storage (latosol and argisol). The second class includes soils with high runoff generation capacity and low capacity for water storage (neosol, gleysol, and cambisols). The final combination of reclassified soil types was used to generate HRU classes.

The land-use and cover map was also simplified by grouping some of its classes by hydrological similarity in order to facilitate analyses of the influence of vegetative change influence. For reclassification, the "Forest" class was considered to include natural vegetation (Atlantic forest), farm forestry and agroforestry. Similarly, natural and planted pastures were grouped into the "Pasture" class. With the simplified data, the 10 URHs were generated. Figure 2 shows the sub-basins and URHs created.

Figure 2. Sub-basins and HRUs created for the simulation in the MGB-IPH model.

Before the modeling, precipitation and flow data needed to be discretized according to the location of their pluviometric and fluviometric station location in the mini basin. Soon after, the climate data were prepared, in which the climatological averages and the daily climate files were generated. Vegetation data were also prepared, characterizing each HRU by the magnitude of albedo (a), leaf area index (LAI), canopy height (h) and surface resistance (R_s) parameters. The values adopted for each HRU were according to those cited by Collischonn (2001). It is important to mention that, for HRUs that had agricultural land uses, the vegetation parameters values were attributed by means of an arithmetic mean of the values of the parameters of the HRUs that contemplate the uses pasture and agriculture, as was done in the work of Collischonn (2001).

The model was manually calibrated with subsequent automatic calibration. The automatic calibration process occurred only for the sub-basins Number 01, 06 and 08, which obtained inferior performance after manual calibration. The results were compared visually from the hydrograms and flow permanence curves and from three performance metrics: the Nash and Sutcliffe efficiency coefficient (ENS), the R^2 adjusted for the flow logarithm (ENSlog) and the relative error of the total volume of the hydrograms (ΔV). The calibration period used was from 1990 to 2005, while the validation step was the period from 2006 to 2014.

2.5. Deforesting scenarios

A major problem regarding land-use and occupation changes in the Doce River Basin lies in the replacement of the Atlantic Forest biome (natural landscape) into other uses. In order to evaluate the impact that land use and occupation has on the hydrological process, we estimate the discharge in 2000 using the MGB-IPH and compare with other five scenarios based on hypothetical levels of deforestation. The hydrological model considered scenarios where, initially, 100% of the basin was covered by forests. Simulations were then carried out using progressive stages of deforestation (25%, 50%, 75%, and 100%), with pasture in substitution for the existing forest.

In order to analyze the effects of deforestation on the discharge, the simulation was repeated for each scenario considering the same historical period from 2000 to 2014, and climatological and precipitation data. Thus, the effects of the impacts of land-use changes on climate and precipitation were not considered in the paper. The values of the vegetation parameters adopted for the forest and pasture were the same as for calibration, while the soil parameters values were the ones found at the end of the automatic calibration.

3. RESULTS AND DISCUSSION

The calibration process considered 11 sub-basins, since there was no fluviometric station downstream from the sub-basin (11). Thus, the modeling results of sub-basin (11) were analyzed and not those of sub-basin (12). When comparing the results from the validation period with the calibration period, the results were slightly different for most of the stations, and the values estimated by the model showed good agreement with those monitored in the field, according to the performance metrics used. The flow estimations for all the scenarios proposed are presented numerically in Table 1 and graphically in Figure 3.

Table 1. Maximum, minimum and mean annual discharges per year and annual runoff difference compared to the actual situation in 2000, obtained for the period between 2000 and 2014 for sub-basin 11.

SCENARIOS	Q_{Mean} (m ³ /s)	ΔQ_{Mean} (%)	Q_{Max} (m ³ /s)	ΔQ_{Max} (%)	Q_{Min} (m ³ /s)	ΔQ_{Min} (%)	Q_{Med} (mm/ano)	ΔQ (%)
Actual simulation (year 2000)	847,09	-	4495,97	-	316,41	-	3656,76	-
0% Past. and 100% Flor.	985,12	16,29	4055,55	-9,80	483,28	52,74	4275,82	16,93
25% Past. and 75% Flor.	937,92	10,72	4124,24	-8,27	430,43	36,04	4070,43	-4,80
50% Past. and 50% Flor.	890,72	5,15	4198,59	-6,61	377,57	19,33	3865,02	-5,05
75% Past. and 25% Flor.	843,53	-0,42	4274,67	-4,92	324,67	2,61	3659,61	-5,31
100% Past. and 0% Flor.	796,33	-5,99	4353,98	-3,16	271,74	-14,12	3454,22	-5,61

Figure 3. Monthly average discharge (m³/s) of the deforestation scenarios.

According to the results, in the first three scenarios, there was not an increment in the annual average discharge, while there was in situations with 75% and 100% natural vegetation removal. This result was expected, since the land-use/cover map used (year 2000) has 70.29% pasture and agricultural uses, and thus, only the scenarios with the highest percentages of pastures (75% and 100%) would imply in reductions in the annual average discharge. Considering all the scenarios, the one that considers 100% cover of natural forest has the largest increase (16.29% - 142.5 m³/s) in an annual average flow, corresponding to 619 mm increase in annual flow. The average annual outflow for the 100% forest maintenance scenario was 985.12 m³/s. Regarding the maximum annual discharge, in all the scenarios there was a decrease compared to the scenario of 2000. On another hand, the minimum annual flows increased in all scenarios, except in the one with 100% pasture cover.

A 25% deforestation caused a rise in the annual maximum discharge of 68.69 m³/s and a reduction in the annual minimum flow of 52.85 m³/s, and an annual average of 47.2 m³/s, corresponding to a decrease in the outflow average of 205.4 mm/year when compared to the complete forest cover. This result is not consistent with the data found by Bosch and Hewlett (1982) and Collischonn (2001), who estimated, respectively, average increases of 10 to 25 mm and of 9 mm for every 10% reduction of deciduous forest. However, these results were measured in small basins (Bosch and Hewlett, 1982) or in a 26,900 km² basin with high slope and soil type that favors the rapid generation of large floods (Collischonn, 2001). Similarly, when the 50, 75 and 100% pasture scenarios were simulated, there were reductions in the average and minimum annual discharges and a rise in the maximum annual flow.

It was observed that the 100% substitution into pasture caused a 5.99% (50.76 m³/s) reduction in the average annual discharge, while in the scenario of 100% forest caused a rise of 16.29% (138.03 m³/s), when compared to the year 2000 simulation. This result indicates a decrease in annual mean flows in deforestation scenarios, which is in agreement with the average behavior of the results obtained in experimental basins (Bosch and Hewlett, 1982; Collischonn, 2001). However, Caram (2010) and Beserra (2016), when performing hydrological simulations using MGB-IPH in watersheds located in the Brazilian Southeast region, as well as in the Doce River Basin, also found results contrary to the average behavior observed in experimental basins. Additionally, during long drought periods, the average flow tends to suffer more influence from the base flow, which reduced the average annual discharge. The decrease in annual mean flow rates in deforestation scenarios, especially in drought periods, can be explained by the greater capacity of storing water in the soil covered by forests. While the volume of water available for grazing is already depleted, forest vegetation continues to perspire at a normal rate, demonstrating one of the negative effects of deforestation and land conversion on pasture, aggravating the problem of water shortage in the basin.

The results suggest a rise in flood possibilities due to deforestation processes, which are consistent with the results found by Bayer (2014) and Andreassian (2004). On another hand, deforestation process results in the reduction of the annual minimum flows, also are in agreement with the previous papers (Caram, 2010; Beserra, 2016). The increase in floods caused by deforestation can be attributed to the hydrological role of surface water absorption forests and the regularization of liquid flows, avoiding floods. In this way, the replacement of forests by pastures or deforestation causes an increase in the occurrence of floods.

The permanence curve of sub-basin 11 was also analyzed (Figure 4). Related to the impact of deforestation on the minimum flows, we evaluated the Q95 flows (flows associated with the 95% permanence). There was a reduction of 11% in the scenario of 25% of deforestation, 23% in the 50% of deforestation, 34% in the 75% of deforestation and 46% in the 100% of deforestation.

Figure 4. Permanence curve in sub-basin 11.

4. CONCLUSION

The MGB-IPH model had satisfactory adjustments for daily flow simulation in calibration (1990-2005) and validation period (2006 - 2014). It proved to be a powerful tool in hydrological simulations, suitable in the management of the water resources of this basin.

We verified that the replacement of forests by pastures caused reductions in annual average discharge, indicating a decrease in average flows in deforestation scenarios. On another hand, deforestation caused an increment in floods, unlike that of the minimum annual flows, which reduced with deforestation. Although these results are not in agreement with the average behavior of those obtained in experimental basins, they are consistent with studies carried out in other basins located in the Brazilian Southeast region.

Finally, the results from these simulated deforestation scenarios allow the anticipation of deteriorating problems that already exist in the basin, such as floods, due to the increments in maximum flows, and water scarcity with minimum flow reductions.

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Analysis of cloud condition on Sentinel-2 MSI and Landsat-8 OLI images of a public supply lake in Belém-Pará-Brazil

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ABSTRACT

The eutrophication process leads to reduced water quality and economic losses worldwide. Furthermore, it is possible to apply remote sensing techniques for monitoring of aquatic environments. In this paper, we analysed the combined use of Sentinel-2 Multispectral Instrument and Landsat-8 Operational Land Imager data to monitor a eutrophic aquatic environment under adverse cloudy conditions, from July 2016 to July 2018. Data pre-selection was performed, and then the images were acquired for further investigation. After that, we created a key to the interpretation of cloud conditions for the study area and grouped each of 125 scenes in a Principal Component Analysis (PCA). The PCA grouped months with similarities in cloud conditions, highlighting their patterns in terms of the rainy and dry seasons for the study area. Another interesting result was that, even under the inherent adverse cloud regime of the Amazon, the combined use of both free satellite imagery data could be useful for further analyses, such as measuring of chlorophyll a, coloured dissolved organic matters, total suspended solids and turbidity. However, we highlight that, firstly, studies must be made to validate the data in situ, so that monitoring programs can be built through remote sensing applications.

Keywords: amazon waters, bolonha lake, remote sensing.

Análise das condições de nuvens em imagens Sentinel-2 MSI e Landsat-8 OLI de um lago de abastecimento público em Belém-Pará-Brasil

RESUMO

O processo de eutrofização leva à redução da qualidade da água e a perdas econômicas no mundo todo. Além disso, é possível aplicar técnicas de sensoriamento remoto para o monitoramento de ambientes aquáticos. Neste estudo, objetivou-se analisar o uso combinado dos dados Sentinel-2 Multispectral Instrument e Landsat-8 Operational Land Imager para o monitoramento de um ambiente aquático eutrofizado sob condições adversas de nuvens, de julho de 2016 a julho de 2018. Foi realizada a pré-seleção dos dados e posteriormente as cenas foram adquiridas para uma investigação mais aprofundada. Criou-se uma chave de

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interpretação para as condições de nuvens da área de estudo e foram agrupadas cada uma das 125 cenas em uma Análise de Componentes Principais (ACP). A ACP agrupou meses com similaridades nas condições de nuvens, destacando seus padrões em termos de estação chuvosa e seca para a área de estudo. Outro resultado interessante foi que, mesmo sob o regime adverso de nuvens inerente da Amazônia, o uso combinado de ambos dados de imagens de satélites gratuitos pode ser útil para uma análise mais aprofundada, como a mensuração de clorofila a, matéria orgânica colorida dissolvida, sólidos suspensos totais e turbidez. No entanto, destaca-se que, primeiramente, investimentos devem ser realizados de forma a validar dados in situ, para que um programa de monitoramento possa ser construído através de aplicações de sensoriamento remoto.

Palavras-chave: águas amazônicas, lago bolonha, sensoriamento remoto.

1. INTRODUCTION

Eutrophication is a process that causes water deterioration in lentic environments (Lobato *et al.*, 2015) and also affects the spatial distribution of aquatic macrophytes (Madgwick *et al.*, 2011; Søndergaard *et al.*, 2010). These water vegetation contributes to primary productivity, sediment accumulation, provision of food and complex habitat for aquatic and semi-aquatic biota (Poikane *et al.*, 2018). Furthermore, macrophytes act as integrators of environmental conditions to which they are exposed and thus can be used as long-term indicators for water-quality monitoring (Pall and Moser, 2009; Melzer, 1999).

Studies highlighted that current water-quality evaluation is limited due to three main factors: i) in-situ sampling and measurements of water-quality parameters are labour-intensive, time-consuming and expensive; ii) analysis of the spatial and temporal variations and water quality trends in large water bodies is almost impractical; and iii) the collected in-situ data is not 100% accurate considering the possibility of field-sampling or laboratory error (Gholizadeh *et al.*, 2016; Ritchie *et al.*, 2003).

In this context, remote sensing (RS) data and techniques offer a feasible means to explore spatial and temporal information, bringing a variety of insights related to tropical ecosystems (Martins *et al.*, 2018; Yang *et al.*, 2013). Among the RS possibilities for water quality monitoring, it is relevant to mention the use of open source data, such as Sentinel-2 Multispectral Instrument (S-2 MSI) and Landsat-8 Operational Land Imager (L-8 OLI) (Pahlevan *et al.*, 2017; 2019), which we used in the analysis of this paper.

As a risk for future works in monitoring the environment, the US government started the process of reviewing the data fees for Landsat family products (Popkin, 2018). This payment would be a step backward for Earth observation, since more than 100,000 papers have been produced since 2008, when the Landsat products started to be freely available. Stone (2010) reinforces the importance of the free distribution of satellite data, once it has applicability in a vast range of public and private sectors of society.

S-2 MSI is composed of two individual satellites, 2A and 2B; the first one was launched in June 2015 and the second one in March 2017. They have thirteen spectral bands, four at 10 m, six at 20 m and three at 60 m of spatial resolution. L-8 OLI was launched in February 2013. The satellite has nine spectral bands; except for the panchromatic (15 m), all the other bands work at 30 m of spatial resolution. The synergetic use of S-2 MSI and L-8 OLI for monitoring provides a temporal resolution of 2.9 days (Li and Roy, 2017) and, after using image fusion techniques, a spatial resolution of 10 meters, is possible and usable for both images datasets aforementioned (Beltrão and Teodoro, 2018).

Applications with time series of Landsat products are a possibility for mapping and behaviour analysis of small water bodies in the Amazon (Arvor *et al.*, 2018). Landsat historical

series also offers the opportunity to analyse the ecological regime of floating macrophyte greenness throughout vegetation indexes (Terborgh *et al.*, 2018). However, in the Amazon, the seasonal variability of the main atmospheric constituents represents a challenge for optical RS (e.g., cloudiness regime, the high aerosol burden in the dry season, among others) (Martins *et al.*, 2018).

From this perspective, we aimed to analyse the combined use of S-2 MSI and L-8 OLI data in the Brazilian Amazon, considering a eutrophic aquatic environment under adverse conditions of clouds. Our study also aimed to build knowledge related to the possibilities of monitoring aquatic ecosystems in the region.

2. MATERIAL AND METHODS

2.1. Study Area

The Bolonha and Água Preta Lakes supply nearly 63% of the drinking water of the Metropolitan Region of Belém (MRB) (Pará, 2013). Both lakes are refuelled by the Guamá River waters (Bahia *et al.*, 2008). They are located inside the Utinga State Park-Conservation Unit (USPCU) and cover 17.29% of its total area (240.85 out of 1393.088 ha) (Pará, 2013).

One of the main objectives of the creation of the USPCU was to ensure the water potability for the population of MRB (Pará, 2013). However, the pollution of Bolonha Lake due to the immoderate discharge of wastewater is causing its eutrophication and the overgrowth of water macrophytes over its surface (Pará, 2013; Ribeiro, 1992). Figure 1, made in QGIS 2.18.17, shows the location of the Bolonha Lake, used as the object of study.

Figure 1. Location of the Bolonha Lake in Belém, Pará, Brazil.
Source: ESA (2018).

2.2. Image Selection and Key of Interpretation of the Cloud Conditions

We considered all available S-2 MSI and L-8 OLI images from July 2016 to July 2018. We acquired the selected images in August 2018 in the Copernicus Open Access Hub (for S-2

MSI) and the Earth Explorer of the United States Geological Survey (for L-8 OLI). After that, we classified the collected images for the Bolonha Lake in three different cloud conditions: i) lake without cloud cover; ii) lake under partial cloud cover; and iii) lake entirely covered by cloud (Table 1). The S-2 MSI's operations phase images were excluded because the atmospheric correction could not be applied to them; hence, they are not suitable for most of the RS applications.

Table 1. Key of Interpretation applied to the study area. The S-2's coloured compositions in the example column were standardised as RGB: B4, B3 and B2.

Condition	Characteristics/Description	Example
Lake without cloud cover	Cloud-free images of the surface of the lake, including water mass, macrophytes and surrounding primary vegetation (e.g., S2A 20180630)
Lake under partial cloud cover	Images that showed the lake under adverse conditions, but with a clear distinction between macrophytes and water mass (e.g., S2A 20180725)	
Lake entirely covered by cloud	Images not suitable for optical use, due to cloud obstruction of water mass, macrophytes and surroundings of the lake (e.g., S2A 20180426)	

Source: ESA (2018).

This optical analysis was applied for all assessed images of the Bolonha Lake, scene by scene, and, even though there were scenes where the lake was under partial cloud cover, we distinguished which one of those scenes were optically useful for RS applications. The criterion we choose for this was the qualitative aspect of cloud fragments and shadows of cloud over the surface of the lake. With this criterion, we considered scenes as optically useful in situations where the cloud interferences could not affect the distinction of the water mass, the water vegetation and the primary vegetation around the Bolonha Lake.

We applied a quantitative approach, through PAST 3 software, to the filtered data using Principal Components Analysis (PCA) (Finkler *et al.*, 2015; Venkata Krishnamoorthy and Reddy, 2019) for the useful images of the Bolonha Lake. In this analysis, we considered the groups of images classified as “lake without cloud cover” and “lake under partial cloud cover” that surpassed the criterion from the cloud interference as useful data; the remaining scenes summed up to not suitable scene data.

3. RESULTS AND DISCUSSION

3.1. Data Classification and Characterization

In the Bolonha Lake region, most of the S-2A MSI scenes were found in operational phase until June 2016, which left us the option to analyse only the subsequent scenes – in such a way

that the combination of proper S-2 MSI and L-8 OLI images were considered. After the launch and operational phase of S-2B, the number of scenes to be investigated increased considerably.

We found 125 scenes for the study area within the satellite imagery data in the period assessed, from which 77 scenes were from the S-2 MSI satellite, with an annual distribution as follows: 11 from July to December 2016, 27 from January to December 2017 and 39 from January to July 2018. For the L-8 OLI satellite, we found 48 scenes, distributed as follows: 12 from July to December 2016, 23 from January to December 2017 and 13 from January to July 2018. The S-2 MSI and L-8 OLI scenes of the Bolonha Lake were classified and quantified in terms of their suitability for RS applications, as shown in Figure 2.

Figure 2. Classification of the suitability of S-2 MSI and L-8 OLI scenes for RS applications in terms of the cloud condition for all available data of the Bolonha Lake, from July 2016 to July 2018.

Due to its shorter revisit time (5 days) (ESA, 2015), S-2 MSI presented more available data than L-8 OLI (16 days) (USGS, 2016). The different sensing periods for the same area assisted in the selection of the optically useful images, improving temporal resolution for monitoring, agreeing with what is argued by Li and Roy (2017), e.g., in August 2017 there were four cloud-free images of the Bolonha Lake, being two of each satellite.

The gap of optically useful scenes between the months of March and May 2017 occurred because the S-2B satellite was still under the pre-launch phase (ESA, 2015). Furthermore, the L-8 OLI in the same period provided four scenes of the lake entirely covered by cloud and one scene where the Bolonha Lake was under partial cloud coverage – but with almost all pixels over the lake obstructed by clouds – i.e., those data were not suitable for RS applications.

The optical analysis and application of the key of interpretation resulted in 49 scenes of the study area suitable for RS applications, of which 34 were from S-2 MSI and 15 from L-8 OLI. We found around one useful scene per month of the Bolonha Lake (October 2016, March, April and May 2017 and March 2018 were the only months with no usable image found). This implies that the monitoring of aquatic environments using S-2 MSI and L-8 OLI imagery data is feasible even under the inherent excessive cloud regime of the Amazon (Martins *et al.*, 2018).

The results of the PCA of the first (78.6%) and second (21.4%) components, suitable S-2 MSI and L-8 OLI data and total satellite available data, respectively, pointed out three different groups (Figure 3). The similarities were found as follows: i) July to November 2016, January to July 2017 and September 2017 (red); ii) December 2016, October to December 2017 and February to May 2018 (green); and iii) August 2017, January 2018 and June, July 2018 (blue).

Figure 3. PCA illustrating the groups of similar months with overall cloud condition and useful S-2 MSI and L-8 OLI data for the Bolonha Lake.

The red group were assigned together due to the low availability and suitability of satellite data in these months. Prior to September 2017 (Figure 2), the S-2A was operating alone and giving a few imagery data of the study area. The temporal resolution of L-8 OLI could only delivery two scenes per month. Added to those factors, we highlight that the rainy season of USPCU occurs during December until May (INMET, 2019), which corroborates the fact that during this period the images are found covered by cloud more frequently.

The similarity of the green group, except for December 2016, was due to the launch of S-2B satellite, which increased the temporal resolution over Bolonha Lake. This group presented more suitable optical data than the red group, even during the rainy season.

Lastly, the blue group, except for January 2018, was under the dry season in the USPCU area, i.e., the formation and density of clouds over the study area was much smaller than in the rainy season. This characterised a period with high availability of satellite data, in which almost all of them are suitable for optical RS applications; e.g., June and July 2018 both presented 16 available satellite data, from them, 14 scenes were optically useful for analysis of water parameters and its surface macrophytes of the Bolonha Lake.

There are few studies that attempted to demonstrate the suitability of combined S-2 MSI and L-8 OLI imagery (Pahlevan *et al.*, 2019). Furthermore, investigations of cloud patterns usually select bigger areas and use different satellite instruments, such as Moderate Resolution Imaging Spectroradiometer (MODIS) (Garcia-Carreras *et al.*, 2017).

Researches have highlighted different patterns of cloud cover over forested and deforested areas across distinct regions. Ray *et al.* (2003) studied the high occurrence of cumulus clouds over native vegetation areas in Southwest Australia. Teuling *et al.* (2017) analysed the

substantial increase in cloud cover over large forest regions in Western Europe. Wang *et al.* (2009) investigated patterns of shallow cloudiness over deforested areas in Western Amazon. Durieux *et al.* (2003) found increased cloud cover over deforested regions due to the enhancement of seasonal contrasts in Eastern Amazon.

Considering the data from automatic weather stations in Eastern Amazon, Germano *et al.* (2017) analysed the breeze circulations inside USPCU between 2003 and 2012. The cloud-correlated data were precipitation, wind speed and direction. The results showed that the effects of the sea breezes on the precipitation were more evident in Belém and other farther inland cities, which experienced a high frequency of precipitation from 15:00 to 21:00 UTC. Therefore, we can infer that the satellite data for the study area were very likely to be covered by clouds, considering the start sensing period of both S-2 MSI (13:42 UTC) and L-8 OLI (13:22 UTC).

3.2. Optically Active Water Parameters

The goal of most aquatic RS is to extract the radiance of interest from all the other radiance components being recorded by the sensor system. To identify the organic and inorganic constituents in the water column (e.g., chlorophyll a or total suspended solids), it is necessary to isolate the subsurface volumetric radiance (L_v) from the total radiance (L_t) (Equation 1). This process usually involves careful radiometric correction of the remote sensor data to remove atmospheric attenuation (L_p), surface sun-glint and other surface reflection (L_s), the radiance that entered the water column but was scattered into the upper hemisphere before reaching the bottom (L_c) and bottom reflectance (L_b). However, it is only possible to isolate and calculate L_v within a region that is not cloud-shrouded (Legleiter and Roberts, 2005; Jensen, 2014).

$$L_v = L_t - (L_p + L_s + L_c + L_b) \quad (1)$$

RS data and techniques have been widely used to map the open water characteristics and their changes (Zhang *et al.*, 2018). Some of the water parameters optically active and the RS data used for these applications in the literature investigated are detailed in Table 2.

Table 2. Commonly measured and optically active water qualitative parameters using RS.

Water Quality Parameter	References	Satellite/Sensor Data Used
Chlorophyll a (mg L ⁻¹)	Novo <i>et al.</i> (2013) Watanabe <i>et al.</i> (2017)	L-5 TM ¹ S-2 MSI, L-8 OLI
Coloured Dissolved Organic Matters (mg L ⁻¹)	Giardino <i>et al.</i> (2014) Ruescas <i>et al.</i> (2018)	MODIS, L-8 OLI, RapidEye S-2 MSI, S-3 OLCI ²
Total Suspended Solids (mg L ⁻¹)	Umar <i>et al.</i> (2018) Pahlevan <i>et al.</i> (2019)	L-5 TM S-2 MSI, L-8 OLI
Turbidity (NTU)	Rudorff <i>et al.</i> (2018) Sakuno <i>et al.</i> (2018)	L-5 TM, L-8 OLI, MODIS S-2 MSI

¹Landsat 5 Thematic Mapper; ²Sentinel-3 Ocean and Land Colour Instrument.

Gholizadeh *et al.* (2016) described the satellites possibilities for monitoring of water parameters. For these authors, with the use of free Landsat data, it is possible to investigate the main water parameters that have optical responses. At the release date of their paper, the S-2 MSI was recently launched, so there was no trustworthy paper released yet, so the authors did not consider it in their article. However, in Table 2, we elucidate the possibilities of also using the S-2 MSI for the main water parameters estimation. The options of the identified optically

active water parameters, together with the number of images available during the year, per month, opens the opportunity of spatio-temporal monitoring the Bolonha Lake with reduced cost and for constant periods, increasing the understanding of the dynamics of this water body.

4. FINAL CONSIDERATIONS

Our study analysed the optically useful RS data for the USPCU, considering the possibility of further analysis for the study area and its qualitative water parameters. We could conclude that the synergic use of S-2 MSI and L-8 OLI surpasses the cloud issue, granting almost a monthly optical useful image data for small areas, such as Bolonha Lake. However, joint efforts of the public actors interested in the area must be made to validate the data locally during the rainy and dry seasons.

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Influence of organic mulching on drip irrigation management of cabbage cultivation

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ABSTRACT

Irrigation has been pointed out in recent decades as the major consumer of water. Considering the need for a rational use of water resources and the importance of mulching for planting, this study evaluated the effect of organic mulching (sugarcane straw) under soil water storage for cabbage crop development. This research was conducted at CCA/UFSCar, and the experimental design was a completely randomized design with four replications and two treatments with and without sugarcane straw. Water application was performed by a drip irrigation system and irrigation management was determined from data collected by TDR. The use of organic mulching did not interfere with cabbage productivity under greenhouse conditions. The straw presents efficiency for reduction of soil evaporation and, consequently, increases water-use efficiency. The soil cover made it possible to save up to 28.1 mm (14.5%) of the water depth.

Keywords: sugarcane straw, TDR, trickle irrigation, water resources.

Influência da cobertura morta orgânica no manejo da irrigação por gotejamento do cultivo de repolho

RESUMO

A irrigação tem sido apontada nas últimas décadas como o maior consumidor de água. Considerando a necessidade de um uso racional dos recursos hídricos e a importância da cobertura morta para o plantio, o objetivo deste estudo foi avaliar o efeito da cobertura morta orgânica (palha de cana-de-açúcar) sob o armazenamento de água no solo para o desenvolvimento da cultura do repolho. Esta pesquisa foi conduzida no CCA/UFSCar e o delineamento experimental foi inteiramente casualizado, com quatro repetições e dois tratamentos com e sem palha de cana-de-açúcar. A aplicação de água foi realizada por um sistema de irrigação por gotejamento e o manejo da irrigação foi determinado a partir dos dados coletados pelo TDR. O uso da cobertura orgânica não interferiu na produtividade do repolho em condições de casa de vegetação. A palha apresenta eficiência para redução da evaporação do solo e, consequentemente, aumenta a eficiência do uso da água. Foi possível economizar até 28,1 mm (14,5%) de profundidade da água através da cobertura do solo.

Palavras-chave: irrigação localizada, palha de cana-de-açúcar, recursos hídricos, TDR.

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1. INTRODUCTION

In recent years, the intensive and sustainable use of natural resources in agriculture has been the subject of several studies, with water and nutrients being the most limiting factors of agricultural production. According to Urbano *et al.* (2017), agricultural activity is responsible for 70% of the world's freshwater consumption; in Brazil this index reached 67.2% in 2016 and the forecast is that by 2030 the removal of water from natural resources will increase by 30% (ANA, 2017). Irrigation systems need to be operated with high levels of uniformity and efficiency in order to reduce water losses, leading to a rational food production in a growing world population. In this scenario, the search for more efficient irrigation methods that require fewer water resources and provide better results in productivity and quality has been intensified (Santoro *et al.*, 2016).

Localized irrigation has been gaining ground for being one of the most efficient methods of water application, and the drip system has been recommended for most crops. In localized irrigation, water is applied in small volumes and high frequencies directly to the root zone of plants in order to maintain the moisture in the root zone close to the field capacity, allowing the best water use (Bizari *et al.*, 2016). In addition, drip irrigation has several advantages, such as an increased water-use saving, potential to provide optimal management conditions for the development and productivity of plants, possibility of introducing a high level of automation, reducing the use of labor, and reducing the incidence and spread of phytopathogens.

In addition to irrigation management, other cultivation practices are necessary for the crop to produce its maximum potential, such as the use of organic mulching on the soil. Soil coverage with plant residues is presented as an agricultural practice that conserves the natural processes that maintain a stable temperature and reduces natural water losses by the evaporation process on the soil surface, providing an increase in water-use saving (Cortez *et al.*, 2015). In this case, straw acts as a mechanical and thermal barrier on the soil, providing better water conservation (Murga-Orillo *et al.*, 2016). Moreover, this practice allows maintaining or increasing soil organic matter content and mobilizing and recycling nutrients from residue decomposition (Silva *et al.* 2011), being effective for weed control (Moraes *et al.*, 2013).

Considering the need for a rational use of water resources and the importance of mulching for planting, this study evaluated the effect of organic mulching (sugarcane straw) under soil water storage for cabbage crop development.

2. MATERIAL AND METHODS

The experiment was carried out in a greenhouse located in the experimental area of the Department of Natural Resources and Environmental Protection of the Center of Agricultural Sciences of the Federal University of São Carlos (DRNPA/CCA/UFSCar), Araras, SP, Brazil (22°18'00" S, 47°23'03" W, and altitude of 701 m). According to Köppen system, the regional climate is classified as a mesothermal climate with warm and humid summers and dry winters.

The predominant soil in the experimental area is a very clayey Red Latosol (Oxisol) according to the Brazilian Soil Classification System (Santos *et al.*, 2013). The analyses for characterizing soil physical and chemical attributes (Table 1) were conducted at the Laboratory of Soil Fertility and the Laboratory of Soil Physics of the UFSCar following the methods described by EMBRAPA (2011).

The experimental design was a completely randomized design with two treatments and four replications alternately arranged. Treatments consisted of beds with (T1) and without (T2) sugarcane straw, which were used as controls.

Table 1. Physical and chemical characteristics of the soil, 0-0.20 m depth.

Parameters	Units	Content
Sand	%	20
Silt	%	19
Clay	%	61
Field capacity	m ³ m ⁻³	0.34
Permanent wilting point	m ³ m ⁻³	0.17
Total porosity	m ³ m ⁻³	0.51
Bulk density	kg m ⁻³	1300
Soil particle density	kg m ⁻³	2650
Basic infiltration velocity	cm h ⁻¹	13.20
pH H ₂ O	-	6.00
Phosphorus	mg dm ⁻³	68.00
Organic matter	%	28
Potential acidity	mmol _c dm ⁻³	26.00
Potassium	mmol _c dm ⁻³	2.50
Calcium	mmol _c dm ⁻³	31.00
Magnesium	mmol _c dm ⁻³	13.00
Sum of basis	mmol _c dm ⁻³	46.30
Cationic exchangeable capacity	mmol _c dm ⁻³	72.30
Base saturation	%	64
Sulfur	mg dm ⁻³	72.00
Boron	mg dm ⁻³	0.10
Copper	mg dm ⁻³	4.80
Iron	mg dm ⁻³	92.00
Manganese	mg dm ⁻³	1.80
Zinc	mg dm ⁻³	1.40

The greenhouse had eight beds 2.1 m wide and 2.1 m long, totaling an area of 4.41 m². Each bed was composed of 28 plants of the hybrid cabbage Astrus Plus (*Brassica oleracea* L. var. *capitata* L.), whose seedlings were transplanted manually, spaced 0.70 m between rows and 0.30 m between plants in a triangular configuration.

Relative air humidity and temperature were monitored daily over the experimental period by using a meteorological station installed inside the greenhouse. The necessary phytosanitary treatments were also carried out, as well as manual weed elimination.

The amounts of nutrients applied via fertigation were determined considering Table 1 and following the doses according to Raji *et al.* (1997). Planting fertilization was carried out with a solid fertilizer in a half-moon application of 7 g potassium sulfate plant⁻¹, 0.6 g potassium nitrate plant⁻¹, 25 g single superphosphate plant⁻¹, 1.4 g calcium nitrate plant⁻¹, and 0.5 g boric acid plant⁻¹. Topdressing fertilization was carried out via fertigation every fortnight until 60 days after planting, with 1.4 g potassium nitrate plant⁻¹ being applied and 3 g calcium nitrate plant⁻¹ per application.

After manual seedling transplanting and planting fertilization, 9 t ha⁻¹ of sugarcane straw were distributed in the beds (Pereira *et al.*, 2002) (Figure 1).

Water application was performed by a drip irrigation system with 4 L h⁻¹ self-compensating emitters installed near each plant base. Two TDR probes with 0.20 m long rods were buried vertically within the working area of each bed.

Figure 1. Greenhouse after planting fertilization and sugarcane straw distribution and detail in the bed with organic mulching.

Before the beginning of the experiment, a test was performed to verify the uniformity of water distribution of the localized irrigation system. In this test, flow rates were measured in four emitters per bed, one per lateral row. Water application uniformity was estimated using the flow rate of each assessed emitter as a function of the Christiansen's uniformity coefficient (CUC), determined by the Equation 1 described by Andrade *et al.* (2017) and Santoro *et al.* (2016):

$$CUC = 100 \left[1 - \sum_{i=1}^n \frac{(q_i - q_m)}{N_{qm}} \right] \quad (1)$$

Where:

- q_i - flow rate of each emitter ($L h^{-1}$);
- q_m - average flow rate of emitters ($L h^{-1}$);
- N_{qm} - number of emitters.

Irrigation management was determined from data collected daily by using a Campbell Scientific® TDR100, thus maintaining soil moisture in the root system area at field capacity ($0.34 m^3 m^{-3}$) and avoiding water stress of plants. In addition, management was performed separately for each treatment. Readings were registered daily at 7:00 h and irrigation applied three times a day (8h00, 12h00, and 17h00).

TDR probe provided the apparent dielectric constant (K_a) of soil, which was replaced in Equation 2 (Souza *et al.*, 2017), obtained from the calibration for this soil.

$$\theta = 0.000005Ka^3 - 0.0003Ka^2 + 0.0161Ka^1 + 0.0132 \quad (2)$$

Where:

- θ - soil moisture;
- K_a - apparent dielectric constant of soil.

Irrigation water depth was calculated by means of soil moisture content of each treatment, as Equation 3.

$$NID = (\theta_{fc} - \theta_{TDR}) \times d_{eff} \times 1000 \quad (3)$$

Where:

- NID - net irrigation water depth (mm);
- θ_{fc} - soil moisture at field capacity;
- θ_{TDR} - soil moisture obtained with Souza *et al.* (2017) equation;
- d_{eff} - effective root depth (0.25 m).

TDR probe also provided values of soil electrical conductivity. However, Equation 4 (Souza *et al.*, 2017) was necessary to suit the soil used.

$$EC = 0.0303 + (4.602 \times EC_{TDR}) - (0.7 \times \theta) \quad (4)$$

Where:

EC - soil electrical conductivity (dS m⁻¹);

EC_{TDR} - electrical conductivity provided by TDR (dS m⁻¹);

θ - soil moisture obtained with Souza *et al.* (2017) equation.

Cabbage was harvested about 90 days after planting the seedlings, only 10 plants were selected, corresponding to the useful area of the bed, not considering the border plants. Harvested plants were weighed in an analytical balance in order to obtain the fresh mass of each bed.

After harvesting, a foliar analysis was performed to verify the mineral composition of cabbage leaves of both treatments, including the macronutrients nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), and sulfur (S), and the micronutrients boron (B), copper (Cu), iron (Fe), manganese (Mn), and zinc (Zn). For this analysis, samples were obtained by removing four wrapping leaves from the cabbage heads of the useful area of each bed, totaling 32 diagnostic leaves.

At the end of the experiment, the water use efficiency (WUE - kg m⁻³) was estimated through the relationship between productivity of each treatment and the amount of water used during the crop cycle.

The results of soil moisture, water depth, soil electrical conductivity, and fresh mass were analyzed statistically by means of the t-test at 5% significance level using the software R version 3.2.0 (R Core Team, 2015).

3. RESULTS AND DISCUSSION

During the cabbage cultivation cycle, three flow tests were carried out to determine the distribution uniformity of the emitters after a six-minute system run time. It was possible to obtain a uniformity coefficient of 94.1% and an average flow of 4.04 L h⁻¹.

The averages of temperature and relative air humidity during the experimental period were 23°C and 77%, ranging from 18.4 to 27.1°C and 64.7 and 91.9%, respectively (Figure 2). These conditions were favorable for crop growth, as observed by Silva Júnior (1989), who states that cabbage production is favored in mild climate conditions.

Regarding soil moisture variation during the cabbage cycle (Figure 3), sugarcane straw provided 3.7% increase in the average soil water storage, decreasing the water depth to be applied by irrigation when compared to the treatment without organic mulching. A statistically significant difference was observed between the average moisture of treatments, with a p-value equivalent to 0.001367.

Stone and Moreira (2000) observed that mulching efficiency from crop residues is the most important factor to explain the higher water content found in soils under the no-tillage system when compared to the conventional system, since straw acts in the first phase of the water evaporation process, reducing the rate of daily evaporation due to the reflection of radiant energy. In addition, Ferreira *et al.* (2015) reported that the highest water conservation in the mulch system was related to the straw remaining on the soil surface, providing a greater reflection of the incident solar radiation and, therefore, acting as a barrier against water evaporation.

Figure 2. Daily averages of temperature and relative air humidity of the greenhouse during the experimental period.

Figure 3. Variation and average soil water content during cabbage cycle for different treatments (T1- with sugarcane straw and T2 - without sugarcane straw).

The total water depth applied during the cabbage cycle was determined by means of the data collected daily (Figure 4). The total water depth was 165.4 mm with the mulching treatment and 193.5 mm in the treatment without sugarcane straw. Comparatively, a 28.1 mm savings of water was observed in the organic mulching treatment, T1. According to the statistical test, a significant difference was observed in the water depths between treatments, with a p-value equivalent to 0.0002400.

Figure 4. Water depths applied daily during the cabbage cycle.

The values of soil electrical conductivity during the cabbage cycle obtained from data collected through TDR100 are shown in Figure 5. The averages of treatments with and without organic mulching were 0.0198 and 0.0183 dS m⁻¹, respectively. The statistical analysis showed no significant difference between treatments, with a p-value equivalent to 0.8426. Thus, organic mulching did not influence soil electrical conductivity, i.e., it did not retain salts in the soil and did not alter the availability of nutrients to the plants, acting on this variable in a similar way to the treatment without sugarcane straw.

Figure 5. Soil electrical conductivity during the cabbage cycle.

Two electrical conductivity peaks can be noticed in both treatments when analyzing Figure 5. This behavior may be explained by the drip irrigation, which tends to decrease the content of

salts near the plant, thus reducing soil electrical conductivity over the days after the peak, and by the fertigation carried out on March 9 and 23, which contained potassium nitrate. According to Bonini *et al.* (2014), the values of electrical conductivity found are within the limits of normality, since a soil is considered as saline when its values exceed 4 dS m^{-1} , which may impair crop development.

The sugarcane straw provided a greater functionality for the reduction of water evaporation and the economics of the applied water depth, not interfering with the productivity parameter, T1 - 54.2 t ha^{-1} and T2 - 55.2 t ha^{-1} . No significant difference was observed regarding the fresh mass of each treatment, with a p-value equivalent to 0.7904. This result is in accordance with Moura *et al.* (2006), who also observed that the plant stand, productivity, diameter, height, and compactness of cabbage heads were not significantly affected by treatments, i.e., productivity and quality of cabbage in the mulching treatment were similar to those obtained in the treatment without mulching, even with a lower amount of water (Figure 6).

Also, considering the volume of water applied through irrigation, water-use efficiency (WUE) was 32.8 kg m^{-3} in the treatment with cabbage cultivated with mulch and 28.5 kg m^{-3} in plants without soil cover. The WUE result confirms that to produce the same amount of cabbage with straw it is necessary to provide approximately 85.5% of the water demanded for the conventional system. According to Medrano *et al.* (2015), this greater WUE is due to the presence of organic mulching in an adequate amount, reducing the losses by water evaporation from the soil profile. This water saving is in accordance with Freitas and Landers (2014), who reported an average reduction in the demanded water for irrigation of 15% in systems that use mulching on the soil when considering different crops. In fact, straw provides a slower depletion of water in the soil, increasing the irrigation shift and the application of less water during the crop cycle, in addition to favoring a reduction in the operational irrigation costs.

Figure 6. Plants from the treatment without (A) and with sugarcane straw (B).

A possible reason for this insignificant difference in cabbage production may be related to the irrigation management. In this case, irrigation was carried out individually for each treatment in order to maintain the root system area with moisture in the field capacity and with a sufficient amount of water to supply the water requirements of plants, i.e., without excesses or deficits, avoiding plant stress.

No significant difference was observed between treatments for leaf analysis (Table 2).

Thus, the levels of macro- and micronutrients were similar for plants submitted to the treatments with and without organic mulching.

Table 2. Average mineral composition of macronutrients (g kg⁻¹) and micronutrients (mg kg⁻¹) in the cabbage leaves for both treatments.

Treatment	N	P	K	Ca	Mg	S	B	Cu	Fe	Mn	Zn
1	43.50	1.97	16.05	38.41	5.92	8.03	20.25	73.30	168.28	91.56	38.17
2	44.50	1.99	12.80	39.66	6.33	8.53	24.75	11.33	194.72	96.07	24.96

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Other studies have shown results different from those found in our study. Faber *et al.* (2001) found that root system growth in the superficial soil layers can be favored by the presence of mulching, facilitating the absorption of nutrients such as phosphorus, which is accumulated in the superficial layers due to its low mobility. In addition, Bremer Neto *et al.* (2008) stated that the balance between mineralization and immobilization of soil N could be affected by mulching distribution in order to reduce its availability to plants and require complementary fertilization of this nutrient.

The insignificant difference in nutrient content between treatments may be due to the short duration of the experiment. In the study of Mendonça *et al.* (2019), for example, notice that 110 days is a short time to observe soil salinization due to the fertigation in the tomato crop.

4. CONCLUSIONS

The use of organic mulching did not interfere with cabbage productivity under greenhouse conditions. The straw increases the efficiency of irrigation since it reduces soil evaporation, and consequently increases water-use effectiveness. The soil cover made it possible to save up to 28.1 mm (14.5%) of water depth.

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Water quality in Ponte Pensa Aquaculture Park, Solteira Island Reservoir, SP, Brazil, where fish are cultivated under great-volume cage system

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ABSTRACT

This study analyzes spatial and temporal variations in physical and chemical water variables due to the influence of fish culture in a cage system (20x20x3 m) in Ponte Pensa Aquaculture Park (Solteira Island Reservoir), before and after cage installation. Water samples were collected on a monthly basis in the subsurface of three sampling sites (August/2011 to July/2013): downstream from the farming site (S1), at the farming site (S2), and upstream from the farming site (S3) (n = 72). Water temperature, pH, dissolved oxygen, transparency, turbidity, electrical conductivity, ammonium ion and total phosphorus were assessed. The principal component analysis (PCA) was applied to joint analysis data. PCA results explained the 61% joint variability in data of the first two components. Fish farming in cages caused significant temporal changes, mainly in January 2013, as well as sudden spatial changes in water physical and chemical variables. Fish culture influenced the quality of the water; such influence was mainly indicated by variations in ammonium ions and in total phosphorus concentrations, which were confirmed through PCA application. PCA results showed two distinct temporal phases: Phase 1 - prior to fish-cage farming, when limnological conditions were characterized by the highest dissolved oxygen concentrations and by high temperatures; Phase 2 - post-production period, when limnological conditions were characterized by the highest total phosphorus and ammonium ion concentrations.

Keywords: ammonium ion, fish farming in cages, total phosphorus.

Influência da piscicultura em tanques-redes de grande volume na qualidade da água do Parque Aquícola Ponte Pensa, Reservatório Ilha Solteira, SP, Brasil

RESUMO

O objetivo do presente estudo é analisar a variação espacial e temporal das variáveis físicas e químicas da água na zona de influência da tilapicultura em tanques-redes (20x20x3 m) no parque aquícola Ponte Pensa (Reservatório de Ilha Solteira), abrangendo os períodos pré e pós-introdução da produção. Amostras da água foram obtidas mensalmente (agosto/2011 a julho/2013) na subsuperfície em três estações de amostragem: jusante da área de criação (S1), local de criação (S2) e a montante (S3) (n=72). Foram avaliadas a temperatura da água, pH, oxigênio dissolvido, transparência, turbidez, condutividade elétrica, íon amônio e fósforo total. Utilizou-se a PCA para análise conjunta dos dados. A PCA explicou 61% da variabilidade conjunta dos dados nos dois primeiros componentes. As atividades da piscicultura em tanques-rede provocaram alterações temporais, detectadas especialmente a partir de janeiro de 2013, e sutis alterações espaciais nas variáveis físicas e química da água. O íon amônio e o fósforo total foram as variáveis que mais indicaram a influência desta atividade na qualidade da água, confirmados pela PCA que demonstrou duas fases temporais distintas: Fase 1- em que as condições limnológicas antes da produção de peixes em tanques-rede foram caracterizadas pelas maiores concentrações de oxigênio dissolvido e altas temperaturas; e Fase 2 – período pós-produção, que foi caracterizada pelas concentrações mais elevadas de fósforo total e íon amônio.

Palavras-chave: fósforo total, íon amônio, piscicultura.

1. INTRODUCTION

The global population is estimated to reach 9.7 billion people by 2050, and this population growth has increased the yearly demand for food. This scenario is pushing basic production sectors to increase productivity and to expand their production sites (FAO, 2014). Aquaculture is one of the fastest growing activities in the food industry worldwide; it reached the historical record of 90.4 million tons in 2012, and fish farming in cage systems accounted for 66.6 million tons of this total (FAO, 2014).

According to the Brazilian Institute of Geography and Statistics (IBGE), fish farming in continental waters in the country totaled 507.12 t in 2016; it increased by 4.4% in comparison to the total fish production in 2015 (IBGE, 2016). The farming of tilápias increased by 9.3% in the same period (239.09 milt, in 2016). São Paulo State ranked third among domestic producers at 48.35 t, a 47.5% increase in comparison to the previous year. Santa Fé do Sul, Northwestern São Paulo State, ranked first in the national ranking of counties, with 5.360 t (IBGE, 2016; Barroso *et al.*, 2018). This production growth resulted from the expansion of projects focused on assembling cages for fish farming in aquaculture parks installed in mid- and large-sized hydroelectric power-plant reservoirs (Mallasen *et al.*, 2012).

An aquaculture parks is defined as a continuous physical space within a delimited water medium, which covers a set of similar water sites whose intermediate physical spaces allow the development of other activities along with aquaculture practices. In their turn, aquaculture sites are continuous physical spaces in delimited water media used for aquaculture (Brasil, 2003).

The first aquaculture parks to become operational were located in Itaipu (PR), Castanhao (CE), Solteira Island (MS, MG and SP), Furnas (MG), Tres Marias (MG) and Tucuruí (PA) reservoirs. These six reservoirs rely on 42 aquaculture parks that together comprise 28,500

hectares of water, whose production capacity is able to supply approximately 269 thousand tons of fish per year to the market (Araújo, 2016).

Nowadays, there are aquaculture parks operating in reservoirs all around the country; 15 sites in Solteira Island Reservoir were categorized as appropriate for aquaculture park implementation, among them, Ponte Pensa Aquaculture park, whose support capacity is to 4.59 t/year (David *et al.*, 2015). Ponte Pensa Aquaculture Park, as well as other ones, were implemented in Brazilian reservoirs based on studies that have taken into account some environmental, sustainable, social and economic aspects. Results in these studies have pointed out good water quality conditions for aquaculture in cage system in these sites (Bueno *et al.*, 2008; David *et al.*, 2015).

Fish farming in cage systems is an intensive production modality, but it has high potential to cause environmental impact, since feed waste and fish excreta are released into the aquatic environment. These discharges increase nitrogen and phosphorus concentrations in the water and lead to artificial eutrophication processes (Guo and Li, 2003; Alves and Baccarin, 2005; Bueno *et al.*, 2008; Guo *et al.*, 2009; Mallasen *et al.*, 2008; 2012).

Fish cage farming influence on water limnological characteristics depends on fish production intensity, on waste dispersion, on the quality of the feed and on the assimilation ability of the environment where the cages are installed in (Borghetti and Ostrenky, 1999; Mallasen *et al.*, 2012; Ayroza *et al.*, 2013).

Some studies (Fernandes *et al.*, 2001; Piedrahita, 2003; Borges *et al.*, 2010; Mallasen *et al.*, 2012) emphasize the importance of assembling cages in reservoirs presenting favorable hydrodynamic characteristics, since these features allow nutrient dispersion from the organic load in the production system and water quality disturbance assimilation in sites used for fish farming. Therefore, the definition of aquaculture parks, such as Ponte Pensa, is essential for aquaculture development planning, since farming sites are delimited based on multidisciplinary studies (morphometry, climatology and hydrodynamics.) aimed at determining hydrodynamically favorable sites for fish farming in cage systems (David *et al.*, 2015).

Studies that have evaluated the influence of fish-cage farming on the limnological characteristics of sites where this system is operational help in better understanding the dynamics of the cage system and its relationship with the environment. Moreover, they highlight the efficiency of hydrodynamic characteristics observed in delimited sites.

The aim of the present study was to analyze spatial (horizontal) and temporal (monthly) variations in water physical and chemical variables in sites influenced by fish farming in great-volume cage systems (1,200 m³; 20x20x20 m), such as Solteira Island Reservoir (Ponte Pensa Aquaculture Park), before and after farming site implementation. It is worth highlighting that these great-volume cage systems have been recently installed in Brazil.

The evaluation of the environmental variability of abiotic data in relation to the (temporal) months of study and the sampling stations (spatial) was performed through principal component analysis (PCA). The PCA is a multivariate statistical technique that transforms a set of original variables into a smaller set of independent variables called principal components, which in turn retain the maximum information of the total variation contained in the data (Hongyu *et al.*, 2015).

2. MATERIALS AND METHODS

The study was carried out in Fish Farm Geneseas Aquacultura Ltda, which is located in Ponte Pensa Aquaculture Park. This park holds part of Ponte Pensa River, which feeds Solteira Island hydroelectric power plant reservoir, SP, Brazil (Figure 1). The location and characteristics of Solteria Island Reservoir and of Ponte Pensa Aquaculture Park were addressed by Rosini *et al.* (2016).

2.1. Climate Variables

Rainfall and air temperature information for the period between August 2011 and July 2013 in the assessed region was provided by the Integrated Center for Agrometeorological Information (CIIAGRO) of the Department of Agriculture and Supply of São Paulo State.

2.2. Sample collection and collection period

Sample collection started in August 2011, five months before cage system of fish farming was operational. The first cages were installed in Jan/Feb 2012; there were two modules of eight large cages ($1,200 \text{ m}^3$; $20 \times 20 \times 3 \text{ m}$) in the site at the end of the experimental period.

A "van Dorn" collecting bottle was used to collect water samples on a monthly basis from the subsurface of sites impacted by fish-cage farming for two years (August 2011 to July 2013) ($n=72$). The samples were used in the analysis applied to find the limnological conditions (total phosphorus ($\mu\text{g L}^{-1}$) and the ammonium ion concentration ($\mu\text{g L}^{-1}$). Three sampling sites were evaluated: S1 (750 m upstream from the site with the cages downstream in the Ponte Pensa River, coordinates $20^\circ 16' 134'' \text{ S}$ and $50^\circ 59' 107'' \text{ W}$, mean depth 30m), S2 (the farming site, coordinates $20^\circ 16' 452'' \text{ S}$ and $50^\circ 58' 812'' \text{ W}$; mean depth 27 m), and S3 (650 m downstream from the site with cages upstream in the Ponte Pensa River, coordinates $20^\circ 16' 853'' \text{ S}$ and $50^\circ 58' 980'' \text{ W}$, mean depth 26 m) (Figure 1). Sampling sites were delimited with GPSMAP 76CS / Garmin.

Figure 1. (A) The arrow indicates the location of Solteira Island Reservoir in São Paulo State, Brazil. (B) The rectangle indicates the location of the Ponte Pensa River arm in the Solteira Island Reservoir. (C) Genesee fish-cage farming site ($20^\circ 17' \text{ S}$ and $50^\circ 58' \text{ W}$) is delimited by the dark line and the Ponte Pensa River arm is marked by the light line. (D) Location of sampling sites S1, S2 and S3.

Source: Rosini *et al.* (2016).

2.3. Analysis of Water Physical and Chemical Variables

Water temperature profile ($^{\circ}\text{C}$), pH, electrical conductivity ($\mu\text{S cm}^{-1}$), water turbidity (NTU) and dissolved oxygen (mg/L) values were obtained *in situ* with the water quality multiparameter meter YSI Professional Plus (ProPlus). Water transparency (m) was determined based on Secchi disk disappearance depth; total phosphorus ($\mu\text{g L}^{-1}$) and ammonium ion ($\mu\text{g L}^{-1}$) concentrations were analyzed based on Valderrama (1981) and Solorzano (1969), respectively.

The collected data were compared to values established by CONAMA Resolution N. 357/2005 (CONAMA, 2005) in order to verify whether or not they remained within the limits for Class II water bodies, which include aquaculture sites.

Monthly data about the amount of consumed feed were provided by Geneseas Aquacultura Ltda to assess feed waste influence on the quality of water.

2.4. Statistical Analysis

Abiotic data were subjected to joint evaluation based on the principal component analysis (PCA); the analyses were carried out in the PC-ORD software, Version 4.0, for Windows (McCune and Mefford, 2011). Data were transformed through $[\log(x + 1)]$.

3. RESULTS AND DISCUSSION

Based on rainfall and air temperature data, the assessed region counts on two specific seasons: wet summer and dry winter. The lowest rainfall values (below 10 mm) were recorded in winter - August/September 2011, July/August 2012 and July 2013 -, whereas the highest values for this variable (above 214 mm) were recorded in summer, January 2012 and January/March 2013 (Figure 2).

Figure 2. Mean monthly air temperature ($^{\circ}\text{C}$) and rainfall (mm) variation in Santa Fé do Sul/SP during the study period.

There were no significant abiotic changes between sampling sites. Water temperature was close to 26.5°C ; the lowest temperatures were recorded in July/2012 (22.6°C) and the highest in February/2012 (30.7°C) (Figure 3A).

There was a drop in dissolved oxygen (DO) values at Station 2 in February 2012, after the fish-cage farm became operational. The lowest DO concentrations were observed at this site, mainly after the second half of 2012 (Figure 3B), although the recorded values (above 4.0 mg L^{-1}) remained in the specifications recommended for tropical fish farming (Boyd, 1990; Kubitz, 2000) and within the limit established by CONAMA Resolution N. 357/2005 for Class II water bodies, including aquaculture sites.

Figure 3. Temporal variation in the assessed variables - water temperature ($^{\circ}\text{C}$) (A), dissolved oxygen (mg L^{-1}) (B), pH (C) and water transparency(m) (D) - at the collection sites throughout the study period.

According to the recorded pH Values, water at the site was alkaline; but these values decreased in February 2012 (Figure 3C) and became similar to the ones recorded for dissolved oxygen at Station 2. Transparency values ranged from 2.0 to 8.3 m. There was a decrease of approximately 4.0 m in water transparency in March, April and May 2012 and 2013, in comparison to months analyzed before this time. Water transparency (2.0 m) recorded the lowest values in March 2013 at Station 2 (Figure 3D). Water turbidity varied between 1UNT and 8UNT; different from water transparency, it had significant increase in March, April, May and June 2012/2013 in comparison to the other months (Figure 4A).

Conductivity values were always lower than $55 \mu\text{S cm}^{-1}$; however, in May, June and July 2013, there was sudden conductivity increase in comparison to the other months (Figure 4B). Ammonium ion concentrations showed remarkable increase in the last seven collection months at the three collection sites (January to July 2013); rates reached values 100% higher than the ones recorded in previous analyses (Figure 4C). Total phosphorus concentrations also increased throughout the study period, mainly in September 2012. These concentrations were overall higher in 2013 in comparison to values recorded in the same period in 2012 (Figure 4D). Although the analyzed variables recorded little variation in the sampling sites since they became operational (February 2012), total phosphorus concentrations were higher than $30 \mu\text{g L}^{-1}$ at Station 2 (fish-cage farming site) (Figure 4D).

With regard to feed quality, 146 tons of feed were supplied on a monthly basis, on average; the largest amounts of it were supplied in the last months of the study period (May, June and July 2013) (Figure 4E).

Principal component analysis (PCA) explained the 61% joint variability in data of the first two components; this outcome indicated seasonality as the factor coordinating the abiotic changes between August 2011 and December 2012. All sampling units in 2011 and most units in 2012 were on the negative side of axis 1, which is associated with the highest dissolved oxygen (DO) ($r = 0.8$) and pH ($r = 0.6$) values (Figure 5). On the other hand, all sampling units in 2013 were grouped on the positive side of axis 1, which is associated with the highest ammonium ion (NH_4) ($r = 0.8$) and total phosphorus (TP) ($r = 0.5$) concentrations. The highest turbidity (Turb) ($r = 0.6$) values were associated with the highest conductivity (Cond) ($r = 0.4$) values, although at a low correlation (Figure 5). Sampling units in January, February, March, October, November and December 2012 were grouped on the positive side of axis 2, which is associated with the highest water temperatures ($r = 0.8$) (Figure 5).

Based on PCA records, there were two distinct time phases: Phase 1 – when limnological conditions before the fish cage farm was operational (August and December 2011) were characterized by the highest dissolved oxygen concentrations; Phase 2 - post-production period (January to July 2013), which was characterized by the highest total phosphorus and ammonium ion concentrations and by high conductivity and turbidity values (Figure 5). The first and the last quarter of 2012 were associated with the highest temperatures.

According to PCA results, the analyzed abiotic variables were influenced by seasonality. Other authors also pointed out seasonality as one of the factors influencing variations in abiotic factors, mainly water transparency, in studies similar to the current one (Alves and Baccarin, 2005; Borges et al., 2010; Kaggwa et al., 2011; Ayroza et al., 2013; Bartozek et al., 2014).

Although water transparency values were similar in all sites sampled in March, April and May 2012 and 2013, this variable recorded 4.0 m decrease in comparison to the other months in the same time period. Such decrease was not directly related to phytoplankton density increase, since phytoplankton densities were always very low during the whole period (lower than $1,933 \text{ org mL}^{-1}$, as recorded by Rosini et al. (2016)). Summer rainfall has likely influenced transparency by transporting allochthonous material through runoff, a fact that makes water more turbid due to the suspended material.

Figure 4. Temporal variation in the assessed variables - turbidity (UNT) (A), electrical conductivity ($\mu\text{S cm}^{-1}$) (B), ammonium ion concentrations ($\mu\text{g L}^{-1}$) (C) and total phosphorus ($\mu\text{g L}^{-1}$) (D) - at the collection sites during the study period; (E) Tons (t) of feed supplied to the cages in Geneseas Aquacultura, during the study period.

Figure 5. Principal components analysis (PCA) based on seven limnological variables of 72 sampling units (temporal and spatial). Abiotic variables: temperature (Temp), dissolved oxygen (DO), electrical conductivity (Cond), turbidity (Turb), pH, total phosphorus (TP) and ammonium ion (NH_4). Sampling units were identified based on collection year (Δ = 2011, $\circ$ = 2012, $\square$ = 2013), month (Ja: January, F: February, Mr: March, A: April, My: May, Jn: June, Jl: July, Aug: August, S: September, O: October, N: November, D: December) and sampling site (Station 1 = 1, Station 2 = 2 and Station 3 = 3).

Although rainfall influence on transparency should be taken into consideration, fish-cage farming influence should not be ruled out, since there was a decrease in transparency in the last months of the study period (March to June 2013) in comparison to transparency values recorded in the same months in 2012. Despite the high rainfall values observed in February and March 2013, the amount of consumed feed almost doubled in the same months of 2012; this outcome may have contributed to increase the concentration of suspended solids, both due to feed residue and to increased volume of fish feces. These results corroborate Ayroza *et al.* (2013), who recorded higher concentrations of total dissolved solids, high turbidity values and lower water transparency as the amount of feed supplied to fish farms working under cage systems increased.

Dissolved oxygen values were similar to the ones recorded by Mallasen *et al.* (2012) in the same study site chosen for the present research and by Américo *et al.* (2013), who conducted their research in the São Jose dos Dourados River, an area influenced by cages assembled in the Solteira Island Reservoir. Dissolved oxygen values were higher than those recommended for tropical fish farming (4 mg L^{-1}) (Boyd, 1990; Kubitza, 2000) and within the limit established by CONAMA Resolution N. 357/2005 for Class II water bodies, including aquaculture sites. After the introduction of the first cages in the farming sites in February 2012, DO values dropped at Station 2, which was used for fish farming. Several studies (Demir *et al.*, 2001; Veenstra *et al.*, 2003; Alves and Baccarin, 2005; Bristow *et al.*, 2008; Degefu *et al.*, 2011; Kaggwa *et al.*, 2011; Ayroza *et al.*, 2013) have already recorded DO concentration reduction in cage farming sites.

Conductivity values did not exceed $55 \mu\text{S cm}^{-1}$. According to CETESB (2009), conductivity values above $100 \mu\text{S cm}^{-1}$ indicate impacted environments; therefore, the herein-assessed environment was not characterized as impacted during the study period.

Similar to the study by Minello *et al.* (2010), electrical conductivity values tended to

increase during the rainy season, different from what was observed by Alves and Baccarin (2005). Some authors argue that rainfall has a diluting effect, and such dilution could decrease the electrical conductivity values (Alves and Baccarin, 2005; Crossetti and Bicudo, 2005). However, the input of a larger volume of water can increase the amount of dissolved solids transported to the reservoirs through the leaching process; that is likely why higher DO values were recorded in the rainy season. Although there was seasonal influence on electrical conductivity, there was fish-cage farming influence, mainly in the last three months of the study (May to July 2013). The increased conductivity recorded in 2013 in comparison to the same period in 2012 may have happened due to increased ammonium ion concentrations. Higher ammonium ion concentrations can be related to the larger volumes of fish feces and excreta in the water, which result from the installation of new cages and, consequently, from increased fish density in the study site, and from greater feed supply.

Feed waste and fish excreta decompose into ammonium, and total ammonium results from ionized ammonium (NH_4^+) and from non-ionized ammonium NH_3 (Rojas and Sanches, 2006). The presence of dissolved ammonium ions (NH_4^+) mainly depends on pH; when pH is close to 7, or neutral, ammonium is in its ionized form (NH_4^+); when pH is close to 9.5, approximately 50% of the ammonium in the water is in the NH_3 form, and 50% of it is in the NH_4^+ form; when pH is higher than 11, ammonium is in its non-ionized form (NH_3), which is a toxic form for fish and other living things (Koren *et al.*, 2000).

Based on the recorded pH values, ammoniacal nitrogen was within the appropriate range to keep the balance between NH_4^+ and NH_3 fractions, with prevalence of the ionized form (NH_4^+).

Ammoniacal nitrogen concentrations were below the value recommended for fish production - 0.6 mg L^{-1} - (Boyd, 1990); however, there was a gradual increase in the concentration of ammonium ions one year after the cages were installed. There was more than 100% increase in comparison to values recorded for previous months, probably due to the increased ammonium excretion by the fish (feces and urine). The increased amount of feed supplied in the last months of the study confirmed this information and corroborated the herein-recorded results. Villarreal and Torres (2011) and Fu-Guang *et al.* (2009) reported that 60% to 80% of the total nitrogen excreted corresponds to ammonium.

The increased organic matter, nitrogen and phosphorus concentrations in the water are some of the main aspects to be taken into consideration in fish-cage farming, mainly because of the feed fish do not ingest, and because of fish feces and urine (Guo and Li, 2003; Mercante *et al.*, 2004; Alves and Baccarin, 2005; Guo *et al.*, 2009; Américo *et al.*, 2013). Accordingly, increased total phosphorus concentrations in the water are a major issue and has been documented by several studies carried out in sites used for fish-cage farming (Guo and Li, 2003; Alves and Baccarin, 2005; Bristow *et al.*, 2008; Bueno *et al.*, 2008; Guo *et al.*, 2009; Mallasen *et al.*, 2012). The results recorded here corroborate the aforementioned studies, since there was a gradual increase in total phosphorus concentrations after the fish farm in cage system was operational. The highest phosphorus concentrations were recorded in Station 2, which showed values higher than the ones established by CONAMA Resolution 357 for water used for aquaculture ($30 \text{ } \mu\text{g L}^{-1}$), thus indicating the remarkable influence of aquaculture activities on the quality of the water.

Results confirmed by PCA showed that ammonium ions, total phosphorus, turbidity and electrical conductivity were the cage-farming variables having the strongest influence on the quality of the water in Ponte Pensa Aquaculture Park. Based on this outcome, the highest ammonium ion and total phosphorus concentrations, and electrical conductivity were observed in the last seven months of the study, when there was increased feed supply (farming intensification). These values suggest increased fish production and, consequently, increased feed waste, as well as larger volumes of feces and excreta.

4. CONCLUSIONS

Fish farming in cage systems located in Ponte Pensa Aquaculture Park, Solteira Island Reservoir, Santa Fe do Sul County, Brazil, caused significant temporal and sudden spatial changes in water physical and chemical variables. Ammonium ion and total phosphorus concentrations increased over time, mainly at fish-cage farming sites.

Short residence time (21.6 days) and flow rate ($172 \text{ m}^3\text{s}^{-1}$) likely allowed exporting nutrients downstream (Paraná River) and improved the capacity of the environment to assimilate disturbances in the quality of the water caused by the production process. Along with fish-cage farming management, these variables were decisive to mitigate the impact of fish feed and metabolism organic load on the studied aquatic system and on the spatial similarity between sampling sites.

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ABSTRACT

Sustainability encompasses several aspects, one of which is the reuse of nutrient-rich effluent. In recent years, improper disposal of pharmaceuticals and pharmaceutical effluent, which contain recalcitrant compounds, has caused great environmental damage. An alternative disposal strategy that has been studied for several residues is their reuse for agricultural purposes. This work evaluated the potential of using pharmaceutical effluent for fertilizing *Schinus molle*, a native Brazilian species locally known as “aroeira-salsa.” Effluent was pretreated by chemical precipitation, and the solid phase was tested as fertilizer. Seedlings were grown in soil or compost. The soil used was classified as Red-Yellow Acrisol with medium loamy texture and high Al content, which is typically found in the Vale do Paraíba region, São Paulo, Brazil. Two agricultural composts were used as substrate, both from the Taboão Ecological Park located in Vale do Paraíba. Fertilizer application improved the physicochemical characteristics of all growing media, increasing C, N, P, K, Mg, and Ca contents, base saturation, and pH, while decreasing Al content and density. Fertilization improved nutrient absorption and plant growth.

Keywords: fertilizer, pharmaceutical effluent, sustainability.

Avaliação do uso de fertilizante obtido a partir de efluente de indústria farmacêutica

RESUMO

A sustentabilidade envolve um número grande de aspectos. Entre eles, o de aproveitamento de nutrientes presentes em efluentes tratados com diferentes tipos de processos. Nos últimos anos, têm sido detectados danos ambientais decorrentes do despejo de fármacos e de efluentes da indústria farmacêutica, em função da difícil recalcitrância de alguns componentes das formulações. Um destino que tem sido estudado para diversos tipos de rejeitos é a aplicação no solo com finalidade agrícola. Visando o aproveitamento de efluentes da indústria farmacêutica

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(e consequente redução de um passivo ambiental), foi desenvolvido e utilizado um fertilizante obtido a partir da precipitação química (pré-tratamento) de um efluente. O fertilizante obtido foi testado para espécie *Schinus molle*, popularmente denominada de aroeira-salsa no Brasil. As plantas se desenvolveram tendo como substrato solo ou compostos orgânicos. O solo utilizado foi um Argissolo Vermelho-Amarelo textura média, característico da região do vale do Paraíba, Estado de São Paulo, Brasil, com alto teor de alumínio. Foram utilizados dois compostos orgânicos, originados de resíduos agrícolas, ambos provenientes do Parque Ecológico do Taboão, localizado no Vale do Paraíba. A aplicação do fertilizante em todos os substratos melhorou as suas características em relação aos teores de C, N, P, K, Mg, Ca, saturação por bases, pH, Al e densidade. Também a absorção de nutrientes e o crescimento das plantas foi beneficiado com a aplicação do fertilizante.

Palavras-chave: efluente de indústria farmacêutica, fertilizante, sustentabilidade.

1. INTRODUCTION

Environmental sustainability involves multiple aspects. One is the reuse of waste materials rich in nutrients and organic matter. Adequate disposal of nutrient-rich effluent prevents chemical and microbiological contamination and reduces environmental impact. Incorporation of wastewater into the soil or other agricultural substrates for fertilization purposes is an interesting alternative from a sustainability and environmental conservation perspective. Wastewater treatment can produce profitable materials, thereby favoring sustainable economic activities and fostering the creation of economically viable environmental programs and new waste disposal sites for industries. Such projects reduce environmental pollution and impacts and raise public awareness of environmental issues (Gouveia, 2012).

Effluents must meet several requirements prior to discharge into water bodies, according to CETESB Article 18 (CETESB, 2002) and CONAMA Resolution No. 430 (CONAMA, 2005). Thus, adequate treatment of wastewaters is of extreme importance for environmental preservation and law compliance. In some cases, depending on the recalcitrance of its components, wastewater may require a post-treatment step to meet discharge standards, such as biological treatment.

Pharmaceutical industries generate large amounts of waste with high organic content. Residues resulting from the production of antibiotics are an example of materials that contain recalcitrant components of high molecular weight. At high concentrations, these compounds are difficult to treat by conventional methods. For this reason, pharmaceutical wastewater is frequently dried and incinerated rather than treated, especially when the cost/benefit ratio is high. It thus becomes necessary to resort to emerging technologies or to a combination of two or more processes (for instance, chemical precipitation and advanced oxidation process) to treat pharmaceutical wastewater.

Discharge of pharmaceuticals and untreated pharmaceutical wastewater into domestic sewer lines, surface water, and groundwater has caused great environmental damage (Melo *et al.*, 2009). These wastes contain compounds of several therapeutic classes, such as hormones, analgesics, and antibiotics.

The agricultural industry generates large amounts of solid waste, including straw, crop residues, and animal wastes. Hundreds of metric tons of organic waste are generated annually in Brazil, which can be either a serious source of pollution if untreated or a valuable agricultural input to replace industrialized fertilizers.

Composting, one of the most environmentally friendly alternatives for recycling organic waste, is a controlled process of organic waste degradation under aerobic conditions. Compost can supply essential nutrients, especially carbon, for plant growth and also improve some physical characteristics of the soil, including soil density, a property that varies according to

soil compaction. The decrease in soil density promoted by the addition of organic matter facilitates root penetration and increases total porosity, macroporosity, water permeability, and water infiltration (FAO, 2005).

An environmentally sustainable agricultural system requires that soil nutrient and organic matter reserves be preserved. Carbon and nitrogen are important for plant growth. Stable soil organic matter is a source of essential nutrients for plant development (LAL, 2008; Misiewicz *et al.*, 2017).

Achieving sustainability is the goal of various agricultural production systems (Karlen and Rice, 2017). Agroforestry, for instance, has seedling cultivation and tree planting as some of its core practices. Different substrates are used to grow seedlings until they can be planted. Substrate is a medium for growing plants *ex situ*. Mineral soil was the first substrate used for *ex situ* cultivation. Currently, most substrates consist of a combination of two or more components, which can be chosen from a large variety of materials.

Many studies showed that soil amendment with organic waste positively influences soil characteristics and plant development. Gomes *et al.* (2004) conducted a study on the cultivation of *Anadenanthera colubrina* in Red-Yellow Acrisol and reported that nutrient addition promoted an increase in base saturation, which in turn improved plant growth and resulted in increased dry weight. Cultivated species require the addition of adequate levels of nutrients to the culture medium. Soil organic matter is the primary source of nutrients in cultivated soils and serves as a temporary nutrient reserve. Its importance in maintaining and improving physical characteristics of the soil, such as water and air infiltration, water retention, and soil aggregation, and in controlling the fate of pesticides is well known (Srinivasan *et al.*, 2012).

Maintaining and increasing soil organic matter levels in the highly weathered soils of tropical regions is a great challenge. In tropical agricultural areas, organic residues and fertilizers are frequently applied to the soil to enhance microbial activity and mineralization rates (Guimarães *et al.*, 2014).

Studies analyzed the effects of wastewater or effluent irrigation. Depending on the characteristics of the material, either positive or negative effects may follow. A comprehensive review of this topic was published by Kaur and Sharma (2013). The study provided a list of effluents that can be used for fertilization purposes but underscored that some may negatively impact physical and chemical soil characteristics. Kalyva (2017) presented a review of the disposal of pharmaceuticals in the environment, discussing their fate, transport, aquatic and terrestrial pollution potential, and risks to populations. Probably because of the well-known negative impacts of pharmaceuticals on the environment, few works focus on investigating the beneficial effect of pharmaceutical wastewater irrigation.

This study aimed to evaluate the potential of using a fertilizer obtained from pharmaceutical wastewater to grow *Schinus molle* in different substrates. The effects of fertilizer addition on chemical and physical characteristics of substrates, plant growth, and nutrient absorption were evaluated.

2. MATERIALS AND METHODS

Pharmaceutical effluent, specifically, effluent from syrup production, was used to obtain the fertilizer. In addition to pharmaceuticals, the effluent had a high content of sucrose, N, K, Mg, Na, and P (in the form of phosphate). The presence of these elements and the high C content (present in sucrose) makes this material a potential source of plant nutrients.

As the effluent had a high organic load, treatment by oxidation processes would not be feasible. Organic matter was therefore chemically precipitated via an innovative process. Sucrose was the major component of the precipitate. Detailed information about the precipitation reaction will not be presented in this paper because the process is being patented. After precipitation, pretreated wastewater was submitted to vacuum filtration to separate the

solid phase from the liquid phase. A calcium hydroxide ($\text{Ca}(\text{OH})_2$) solution was added to the solid phase at 1:3 (w/w). The mixture was oven-dried at 105°C to avoid chemical and microbiological degradation during storage. The dried material was ground and sieved through 2 mm sieves, affording the fertilizer used in plant experiments. Grinding and sieving provides powders with standardized particle sizes and promotes particle homogenization, which improves soil reactivity. The liquid phase obtained after filtration was not used in this work. It will be treated by an oxidation process to meet the legal requirements for wastewater discharge. Figure 1 shows a flow diagram of the process used to obtain the fertilizer. The process can be used in an industrial setting.

Figure 1. Scheme for obtaining fertilizer from untreated pharmaceutical effluent. Process A is used in an innovative process (patent pending).

S. molle (popularly known in Brazil as “aroeira-salsa”) was used for the experiments. Samples were collected from the Taboão Ecological Park, Lorena, São Paulo, Brazil ($22^\circ47'26''$ S $45^\circ66'11''$ W). The species grows rapidly and has a high ornamental value in the Zona da Mata mesoregion of Minas Gerais (Mafia *et al.*, 2004). It is naturally found in semi-deciduous seasonal forests in Paraná and in other ecosystems. The species is also available in nurseries throughout Vale do Paraíba.

We evaluated the effects of two factors, substrate and fertilization, on plant growth and nutrition. Three types of substrates were evaluated with and without the addition of fertilizer, in triplicate, totaling 18 experiments.

Soil or composts (obtained by aerobic composting) were used as substrates. Red-Yellow Acrisol with medium loamy texture (EMBRAPA, 2006) was collected at a depth of 0–20 cm. The soil is typically found in the study region. Composts were produced in the Taboão Ecological Park, located in Vale do Paraíba. Compost 1 was obtained from a nursery that grows seedlings of trees native to the Atlantic Forest. It was composed of several organic residues generated in the Park, such as pruning and mowing residues, cattle manure, and wastewater from washing tubes and livestock pens, as well as sand and worm humus. Compost 2 was composed of grasses and leaves and was obtained from a nursery in the Taboão Ecological Park. Fertilizer was used at 40 g kg^{-1} soil to provide the necessary amount of nitrogen for nursery growth (Van Raij *et al.*, 1997).

Substrates and fertilizer were sieved through a 2 mm sieve. One-month-old seedlings were planted in 0.25 L black plastic bags and kept at room temperature for 90 days. Plants received the same amount of water throughout the growing period. All nursery practices (establishment of the nursery site, seedling formation, and seedling handling) were in accordance with the guidelines of the Brazilian Agricultural Research Corporation (EMBRAPA, 2016).

After the growing period, substrates and aerial parts of the plants were characterized

chemically and physically following the methods of EMBRAPA (2009; 2011). Aerial parts were oven dried at 65°C and the dry weight was measured. Plant height was also determined. The density of growing media was determined or quantified following the methods of EMBRAPA (2009).

Available Ca, Mg, K, Na, and P were extracted with Mehlich 3 extraction, and exchangeable Al was extracted using a 1 mol L⁻¹ KCl solution. The elements were quantified by inductively coupled plasma optical emission spectrometry (ICP-OES). Chemical oxygen demand (COD) was determined to estimate the amount of C. Organic matter was oxidized by potassium dichromate in acidic medium at high temperature in the presence of a catalyst. Subsequently, absorbance was measured at 620 nm. N content was determined by digesting the sample in acidic medium, followed by distillation using borate/sodium hydroxide buffer to adjust the pH to 9.5. The concentration of ammonia N was quantified spectrophotometrically at 420 nm. Analyses were performed when the plants were collected and at the end of the evaluation period (90 days).

3. RESULTS AND DISCUSSION

Table 1 shows the physicochemical characteristics of the fertilizer. Its neutral pH (7.0), high content of organic carbon and base cations (Ca, Mg, and K), and undetectable Al content are interesting features for agricultural application.

Table 1. Physicochemical characteristics of the fertilizer.

Parameter	Unit	Value
pH	-	7.0 ± 0.0
Al	mg g ⁻¹	<0.001
Ca	mg g ⁻¹	405.85 ± 0.01
Mg	mg g ⁻¹	36.21 ± 0.01
P	mg g ⁻¹	35.96 ± 0.07
K	mg g ⁻¹	111.23 ± 0.01
Na	mg g ⁻¹	16.10 ± 0.01
N	mg g ⁻¹	52.35 ± 0.11
OC	mg dm ⁻³	3100 ± 0.01
Density	kg dm ⁻³	1.017 ± 0.010

OC, organic carbon. Values represent the mean ± standard deviation. Three replicates.

The physicochemical characterization of fertilized and unfertilized substrates is shown in Table 2.

Several beneficial effects of the addition of fertilizer to growing media were observed, such as an increase in pH, cation content, and base saturation and a decrease in exchangeable Al. A three-fold increase in organic carbon content was obtained with the addition of fertilizer as well as an increase in plant-available P and N. Regarding the substrates' physical characteristics, there was a small decrease in soil density, which is also a favorable effect. An increase in Na content was observed in fertilized soil as compared with unfertilized soil; however, the same was not observed for composts 1 and 2. This effect in soil nutrient content may be due to the chemical components used in the process to obtain the fertilizer.

Table 2. Physicochemical characteristics of fertilized and unfertilized substrates.

Parameter	Unit	Soil	Compost 1		Compost 2		
		Unfertilized	Fertilized	Unfertilized	Fertilized	Unfertilized	Fertilized
pH	-	5.7 ± 0.1	7.0 ± 0.0	5.6 ± 0.0	6.7 ± 0.0	5.4 ± 0.0	6.5 ± 0.0
Al	mg g ⁻¹	48.00 ± 0.00	46.50 ± 0.01	37.41 ± 0.02	35.21 ± 0.05	38.41 ± 0.01	31.74 ± 0.05
Ca	mg g ⁻¹	1.04 ± 0.00	84.12 ± 0.01	4.02 ± 0.01	66.12 ± 0.02	10.12 ± 0.01	424.2 ± 0.0
Mg	mg g ⁻¹	<0.001	36.20 ± 0.00	0.01 ± 0.02	3.65 ± 0.01	0.08 ± 0.00	40.1 ± 0.1
P	mg g ⁻¹	23.20 ± 0.01	28.73 ± 0.01	6.08 ± 0.04	29.73 ± 0.01	6.19 ± 0.01	36.9 ± 0.1
K	mg g ⁻¹	87.80 ± 0.01	96.05 ± 0.01	7.20 ± 0.00	97.20 ± 0.03	9.70 ± 0.02	88.01 ± 0.08
V%	-	25 ± 0	45 ± 0	22 ± 0	56 ± 0	20 ± 0.0	50 ± 0
Na	mg g ⁻¹	<0.001	0.013 ± 0.004	<0.001	<0.001	<0.001	1.301 ± 0.003
N	mg g ⁻¹	26.7 ± 0.0	61.4 ± 0.0	0.2 ± 0.0	31.4 ± 0.0	12 ± 0.0	61.4 ± 0.0
OC	mg mg ⁻³	36.20 ± 0.00	106.40 ± 0.01	45.20 ± 0.01	106.4 ± 0.0	32.00 ± 0.01	105.4 ± 0.0
D _s	kg dm ⁻³	1.217 ± 0.020	1.204 ± 0.060	1.227 ± 0.001	1.204 ± 0.010	1.257 ± 0.002	1.214 ± 0.003

There is a lack of research on the use of pharmaceutical wastewater as soil fertilizer, which hinders comparison of the results obtained in the present work. However, various studies have assessed the impacts of different wastewaters on soil. In a long-term field experiment, Al Omron *et al.* (2012) observed an increase in soil organic matter (from 17% to 30%) in sewage-irrigated soils as compared with soils irrigated with well water. The increase of 13 percentage points is considerably lower than that observed in the present study. Sewage irrigation reduced soil bulk density, as observed in the present study with the addition of wastewater-derived fertilizer.

Positive and negative reported effects of wastewater on soil pH have been reported in the literature. Al Omron *et al.* (2012) observed that soil pH decreased with sewage irrigation. Contrarily, other authors (El-Hady, 2007; Osaigbovo and Orhue, 2006; Rusan *et al.*, 2007) reported an increase in soil pH with wastewater irrigation, as was the case in the present study.

Domestic sewage sludge might increase soil fertility. Yadav *et al.* (2002) reported a build up of total N (2908 kg ha^{-1}), available P (58 kg ha^{-1}), and available K (305 kg ha^{-1}) in the soil. In the present study, soil N increased from 31.2 to 49.4 mg kg^{-1} after fertilization. Considering that the lowest soil bulk density was 2.4 kg dm^{-3} , we estimate that the increase in N content obtained in our study (from 75 to 118 kg ha^{-1} , approximately) was substantially higher than that of Yadav *et al.* (2002). P content increased from 13 to 73 kg ha^{-1} , reaching values higher than that of the mentioned study, whereas K content increased from 9 to 216 kg ha^{-1} because the fertilizer was relatively poor in this nutrient.

We emphasize that this type of comparison should be performed with extreme caution. The wastewaters evaluated in the above-mentioned studies are different from the wastewater used in of our study, as are the rates and periods of application, soil type, and plants. The comparisons indicate that the changes in soil parameters observed in this work are of the same order of magnitude as those reported in the literature.

Sodification (Na accumulation) might occur with as a result of frequent wastewater irrigation (FAO, 2007). In this case, Na might displace other cations, such as Ca and Mg. High sodium levels might affect soil structure by reducing soil permeability (Sumner, 1993). In the present study, soil Na content did not increase substantially with fertilizer application. However, seedlings were cultivated for a short period. Long-term field application is necessary to evaluate better the effects of Na on soil and plant characteristics.

Figure 2 shows photographs of plants at 30, 60, and 90 days of cultivation in fertilized and unfertilized Acrisol. Photographs of plants grown in compost 1 and 2, fertilized and unfertilized, are shown in Figure 3. Fertilized plants had a higher growth rate than unfertilized plants; the highest growth rate was that of plants grown in compost 1.

Figure 2. Development of plants grown in Acrisol at 30, 60, and 90 days of cultivation (from left to right). (A). Unfertilized soil and (B). fertilized soil.

Figure 3. Development of plants grown in fertilized (right) and unfertilized (left) composts at 90 days of cultivation. (A) Plants grown in compost 1 and (B) plants grown in compost 2.

Table 3 shows the characteristics of plants cultivated in fertilized and unfertilized growing media.

Table 3. Characteristics of aerial parts of plants grown for 90 days in fertilized and unfertilized substrates.

Parameter	Unit	Soil		Compost 1		Compost 2	
		Unfertilized	Fertilized	Unfertilized	Fertilized	Unfertilized	Fertilized
Al	mg g ⁻¹	0.018 ± 0.006	0.030 ± 0.011	0.004 ± 0.001	< 0.001	0.300 ± 0.021	0.033 ± 0.009
Ca	mg g ⁻¹	0.08 ± 0.01	160.5 ± 0.0	8.50 ± 0.01	85.5 ± 0.0	0.10 ± 0.03	81.0 ± 0.1
Mg	mg g ⁻¹	0.36 ± 0.02	5.30 ± 0.01	0.79 ± 0.01	5.42 ± 0.02	0.50 ± 0.05	8.10 ± 0.02
P	mg g ⁻¹	0.89 ± 0.01	31.1 ± 0.0	0.51 ± 0.02	102.3 ± 0.0	0.92 ± 0.01	89.0 ± 0.0
K	mg g ⁻¹	17.02 ± 0.01	36.60 ± 0.03	30.21 ± 0.03	39.21 ± 0.02	17.30 ± 0.01	37.0 ± 0.0
Na	mg dm ⁻³	<0.001	3.00 ± 0.02	0.550 ± 0.012	7.60 ± 0.01	0.110 ± 0.009	7.10 ± 0.02
N	mg g ⁻¹	12.0 ± 0.0	55.4 ± 0.0	20.1 ± 0.1	56.0 ± 0.0	11.3 ± 0.0	50.0 ± 0.0
OC	mg dm ⁻³	201.2 ± 0.0	638.4 ± 0.0	202.4 ± 0.0	640.3 ± 0.0	203.0 ± 0.0	210.0 ± 0.0
Height	mm	97.1 ± 0.0	151.6 ± 0.0	80.2 ± 0.0	160.2 ± 0.0	80.1 ± 0.0	120.0 ± 0.0
Dry weight	g	4.2 ± 0.00	6.2 ± 0.0	6.0 ± 0.0	10.0 ± 0.0	5.0 ± 0.0	7.0 ± 0.0

OC, organic carbon. Values represent the mean ± standard deviation. Three replicates.

Fertilized plants were taller and had a greater absorption of C, N, and P than unfertilized plants. Plants grown in compost 1 had a lower Ca content than plants grown in compost 2, probably because compost 1 had more nutrients.

The literature presents evidence of the environmental contamination potential of pharmaceuticals. Carter *et al.* (2014) studied the fate (in-soil degradation, transport, and plant uptake) of analytical grade carbamazepine, diclofenac, propranolol, and sulfamethazine in Tepko soil in Australia. Wu *et al.* (2015) studied the fate of pharmaceuticals and personal care products added to soil irrigated with treated wastewater or biosolid-amended soil. Carter *et al.* (2018) studied the sorption, plant uptake, and metabolism of benzodiazepines, a widely used class of pharmaceuticals that are recalcitrant to wastewater treatment. According to the authors, the mechanisms involved in pharmaceutical uptake and transport in plants are very complex.

Few studies focus on the effects of wastewater on plant nutrient uptake. Cruz *et al.* (2016) reported that *Tabebuia* spp. grew at a faster rate in Acrisol fertilized with C, N, P, and Ca than in unfertilized soil. However, because of the complexity of plant uptake and transport mechanisms, it is very difficult to compare the effects of different wastewaters on plant nutrient absorption.

4. CONCLUSION

Effluent-derived fertilizer improved the characteristics of all growing media, increasing pH and C, N, P, K, Mg, and Ca contents while decreasing Al content and soil density. However, fertilization increased Na content. Further research is needed to assess the effects of fertilization on soil Na levels.

Plant growth (measured by dry weight and height) and nutrient uptake were enhanced by fertilization. The best results were obtained using compost 1, as it contained the highest concentration of nutrients.

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Changes in physicochemical and toxicological parameters of waters of Trincadeira's River caused by road construction

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ABSTRACT

Road infrastructures can greatly affect natural areas. A trench and a roundabout were constructed in the city of Francisco Beltrão-Paraná-Brazil over a water body called Trincadeira's River. The present study evaluated the quality of the waters of Trincadeira's River, in physicochemical and toxicological terms, at four points along its course, during and after the trench and the roundabout construction. The results of the physicochemical analysis showed changes in some parameters analyzed near the road infrastructure, in particular, oils and greases, biochemical oxygen demand and dissolved oxygen. The immobility/mortality test with *Artemia salina* L. micro shellfish showed that none of the water at the collection points of the river was toxic. The cytotoxicity test with the bioindicator *Allium cepa* L. revealed no cytotoxic effect in the sample collection done during the road construction. However, after the finalization of the trench and the roundabout construction, a statistically significant increase in the percentage of the mitotic index at the road construction point was noticed. Therefore, awareness and regulatory measures are necessary to oversee activities on water bodies' shores and environs, to ensure a harmonious relationship between human activity and the ecosystem.

Keywords: *Allium cepa* L., *Artemia salina* L., bioindicators, water pollution, water quality.

Alterações dos parâmetros físico-químicos e toxicológicos das águas do Rio da Trincadeira afetadas por uma construção rodoviária

RESUMO

Infraestruturas rodoviárias podem afetar áreas naturais com grande intensidade. Um trevo e uma trincheira foram construídas na cidade de Francisco Beltrão-Paraná-Brasil sobre um

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corpo d'água chamado Rio da Trincheira. O objetivo do presente estudo foi avaliar a qualidade de suas águas, em termos físico-químicos e toxicológicos, em quatro pontos ao longo de seu curso, durante e após a construção da trincheira e do trevo. Os resultados das análises físico-químicas mostraram alterações em alguns parâmetros analisados, em especial, óleos e graxas, demanda bioquímica de oxigênio e oxigênio dissolvido, próximos à infraestrutura rodoviária. O teste de imobilidade/mortalidade com os microcrustáceos *Artemia salina* L. mostrou que nenhum dos pontos de coleta de água do rio foi tóxico. O teste de citotoxicidade com o bioindicador *Allium cepa* L. revelou que não houve efeito citotóxico na coleta da amostra durante a construção rodoviária. No entanto, após a finalização da construção da trincheira e do trevo, verificou-se um aumento estatisticamente significativo da percentagem do índice mitótico no ponto/local de construção da estrada. Portanto, medidas de conscientização e regulação devem ser necessárias para interferir nas atividades realizadas nas margens e nos corpos d'água, buscando uma relação harmoniosa entre as atividades humanas e todo o ecossistema.

Palavras-chave: *Allium cepa* L., *Artemia salina* L., bioindicadores, poluição da água, qualidade da água.

1. INTRODUCTION

Disorganized demographic growth, combined with a lack of urban planning and the irregular handling of agricultural products, can promote the degradation of water bodies. Buildings in general, but especially road infrastructures, affect natural areas with greater intensity than other processes of development, since they can lead to accidental spillages of chemical waste in rivers (Moraes and Santos, 2004), contaminating waters.

Solid or liquid waste from urban development, as well as from construction and demolition, has been the focus of studies in the past few years due to the environmental damage that they can cause. With regard to environmental damage, the lack of supervision of urban civil works and roads tends to worsen the impacts, compromising the quality not only of the soil and air but also of water bodies near urban facilities.

The pollution of a headwater mainly affects the physicochemical characteristics of the water, then systematically destroys the aquatic community, compromising the trophic chain and public health. However, the exclusive use of physical or physicochemical parameters to assess water quality (Von Sperling, 2005) has attracted criticism, in that a more ecosystemic approach is needed, requiring the application of biological assessment studies (Kuhlmann *et al.*, 2001).

Bioindicators respond efficiently to the presence or absence of a contaminant, providing data related to the conditions of a specific environment (Frontalini and Coccioni, 2011). In Brazil, aquatic ecotoxicology has been used, along with chemical and physicochemical parameters, as a water quality parameter, constituting an important monitoring instrument of water resources (Tavares, 2014). In this sense, while chemical analyses identify and quantify concentration of toxic substances, toxicity tests assess the effect of these substances on biological systems.

Luíz *et al.* (2012) recommends that the toxic effect of a sample be evaluated by more than one representative species of aquatic biota, preferably belonging to different trophic levels of the food chain. In this scenario, the immobility/mortality test with *Artemia salina* L. micro shellfish and cytotoxicity test with the vegetable *Allium cepa* L. stand out. The test with *A. salina* is a method that has a good correlation with acute oral toxicity tests *in vivo* (Silva *et al.*, 2010). Similarly, the test with *A. cepa* could be sensitive, and in addition has a positive correlation in prokaryotes and eukaryotes (Fatma *et al.*, 2018), being indicative of risk to human health or the environment.

Hence, the present study evaluated, through physicochemical analysis and ecotoxicological tests with *A. salina* and *A. cepa*, the influence of urban construction activities on the water quality of Trincadeira's River in the municipality of Francisco Beltrão - Paraná, Brazil.

2. MATERIAL AND METHODS

2.1. Study Area

The municipality of Francisco Beltrão, Latitude 26°04'52 "S and Longitude 53°03'18" W, located in the southwest region of the State of Paraná - Brazil (Figure 1), is undergoing urban expansion (Francisco Beltrão, 2012).

Figure 1. Location of the municipality of Francisco Beltrão-Paraná, Brazil. The river basin area contributing to the river headwater contribution of Trincadeira's River (area under study) and its water-sample collection points: River headwater (P1); Residential condominium (P2); Place where the river was channeled for road construction (P3); River mouth (P4).

The region of the study (the watershed basin of the Trincadeira's River) is characterized by agricultural, urban and industrial activities, with an emphasis on beef and dairy cattle, construction of residential condominiums and mining. This headwater has a significant importance to the region, since it supplies the small farms for livestock watering, aquaculture, and even human consumption. In order to facilitate access to the county, a trench and roundabout were built, at the same place, in the West region of the municipality. This required rock demolition, as well as earth work and the insertion of a drain pipeline, to conduct the water of the Trincadeira's River, the course of which was changed close to its mouth at Marrecas's river.

The daily flow of trucks (cargo/transport), buses (urban, intermunicipal and state), agricultural machinery and other automobiles is intense. The roundabout directs the flow of vehicles to an industrial district, the federal university, the agricultural school and the other neighborhoods around the city.

2.2. Sampling

The water samples were collected during October 2014 and after April 2015, the trench and the roundabout construction. In October 2014, the total monthly rainfall was approximately 125 millimeters, and in April 2015 it was 45 millimeters in the city of Francisco Beltrão and the surrounding region (IAPAR, 2016).

Four sampling points were selected along Trincadeira's River (Figure 1): Point 01 - Headwater of the river (6°03'46.4" S; 53°05'50.3" W); Point 02 - Next to a residential condominium (26°04'23.9" S; 53°04'56.2" W); Point 3 - The trench and the roundabout construction (26°04'27.5" S; 53°04'37.9" W); and Point 04 - The river mouth (26°04'29.3" S; 53°04'37.2" W).

The collection and preservation/storage of water samples were carried out according to instructions and methods contained in "Standard Methods" (APHA *et al.*, 2012). The samples were collected with the aid of a plastic bucket, in places of difficult access to the middle of the river water stream, and stored in clean plastic bottles. Where possible, the plastic bottles were immersed at the point of collection in the river, about 30 cm deep. The parameters of pH, Temperature (T), chemical oxygen demand (COD), biochemical oxygen demand (BOD) and dissolved oxygen (DO) were analyzed in the laboratory immediately after collection. For the analysis of ammonia nitrogen, total phosphorus, oils and greases and toxicity, the bottles with samples were stored in a refrigerator at 10°C for up to 1 week or frozen ($T \leq 4^{\circ}\text{C}$) for subsequent analysis. Afterwards, the bottles were thawed until they reached room temperature.

2.3. Physicochemical parameters

The analysis of physicochemical parameters of the water samples of the Trincadeira's River followed the methodologies established by the "Standard Methods" (APHA *et al.*, 2012). The following parameters were evaluated in triplicate: pH, chemical oxygen demand, biochemical oxygen demand, dissolved oxygen, ammoniacal nitrogen (ammonia-N), total phosphorus, oils and greases and temperature.

2.4. Immobility/Mortality tests

The toxicity test was performed with *A. salina* L. by the method proposed by Guerra (2001), with modifications based on ABNT NBR 13373 (2010). Ten nauplii of *A. salina*, in instar stages 2-3, were transferred to test tubes containing 2 mL of samples from the different points of Trincadeira's River, and diluted in a saline solution (30g of sea salt per L distilled water) in the following concentrations: 100%, 50%, 25%, 12.5%, 6.2% and 3.1%, in order to determine the critical range, based on the average number of dead nauplii per concentration of the river's water, based on ABNT NBR 13373 (2010). The negative control group contained only 2 mL

of saline solution (CO^- 1) or 2mL of mineral water (CO^- 2), to confirm the effect of river water and not saline or sweet. Positive control (CO^+) was performed with 0.016 μL of methyl methanesulfonate per mL of mineral water. After 24 hours of incubation (tubes at $25 \pm 1^\circ\text{C}$), the number of dead/immobility nauplii was checked for each replicate to calculate the mean number of dead/immobile nauplii, thus determining the toxicity.

2.5. Cytotoxicity and mutagenicity tests

Cytotoxicity and mutagenicity tests were assessed using the meristematic cells of the root of *A. cepa* L. (onion), prepared by the Feulgen reaction and stained with the Schiff reagent (Fiskesjö, 1985).

First, the onion bulbs were cleaned and placed to generate roots in containers containing filtered, aerated water, in a dark room, with temperature control of $25 \pm 2^\circ\text{C}$. Five onions were used for each treatment sample group. Before each treatment, two roots of each onion were collected, to serve as the control of the bulb (Co-0h). Thereafter, the roots were exposed to treatments with water samples from Trincheira's River and 0.04 mg of cyclophosphamide/mL filtered water (positive control – CO^+), for 24 hours (Tr-24h), according to the proposal proposed by Fiskesjö (1985). The bulbs were then placed again in filtered water, to recover themselves from any damage for 24 hours (Re-24 h). The negative control (CO^-) remained all the time in the filtered water.

The roots collected during the three days of collection (Co-0h, Tr-24h, Re-24 h) of all treatments (Points 1, 2, 3, 4, CO^+ and CO^-) were fixed, hydrolyzed with hydrochloric acid (HCl 1N at 60°C for 10 minutes) and colored with Schiff reagent (45 minutes in the dark). Then, the meristematic region of onion roots was collected, stained with acetic orcein and crushed to produce the blades.

The blades were analyzed using the blind test in light microscopes (40 x). One thousand cells were counted from each onion, differentiating them according to the mitotic phases (Interphase, Prophase, Metaphase, Anaphase, Telophase), to determine the percentage of Mitotic index (MI%) from each sampling time of the evaluated groups, using the following formula: $\text{MI}\% = (\text{number of dividing cells} / \text{total number of cells analyzed}) \times 100$. The cells were also analyzed observing structural changes, like colchicine metaphase or disorganized metaphase, disorganized anaphase or with chromosomes loss, micronucleus and others.

2.6. Statistical analysis

The average of physicochemical analyses, the average of immobility (Immobility test) and the average of MIs (Cytotoxicity test) were performed by the Tukey test ($\alpha = 0.05$; $n = 3$, $n = 4$ and $n = 5$, respectively).

3. RESULTS AND DISCUSSION

The pH values (Table 1) found in the river's water samples, although with a statistical difference ($p < 0.05$) between points and also in the periods during and after the trench and roundabout construction, are in accordance with the limit (6.0-9.0) established by CONAMA legislation N° 357 (2005) for the class of Trincheira's River (Class II of fresh water). According to Von Sperling (2005), aquatic organisms are usually adapted to conditions of neutrality ($\text{pH} \approx 7$), and sudden changes of the water's pH may lead to the extinction of aquatic biota. In the present study, pH ranged from 6.5 to 7.2 (slight acid to neutral), not being considered an abrupt variation (Avvannavar and Shrihari, 2008) or an endangering value to the aquatic ecosystem (Yalçuk and Postalcioglu, 2015). According to Gupta *et al.* (2017), the variations of pH can be attributed to the different seasons and climates of the year during the samplings, resulting in a variation in the concentration of CO_2 incorporated in river waters. Similar pH values were obtained by Gan *et al.* (2008), to evaluate the pH of superficial water runoff of some sampling

points on highways in the Pearl River Basin (Guangzhou-Southern China).

The concentration of total phosphorus (Table 1) also presented significant difference ($p < 0.05$) when compared with the collecting points, during and after the ending of construction, with values that varied from 0.013 (P1) to 0.033 mg L⁻¹ (P2), being all values below what is recommended in the legislation (0.1 mg L⁻¹) (CONAMA, 2005). The highest value for the concentration of phosphorus at Point 2 can be directly related to the pH, because, according to Silva *et al.* (2003), lower values of pH favor the dissolution of substances, increasing their concentrations in the aqueous medium. Other factors that may explain the low phosphorus concentration along the river are the water's flow at the points of collection (lotic stretch), low incidence of rural residences and the absence of agricultural fields. According to Kibena *et al.* (2014) and Rahman *et al.* (2011), the increase in the concentrations of nutrients (NO₃, NH₃, and PO₄) in surface water bodies tends to be related to the presence of fertilizers, synthetic substances, domestic effluents and the decrease of the water's flow (lentic stretches).

Table 1. Average and standard deviation of physicochemical parameters analyzed in triplicate for the different points of the Trincheira's River, during and after road infrastructure construction.

Collecting Points	Time of Collection	Physicochemical Parameters			
		pH	Ammonia-N (mg L ⁻¹)	Total Phosphorus (mg L ⁻¹)	Temperature (°C)
P1	During	6.64±0.01 ^{aA}	VA	0.016±0.001 ^{aA}	26.71±0.01 ^{aA}
	After	7.06±0.09 ^{aB}	VA	0.013±0.001 ^{aB}	23.13±0.05 ^{aB}
P2	During	6.92±0.02 ^{bA}	VA	0.023±0.001 ^{bA}	26.57±0.05 ^{abA}
	After	6.57±0.05 ^{bB}	VA	0.033±0.001 ^{bB*}	23.61±0.17 ^{bB}
P3	During	6.87±0.02 ^{bA}	VA	0.020±0.001 ^{cA}	26.33±0.05 ^{bA}
	After	7.07±0.01 ^{aB}	VA	0.023±0.001 ^{cB}	23.43±0.11 ^{bB}
P4	During	7.05±0.03 ^{cA}	VA	0.020±0.001 ^{cA}	26.51±0.00 ^{abA}
	After	7.19±0.01 ^{cB}	VA	0.026±0.001 ^{dB}	23.33±0.15 ^{abB}
Conama N° 357/2005		6.00-9.01	≤ 3.7	≤ 0.1	-

P1: River headwater; P2: Residential condominium; P3: The trench and the roundabout construction; P4: River mouth. VA: Virtually Absent.

Values with the same letter do not differ statistically among themselves; lowercase is used for the comparison among the four sampling points at each collection time, and uppercase for the comparison between the times of each collection point by Tukey test with a significance level of 5%, n=3.

BOD is an indicator parameter related to the amount of oxygen required in an aquatic environment for a microorganism's respiration over the consumption of organic substances, which are directly associated with the presence of animal and food waste or residential/industrial discharge of untreated sewage introduced into the aquatic environment (Oliveira *et al.*, 2012). In this study, the values ranged from 12.78 to 68.33 mg L⁻¹; O₂ differed statistically ($p < 0.05$) (Figure 2) between the sample points. During the trench and roundabout construction, it could be seen that the values of the BOD, the DO and oils and greases (Figure 2) at any collection point were in disagreement with the values established by legislation (BOD ≤ 5 mg L⁻¹ O₂; DO ≥ 5 mg L⁻¹ O₂; oils and greases = virtually absent) (CONAMA, 2005). A similar phenomenon was observed by Sekabira *et al.* (2010), which showed values higher than the limit of local legislation (BOD ≤ 50 mg L⁻¹ - National Environment Management Authority - NEMA), mainly due to the discharge of industrial effluents in the Nakivubo stream (Kampala - Uganda).

Figure 2. Average and standard deviation of physicochemical parameters analyzed for the different points of the Trincadeira's River, during and after the construction of the road infrastructure.

P1: River headwater; P2: Residential condominium; P3: The construction site of the trench and roundabout; P4: River mouth. COD: chemical oxygen demand; BOD: biochemical oxygen demand; DO: dissolved oxygen. * Data different from those established by law N° 357 of 2005 from CONAMA for Class II. Values with the same letter do not differ statistically among themselves; lowercase is used for comparison among the four sampling points in each collection time, and uppercase for the comparison between the times of each collection point by Tukey test with a significance level of 5%.

The lowest levels of DO ($3.4 - 3.87 \text{ mg L}^{-1}$) related to the presence of organic matter or similar synthetic compounds in the river waters. A similar study carried out in the Dourados and Brilhante Rivers by Sposito *et al.* (2019) related the low OD values found ($3.66 - 4.86 \text{ mg L}^{-1} \text{ O}_2$) to the intense urbanization process (domestic effluent disposal) and agriculture (pesticides and agrochemicals). Arpine and Gayane (2016), studying the water of river Akhuryan (Armenia), observed higher DO values ($10.3 \text{ mg L}^{-1} \text{ O}_2$) and lower BOD ($2.8 \text{ mg L}^{-1} \text{ O}_2$) and COD ($10.5 \text{ mg L}^{-1} \text{ O}_2$) values than those of the present study, aforementioned. However, the Akhuryan waters possess a great potential for self-depuration, because its riverbed features a shapeless rocky profile when, adversely, the Trincadeira's River waters has a reduced and uniform flow rate. In the present study, it was possible to verify a significant increase of the DO concentration after the roundabout construction (Figure 2), setting the values according those recommended by CONAMA (2005). This increase in the DO concentration is possibly due to a reduction in the release of organic substances, such as oils and greases, after the work ended.

According to CETESB (2009), the decomposition process of oils and greases, could reduce dissolved oxygen and the contact surface between the water surface and the atmospheric air. In addition, the increase of BOD and COD concentration could lead to aesthetic and organoleptic problems on the course of the river as well as scum formation, eutrophication and bad smell.

According to Brazilian legislation (CONAMA, 2005), the presence of oils and greases in hydrous bodies should be null. In the present study, these compound were found at all sampled points, with contents ranging from 3.24 to 6.40 mg L^{-1} with significant difference ($p < 0.05$) between the sampling points P1, P3 and P4 (Figure 2). This presence could imply a source of oil or synthetic derivative contamination, indicating pollution from urban waste, sewage

disposal and accidental spills. Katukiza *et al.* (2015) have shown that domestic effluent water charges in the Bwaise III (Kampala - Uganda) showed concentrations of oils and greases above that established by local legislation (10 mg L^{-1}), as well as the situation found in the present study. Highway vehicles release particulates and synthetic lubricants, which are leached by surface runoff during precipitation events until they reach the river. Van Bohemen and Janssen Van de Laak (2003) found similar values for the concentration of oils and derivatives ($3.0 - 8.0 \text{ mg L}^{-1}$) in runoff waters on national, provincial and municipal roads in Holanda. Moreover, during the roundabout construction, there was a high incidence of large machinery movement, such as excavators, bulldozers and dump trucks, which could have accidentally spilled oil, grease, and tar.

Physicochemical data corroborate with those found in the immobility/mortality test with the bioindicator *A. salina* (Figure 3). The waters of Trincheira's River had a high mortality rate at the point of the trench and the roundabout construction after the end of the construction (P3) (17% mortality) (Figure 3). Maddela *et al.* (2016), studying the removal of petroleum hydrocarbons, showed that the higher the concentration of these petrochemical derivatives, the greater was the mortality ($\approx 33,5\%$ - exposure for 24h) of the *A. salina* shellfish.

The statistical data from the immobility/mortality test did not show significant toxicological effect of the water samples, during or after the construction of road infrastructure, because the mortality of organisms exposed to water samples of all four points of the river was statistically similar to negative control mortality.

Figure 3. Average numbers of dead organisms obtained during and after the road infrastructure construction for different concentrations of water samples from Trincheira's River. CO⁻ 1: Negative control with saline solution. CO⁻ 2: Negative control with mineral water. CO⁺: Positive control. P1: River headwater; P2: Residential condominium; P3: The construction site of the trench and roundabout; P4: The river mouth.

Percentages with the same letters do not differ statistically among themselves; uppercase is used for comparison within the same collection time, and lowercase for comparison between the two times of collection, by Tukey test with a significance level of 5%.

Additionally, changes in toxicity were not observed during or after the road construction for any evaluated point, because the average number of dead/still organisms was statistically similar. Similar results were found by Vaz *et al.* (2010), who studied the toxicity of Babitonga Bay (Santa Catarina, Brazil), also with the *A. salina* bioindicator. The data showed that the vast majority of the evaluated points was not toxic to the micro shellfish.

Components in gasoline and diesel fuel, such as benzene and benzo (a) pyrene, are carcinogenic compounds, while the toluene, ethylbenzene, and xylene are classified as toxic. However, when some of these derivatives are poured directly into the water, they have a rapid volatilization, with little tendency to adsorb sediments or to hydrolyze (Finotti *et al.* 2001), which possibly has resulted in lower acute toxicity of water samples from the Trincheira's River, contaminated by oils and greases.

No cytotoxicity was observed by *A. cepa* bioindicator during the trench and roundabout construction. Data in Figure 4 show that there was no statistical difference between the mitotic index of the negative control at the time of sampling (Co-0h, Tr-24h, and Re-24 h) and the groups treated with water samples collected from all points. When sampling times within the same group were compared, control or treaty, no statistical difference was found.

Figure 4. Percentage of Mitotic Indexes (MI) obtained during and after the construction of road infrastructure for water samples from Trincadeira's River. CO⁻: Negative control. CO⁺: Positive control. P1: River headwater; P2: Residential condominium; P3: The trench and roundabout construction; P4: The river mouth. Sampling time: 0 time = Co-0h, Treatment 24 hours = Tr-24 h, Recovery 24 hours = Re-24 h. Percentages with the same three letters do not differ statistically among themselves; uppercase is used for the comparison between three sampling times within the same group or control, lowercase for comparison between each sampling time of the negative control with the same time of each group treated, by Tukey test with a significance level of 5%.

The absence of *A. cepa* cytotoxicity, was also found in waters contaminated with petrochemical derivatives (leaked from a pipeline) (Leme and Marin-Morales, 2008) and effluents from refineries (Gupta and Ahmad, 2012). Similar results, using the same test system as the present study, were found on the surface waters of the Sava River (Croatia) (Radic *et al.*, 2010), on the waters from Ribeirões Varginha River (Califórnia-Paraná-Brazil) and Tabatinga

River (Mandaguari-Paraná-Brasil) (Ferreira *et al.*, 2012), and on the waters from the Peixe River collected in the cities of Garça, Tupã, Flórida Paulista and Ouro Verde (São Paulo-Brazil) (Costa *et al.*, 2015), which also received agricultural, industrial and urban effluents along their courses. Morphological changes and chromosomal alterations were also not observed when exposed to the waters of Trincadeira's River, similar to work by Ferreira *et al.* (2012).

After the road construction, the data of cytotoxicity (Figure 4) show that the groups treated with the waters from Trincadeira's River showed percentages of mitotic indexes progressively larger, from 1 to 4 (P1 = 1.84%, P2 = 2.36%, P3 = 4.00%, P4 = 3.28%). However, only Point 3 (site of the trench and roundabout construction) (24 hours) showed increases in the percentage of mitotic index statistically different from the negative control (24 hours = 1.58%) and P1. The values of physicochemical analysis were supported by those data. Points 3 and 4 presented the highest values of COD and mitotic indexes and smaller levels of DO when compared to other collection points.

The COD of Points 3 and 4 (site of the road construction and the river mouth) presented elevated levels from the first to the second collection (Figure 2). The change from wet weather to the dry weather could cause an increase in the level of contaminants, possibly, due to the decrease in the flow of the river (Kibena *et al.*, 2014). After the roundabout construction, the water sampling was performed during a drier season (Autumn), resulting in a lower flow rate when compared to the headwater (first collection) at the moment the Trincadeira's River was being piped in.

Similar to the studied data, the evaluation of Quatorze River, located in the rural municipality of Francisco Beltrão, showed that three points of this river were not cytotoxic to the *A. cepa* bioindicator. One point presented statistical increase of cell proliferation and one point a greater organic load in the physicochemical analyses (Düsmen *et al.*, 2014). Gomes *et al.* (2015) also used the *A. cepa* to study the cytotoxicity of the Guandu River (Rio de Janeiro-Brazil), contaminated by urban and industrial effluents, and there were increased percentages of mitotic indexes of the onions exposed to these waters.

4. CONCLUSIONS

The data from this study indicate changes in some physicochemical parameters analyzed; in particular, oils and greases, biochemical oxygen demand and dissolved oxygen, close to the road infrastructure. Toxicology tests showed an increase in the number of dead *A. salina* and in the percentage of mitotic index of the *A. cepa* roots, from the headwater to the river mouth. It was most evident at the collection point of the trench and the roundabout construction for both bioindicators.

Therefore, awareness and regulatory measures are necessary to oversee activities performed on the water bodies' shores and environs, to ensure a harmonious relationship between human activities and the ecosystem.

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Autochthonous microalgae cultivation with anaerobic effluent: isolation of strains, survivorship, and characterization of the produced biomass

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ABSTRACT

Six Chlorophyta strains were isolated from the effluent of an anaerobic reactor treating municipal wastewater and identified as *Desmodesmus* sp. L02, *Chlorococcum* sp. L04, *Coccomyxa* sp. L05, *Chlorella* sp. L06, *Scenedesmus* sp. L08 and *Tetradismus* sp. L09. The microalgae strains were cultivated in unsterilized wastewater under laboratory conditions to determine their potential to survive under non-sterile conditions. The strains were also cultivated in sterilized wastewater in order to analyze their nutrient removal potential and characterize the produced biomass. Amongst the isolated microalgae, *Chlorella* sp. L06 had the highest survivorship percentage (90%) for ten days of culture, whilst *Desmodesmus* sp. L02 had the lowest, not exceeding 1.8% after 24h of inoculation. The dried biomass of the isolates showed an average of 28.7% of protein, 15.4% of lipids and 14.8% of carbohydrates, with *Chlorococcum* sp. L04 reaching 29.3% of carbohydrates. In terms of nutrients, nitrogen removal varied from 59.2 to 93%, and phosphorus removal ranged from 79.1 to 95.4%, with *Tetradismus* sp. L09 being the most efficient strain.

Keywords: anaerobic effluent, microalgae, survivorship.

Cultivo de microalgas nativas com efluente anaeróbico: isolamento de cepas, sobrevivência e caracterização da biomassa algácea

RESUMO

Seis cepas de Chlorophyta foram isoladas do efluente de um reator anaeróbico tratando esgoto sanitário e identificadas como *Desmodesmus* sp. L02, *Chlorococcum* sp. L04, *Coccomyxa* sp. L05, *Chlorella* sp. L06, *Scenedesmus* sp. L08 e *Tetradismus* sp. L09. As cepas de microalgas foram cultivadas em efluente não-esterilizado em condições de laboratório para determinar os potenciais de sobrevivência sob condições não estéreis. As cepas também foram cultivadas em efluente esterilizado para avaliar os potenciais de remoção de nutrientes e caracterizar bioquimicamente a biomassa produzida. Entre as microalgas isoladas, *Chlorella*

sp. L06 teve a maior porcentagem de sobrevivência (90%) por dez dias de cultura, enquanto *Desmodesmus* sp. L02 apresentou o menor valor, não excedendo 1,8% após 24h de inoculação. A biomassa seca dos isolados apresentou uma média de 28,7% de proteína, 15,4% de lipídios e 14,8% de carboidratos, com *Chlorococcum* sp. L04 atingindo 29,3% de carboidratos. Em termos de nutrientes, a remoção de nitrogênio variou de 59,2 a 93%, e a remoção de fósforo variou de 79,1 a 95,4%, com *Tetradasmus* sp. L09 sendo a cepa mais eficiente.

Palavras-chave: efluente anaeróbio, microalgas, sobrevivência.

1. INTRODUCTION

Since their inception, wastewater treatment plants (WWTPs) have had a fundamental role in minimizing the impact on water environments on a global level. Until recent years, WWTPs have been implemented with the single objective of protecting the health of humans and the environment. Nevertheless, a paradigm change is occurring in the universe of wastewater treatment, changing the emphasis on the treatment to the focus on recovering the various resources within the wastewater, such as water and nutrients (Puyol *et al.*, 2017). Microalgae-based technologies are perfect candidates to fulfil the aforementioned objectives, considering that these microorganisms grow rapidly, can be cultivated on non-arable lands using wastewaters, promote CO₂ sequestration and can be used to polish several types of effluents.

However, the cultivation of microalgae in wastewater effluents is not a new subject, considering that Oswald and Gotaas (1957) first proposed it in the 1950s, and in the decades since, high-rate algal ponds (HRAPs) have been widely studied – only with the purpose of treating wastewater. The algal biomass produced in these systems has not been traditionally recovered to use as feedstock for bioenergy production, specifically due unsatisfactory productivities and the fact that there are no low-cost and effective harvesting technologies.

Conventionally, microalgae biomass production systems use chemical fertilizers and clean water from the distribution grid, and consequently the high costs for the cultivation are one of the main bottlenecks of microalgae-to-biofuel systems in real scale (Brasil *et al.*, 2017). For this reason, most of the scientific community assumes that to reduce the environmental impacts and improve the economic viability of microalgae-to-biofuel processes, alternative sources of water and nutrients must be used. In fact, numerous authors have already reached high biomass productivities when cultivating microalgae in wastewaters, as evidenced by a recent review on the subject (Lv *et al.*, 2017).

Cho *et al.* (2017) discuss that microalgae that are indigenous to the type of wastewater used as medium could display higher adaptability to grow and remove nutrients from it. However, most studies focus on strains from Microalgae Culture Collections, ignoring the microalgal diversity that is already present in the wastewater. Therefore, this study had the primary objective of isolating microalgae strains from the effluent of a UASB reactor treating municipal sewage, in order to cultivate them in non-sterile wastewater and analyze their survivorship potential. The strains were also cultivated in sterilized wastewater in order to determine their growth, nutrient removal potential and to analyze the biochemical properties of the produced biomass.

2. MATERIALS AND METHODS

2.1. Microalgae isolation and identification

To isolate each strain, borosilicate flasks were filled with the unfiltered effluent from an upflow anaerobic sludge blanket reactor (UASB) treating municipal wastewater, located at an experimental WWTP in the Federal University of Espírito Santo (UFES), closed with cotton

leads and exposed to indirect sunlight for 15 days to stimulate the growth of autochthonous microalgae and other microorganisms. After this period, the raw effluent turned green due to the multiplication of naturally present microalgae cells, which were favored by the environmental change from complete darkness (inside the reactor) to indirect sunlight. Samples from these mixed cultures were used for the isolation procedures, namely the micropipette method followed by the streak plating technique until unialgal colonies were obtained, according to Andersen and Kawachi (2005). The streak plates were incubated with the following conditions: temperature of $27 \pm 3^\circ\text{C}$; luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$; and 12 h/12 h (light/dark) photoperiod. Isolated colonies were kept in liquid sterile effluent media and on agar effluent plates and incubated with the same conditions as the streak plates. Further information on the isolation, identification and maintenance procedures applied herein can be found in Pereira *et al.* (2018).

2.2. Effluent sampling and analysis

The effluent was sampled, filtered with glass fiber membrane filters (GF/C; $1.2 \mu\text{m}$ pore size) and analyzed for pH, volatile solids, chemical oxygen demand (COD), total Kjeldahl nitrogen (TKN) and total phosphorus (TP), according to the Standard Methods for the Examination of Water and Wastewater (APHA *et al.*, 2005).

The main characteristics of the unsterilized filtered effluents (UFE) throughout the experiments ($n = 3$) were: $28.8 \pm 1.5 \text{ mg L}^{-1}$ for TKN; $5.0 \pm 0.7 \text{ mg L}^{-1}$ for TP; $208 \pm 16 \text{ mg O}_2 \text{L}^{-1}$ for COD; and pH of 7.6. On the other hand, the autoclaved filtered effluents (AFE) had: $23.5 \pm 1.4 \text{ mg L}^{-1}$ of TKN; $5.6 \pm 0.6 \text{ mg L}^{-1}$ of TP; $162 \pm 10 \text{ mg O}_2 \text{L}^{-1}$ of COD and pH of 10.

2.3. Survivorship of the isolated strains in unsterilized filtered effluent (UFE)

To verify the survivorship potential of the isolated strains, Erlenmeyer flasks (50 mL) were filled with 30 mL of UFE and 1 mL of each inoculum ($n = 6$), closed with sterile cotton leads, then incubated with a temperature of $27 \pm 3^\circ\text{C}$; luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$; and 12 h/12 h (light/dark) photoperiod over 10 days. Fresh samples were taken daily to determine the cell density (N) with an Improved Neubauer Chamber and a microscope (Carl ZEISS Axioplan-2), using standard counting procedures ($n = 3$). The survivorship potential was determined according to the following Equation 1:

$$\% \text{ Survivorship} = \frac{N \text{ of the isolate}}{N \text{ of all microalgae}} \times 100 \quad (1)$$

Where the cell density (N) of a determined isolate was divided by the cell density of all the microalgae cells present in the counting fields and multiplied by 100.

2.4. Cultivation of the strains, characterization of the biomass and nutrient removal

Each isolated strain was cultivated in borosilicate glass flasks (22 L capacity) with 20 L of autoclaved filtered effluent (AFE) and its respective inoculum under the following conditions: temperature of $27 \pm 3^\circ\text{C}$; luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$; 12 h/12 h (light/dark) photoperiod and continuous air bubbling (1.5 mL s^{-1}). The cultures were monitored daily with an *in vivo* fluorometer during 14 days in order to establish the growth curves. *In vivo* chlorophyll *a* measurements were previously correlated with results from the acetone extraction method 10200-H (APHA *et al.*, 2005) for each microalgae strain. The extraction method was performed with 10 mL ($n = 3$) samples and the readings were carried out with a spectrophotometer DR/2000 (HACH).

On the 14th day the biomass was harvested with 250 mg L^{-1} of $\text{Al}_2(\text{SO}_4)_3$, centrifuged, dried (60°C) and pulverized with a pestle and mortar. The liquid phase was filtered with glass

fiber membrane filters (GF/C; 1.2 μm pore size) and analyzed for all the parameters within Section 2.2. in order to determine the nutrient and COD removal potential of each strain. The dried biomass was used for the biochemical characterization of lipids, proteins and carbohydrates as in Pereira *et al.* (2018).

3. RESULTS AND DISCUSSION

3.1. Microalgae isolation and identification

Microscopic examination of the algal bloom (obtained by incubating natural UASB effluent) showed the presence of several types of green microalgae, diatoms and cyanobacteria, as well as fungi and protozoans. Six Chlorophyta strains were isolated as described in Section 2.1 and identified based on their morphology as *Desmodesmus* sp. L02, *Chlorococcum* sp. L04, *Coccomyxa* sp. L05, *Chlorella* sp. L06, *Scenedesmus* sp. L08 and *Tetradasmus* sp. L09. These six strains were maintained in laboratory according to Lorenz *et al.* (2005) and were used as inocula for both the survivorship and cultivation experiments.

3.2. Survivorship of the isolated strains in unsterilized filtered effluent (UFE)

After the daily cell density monitoring of the cultures, survivorship curves were made for each strain, and the results are depicted in Figure 1.

Figure 1. Growth and survivorship curve of six microalgae strains cultured in unsterilized filtered effluent (UFE) in laboratory at a temperature of $27 \pm 3^\circ\text{C}$, luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$ and 12 h/12 h (light/dark) photoperiod over 10 days. Dashed lines represent the total cell density, including all microalgae, continuous lines represents the isolate's cell density and the orange diamonds represent the survivorship of the isolate at a given time.

Results showed that *Chlorella* sp. L06 was the most adaptable strain, being able to develop and grow in non-sterile wastewater despite the presence of competitors and predators. During the ten days of experiment, this strain had a survivorship of higher than 90% for each day, with an average of 97.7%. As for the other strains, the mean survivorship value was 1.2% for *Desmodesmus* sp. L02, 40.3% for *Chlorococcum* sp. L04, 16.3% for *Coccomyxa* sp. L05, 14.9% for *Scenedesmus* sp. L08 and 47.5% for *Tetradesmus* sp. L09.

Although all the strains were isolated from the same wastewater as the one used to cultivate them, it is natural to assume that different strains have different capacities to thrive in a determined environment. Some of them can be weaker competitors, or more susceptible to environmental changes. This seemed to be the case for *Desmodesmus* sp. L02, due to its inability to develop properly in the UFE medium. On the other hand, *Chlorella* sp. L06 had the best survivorship results, which was expected, considering that species from the genus *Chlorella* have been widely used in studies on cultivation of microalgae biomass linked to wastewater treatment (Lv *et al.*, 2017). A study conducted by Mennaa *et al.* (2015) compared the growth of several microalgae species on non-sterile urban wastewater and obtained better growth results for the *Chlorella* species, when compared with *Scenedesmus* species.

Cho *et al.* (2011) state that the effluents from WWTPs can be used to produce microalgae biomass at a much lower cost, since it does not require nutrient addition, only a pretreatment method such as filtration or UV disinfection for the control of competing microorganisms. The capacity to grow in non-sterile wastewater is a trait that many researchers consider crucial for a microalgae strain, considering the cost reduction in the pretreatment of the wastewater used as media (Guldhe *et al.*, 2017).

3.3. Growth of the isolated strains in autoclaved filtered effluent (AFE)

The microalgae strains were cultivated in 22 L flasks according to Section 2.4. and the growth results, based on chlorophyll *a*, are depicted in Figure 2.

Figure 2. Growth curve, based on chlorophyll *a*, of six microalgae strains cultured in autoclaved filtered effluent (AFE) in laboratory at a temperature of $27 \pm 3^\circ\text{C}$, luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$, 12 h/12 h (light/dark) photoperiod and air bubbling (1.5 mL s^{-1}) over 21 days. The dashed red line represents the day that biomass was harvested from a large sample of the cultures.

The growth results showed that all the isolated strains were capable of growing in AFE and reaching an exponential growth phase, indicating that this type of medium could support microalgae cultivation, despite the fact that its initial pH was 10. Microscopic examination showed that each microalgae culture was clearly dominated by the strain of its respective inoculum. Regarding each strain, *Chlorella* sp. L06 was not only the most adaptable one in UFE, it was also the strain with the best results on AFE with air bubbling. Following the same trend, *Desmodesmus* sp. L02 had the least satisfactory growth results, and *Tetradasmus* sp. L09 was the second best.

3.4. Biochemical characterization of the produced biomass

The dried biomass of each microalgae strain was characterized in terms of biochemical composition and the results are shown in Figure 3.

Figure 3. Biochemical composition of the dried biomass for each strain cultivated in autoclaved filtered effluent (AFE) in laboratory at a temperature of $27 \pm 3^\circ\text{C}$, luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$, 12 h/12 h (light/dark) photoperiod and air bubbling (1.5 mL s^{-1}) over 14 days (bars represent SD and $n = 3$).

The average composition of the biomass was 25.0% protein, 15.4% lipids and 14.8% carbohydrates, all within 66.0% of volatile solids. It can be observed that the protein content was higher than the other fractions in every case (except for *Scenedesmus* sp. L08), followed by lipids and carbohydrates. *Coccomyxa* sp. L05 had the highest protein content amongst the strains (39.7%) and *Scenedesmus* sp. L08 had the lowest (12.9%). Regarding biomass-to-bioenergy processes, the anaerobic digestion of the biomass is an attractive alternative to generate biogas within WWTPs that already use anaerobic systems. However, high protein content on the biomass (low C:N ratios) is often unfavorable for anaerobic digestion, considering the inhibitory effects that NH_3 has over the methanoarchaea community (Fricke *et al.*, 2007).

Mutanda *et al.* (2011) discuss that the co-digestion of the biomass with carbon-rich substrates and the pre-treatment of the biomass are both alternative strategies that can significantly enhance bio-conversion into methane. In fact, Passos *et al.* (2015) achieved higher methane production when the microalgae biomass was pre-treated with physical methods

(thermal, microwave and ultrasound). Other than the production of biogas through anaerobic digestion, the biomass can also be used in the production of ethanol through the fermentation of the carbohydrates. In this case, *Chlorococcum* sp. L04 deserves recognition due to the high carbohydrate content (29.3%).

3.5. Nutrient and COD removal efficiency

Table 1 shows the nutrient and COD removal efficiency from the liquid phase based on different microalgae cultures after the addition of the coagulant.

Table 1. Nutrients and COD concentration in the liquid phase of the microalgae cultures at the beginning of the experiment (in) and after the addition of the coagulant agent (out). The strains were cultivated in autoclaved filtered effluent (AFE) in laboratory at a temperature of $27 \pm 3^\circ\text{C}$, luminosity of $80 \mu\text{mol m}^{-2} \text{s}^{-1}$, 12 h/12 h (light/dark) photoperiod and air bubbling (1.5 mL s^{-1}) over 14 days ($\pm\text{SD}$ and $n = 3$).

Parameters	Units	Cultures/Strains					
		L02	L04	L05	L06	L08	L09
TKN (in)	mg L^{-1}	26.5 ± 0.3	26.5 ± 0.3	26.5 ± 0.3	26.5 ± 0.3	26.5 ± 0.3	26.5 ± 0.3
TKN (out)	mg L^{-1}	10.8 ± 0.3	5.6 ± 1.0	3.9 ± 1.8	8.9 ± 0.8	8.7 ± 0.3	1.8 ± 1.3
TKN removal	%	59.2	78.9	85.3	66.4	67.2	93.2
TP (in)	mg L^{-1}	7.1 ± 0.2	7.1 ± 0.2	7.1 ± 0.2	7.1 ± 0.2	7.1 ± 0.2	7.1 ± 0.2
TP (out)	mg L^{-1}	1.5 ± 0.0	0.7 ± 0.0	0.8 ± 0.0	0.7 ± 0.1	0.8 ± 0.2	0.3 ± 0.0
TP removal	%	78.9	90.1	88.7	90.1	88.7	95.8
COD (in)	mg L^{-1}	184 ± 6	184 ± 6	184 ± 6	184 ± 6	184 ± 6	184 ± 6
COD (out)	mg L^{-1}	93 ± 0.0	101 ± 4.0	82 ± 8.0	64 ± 5.0	76 ± 11.0	58 ± 13.0
COD removal	%	49.4	45.3	55.3	64.9	58.7	68.3

Abbreviations: TKN, total Kjeldahl nitrogen; TP, total phosphorus; COD, chemical oxygen demand.

It can be observed that, at the end of the experiment, the TKN removal varied from 59.2% (*Desmodesmus* sp. L02) to 93.2% (*Tetradismus* sp. L09). Regarding TP, removal efficiency was higher than 80% for all cultures (except *Desmodesmus* sp. L02), with *Tetradismus* sp. L09 reaching 95.8%. In terms of COD, it is interesting to note that its concentration was already low at the beginning of the experiment (184 mg L^{-1}), as expected, since secondary treated effluents from anaerobic treatments are generally low in COD and BOD. However, the removal efficiency of this organic fraction still ranged from 45.3% (*Chlorococcum* sp. L04) to higher than 68.3% (*Tetradismus* sp. L09). COD removal in this case may have been caused by the high photosynthetic O_2 generation inside the flasks, which could enhance the oxidation of organic matter by heterotrophic microorganisms. On the other hand, high COD removal can indicate microalgal mixotrophic nutrition, as discussed by Park *et al.* (2011). Adsorption by the biomass and or filter membrane may have also contributed to the COD removal, since a fraction of the COD is often recalcitrant, therefore difficult to assimilate biologically.

The results for removal efficiency obtained herein are similar to the ones obtained by Mennaa *et al.* (2015), and agree with the range of values reported in a recent review study on the subject of microalgae cultivation in secondary-treated wastewaters (Lv *et al.*, 2017). On the other hand, since the pH of all cultures reached values above 9.0, it might be reasonable to assume that at least part of the nitrogen was lost due to abiotic processes such as ammonia stripping, rather than assimilation by microorganisms (Nurdogan and Oswald, 1995). The same logic can be applied to phosphorus removal due to the chemical precipitation of this nutrient at pH levels higher than 9.0. Nonetheless, in terms of tertiary treatment, we can conclude that nitrogen, phosphorus and COD were efficiently removed from the liquid phase of the effluent in this experiment.

4. CONCLUSIONS

The results showed that *Chlorella* sp. L06 and *Tetrademus* sp. L09 were able to survive and thrive in unsterilized UASB effluent after inoculation, indicating that they were more adapted to this medium than the other microalgae isolates, in terms of competition for nutrients and resiliency to protozoan grazers. The strains were also cultivated in autoclaved UASB effluent, and all of them were able to grow and reach the exponential growth phase.

The dry biomass of the isolates showed, on average, 66.0% of volatile solids, of which 25.0% was composed of proteins, 15.4% of total lipids and 14.8% of total carbohydrates. The microalgae cultures were able to remove up to 93% of nitrogen and 95.4% of phosphorus from the wastewater's liquid phase, improving the UASB effluent quality and converting these nutrients into biomass that can be used as feedstock for the production of biogas or syngas.

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Removal of organic matter from pre-treated domestic sewage in anaerobic biological reactor by a combined system of electrolytic and biological aerobic treatment

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ABSTRACT

Biological processes are the most widespread methods for wastewater treatment. However, they are limited in their ability to degrade toxic and refractory pollutants, contaminants that electrochemical processes can remove. Therefore, this research explored the possibility of treating sewage by an anaerobic biological process followed by an aerobic system integrated to an electrolytic process. Three sequential batch reactors were operated in an automated way. Each of three reactors represented a process: aerobic biological treatment (BR); electrolytic treatment (ER); and a combination of both, the bio electrolytic reactor (BER). Two phases were ran with different electrodes: (Phase 1) stainless steel and (Phase 2) graphite. The electric current was varied from 0.001 to 0.100 A. COD, TS, SS, turbidity, and the zooplankton community were monitored. The highest organic matter removal efficiencies were 86%, 79% and 87% for BR, ER and BER, respectively. The best weekly BER efficiencies for COD removal were 90% and 98%, with current densities of 0.27 A/m² (Phase 1) and 0.05 A/m² (Phase 2). The main conclusions about bio electrolytic process were: (1) it did not achieve organic matter removal high enough to justify its application; (2) inert electrodes are the more indicated; and (3) the zooplankton community was affected by the electric current.

Keywords: bioelectrolytic reactor, electrolytic treatment, removal of organic matter.

Remoção de matéria orgânica de esgoto doméstico pré-tratado em reator biológico anaeróbico por um sistema combinado de tratamento eletrolítico e biológico aeróbico

RESUMO

Os processos biológicos são os métodos mais difundidos para o tratamento de águas residuárias. No entanto, eles têm limitações para degradar poluentes tóxicos e refratários, contaminantes, que os processos eletroquímicos podem remover. Portanto, o objetivo da pesquisa foi verificar a possibilidade de tratamento de esgoto por um processo biológico anaeróbico seguido de um sistema aeróbico integrado a um processo eletrolítico. Três reatores

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sequenciais em batelada foram operados de maneira automatizada. Cada um dos três reatores representou um processo: tratamento biológico aeróbico (RB); tratamento eletrolítico (RE); e a fusão de ambos, o reator bioeletrolítico (RBE). Duas fases foram executadas com diferentes eletrodos: (Fase 1) de aço inoxidável e (Fase 2) de grafite. A corrente elétrica foi variada de 0.001 a 0.100 A. DQO, ST, SS, turbidez e comunidade zooplânctônica foram monitorados. As maiores eficiências de remoção de matéria orgânica foram 86%, 79% e 87% para BR, RE e RBE, respectivamente. As melhores eficiências semanais de RBE para remoção de DQO foram 90% e 98% com densidades de corrente de 0.27 A/m² (Fase 1) e 0.05 A/m² (Fase 2). As principais conclusões sobre o processo bioeletrolítico foram: (1) eles não atingem remoção de matéria orgânica tão alta que justifique sua aplicação; (2) os eletrodos inertes são os mais indicados; e (3) a comunidade zooplânctônica foi afetada pela corrente elétrica.

Palavras-chave: reator bioeletrolítico, remoção de matéria orgânica, tratamento eletrolítico.

1. INTRODUCTION

The most-used processes for sewage treatment are biological treatments. However, these treatment processes have limitations in the removal of organic matter and solids, require a great deal of space, are time-consuming, and do not tend to be entirely effective against barely biodegradable and toxic pollutants (Martínez-Huitle *et al.*, 2015). Further, biological methods also give low degradation efficiency in the treatment of highly concentrated and biorefractory pollutants, especially certain industrial wastewaters containing toxic components such as pesticides, because the activity of microorganisms is inhibited by toxicity. To treat this kind of wastewater, many physical and chemical methods have been introduced (Liu *et al.*, 2010). In relation to anaerobic treatment, its effluents have high BOD concentrations, color and unpleasant odor, resulting in the need for complementary treatment. Therefore, there is a global trend to improve existing sewage treatment processes and continuously enhance the quality of treated effluents, with the purpose of adapting them to environmental requirements and water reuse.

Electrolytic treatment is one of the complementary alternatives to anaerobic biological treatment. In the last few years, electrolytic treatment was associated with the anaerobic or aerobic biological treatment. According to Ganzenko *et al.* (2014), these combined processes are studied before or after electrolytic treatment. In the first case, the goal is to make biodegradable the effluent for subsequent biological treatment. In the second case, the electrolytic process is located after the biological treatment to remove pollutants and their intermediates, which are refractory to anaerobic bacteria. Electrolytic treatment can reduce the effluents' toxicity by transforming persistent substances into biodegradable substances. In addition to the efficiency of electrolytic treatment, this kind of sewage treatment also has several advantages: it uses compact reactors with a smaller physical footprint requiring less land space; there is no need for auxiliary chemicals, which obviates the need to store and transport such chemicals; it can be easily combined with other conventional water treatment technologies; and it can be fully automated (Garcia-Segura *et al.*, 2018).

Electrolytic treatment therefore allows the increasing of the efficiency of conventional biological treatment (Claro *et al.*, 2010). Electrolytic processes have been an option for the wastewater treatment, and they can be used as a single process or coupled with other processes. This type of treatment can be adapted to various applications and can be easily combined with other technologies, and its modular and compact design makes it attractive for the treatment of decentralized wastewater (Radjenovic and Sedlak, 2015). Materials commonly used to make the electrodes are aluminum or iron, because they are cheap and easy to access. However, other materials have been studied, such as oxides of graphite, titanium, ruthenium, platinum, copper

and zinc. When an electrode, during the electrolysis process, does not undergo changes in its structure, even under certain conditions, it is called an inert electrode and its function is the simple exchange of electrons with the solution; examples of inert electrodes are graphite and platinum. The active electrode, besides transferring electrons, chemically participates in electrolysis, being also modified by the action of the electric current, forming metallic ions; examples of active electrodes are iron and copper. In oxidation processes, researchers have been using graphite electrodes, because of their high porosity, which results in a larger surface area. In addition, electrodes composed of platinum, aluminum and steel have been extensively applied (Mattiusi *et al.*, 2015).

The main purpose of this paper was to study the possibility of performing the effluent treatment of an anaerobic biological system by the aerobic biological treatment combined with the electrolytic process, known as bio electrolytic treatment. Another purpose was to verify if there is synergy between the aerobic biological process and the electrolytic process for the removal of organic matter from sewage. In addition, the zooplankton community was evaluated and compared in the aerobic biological process and in the integrated bio electrolytic process, and relations were explored between the relative abundances of the organisms groups with the performance of the sewage treatment processes.

2. MATERIALS AND METHODS

2.1. Design and construction of the bench scale electrolytic system

Figure 1 shows the schematic diagram of the experimental apparatus for operating the bench scale electrolytic system that was conceived for this research. A photograph of the complete experimental system installed in the laboratory can be seen in Figure 2. The system was composed of three glass reactors of 4 liters volume each; the useful volume was 3.6 L. The adopted configuration was an activated sludge as an aerobic biological reactor (BR), an electrolytic reactor (ER) and a bio electrolytic reactor, which combined the two processes, activated sludge and electrolytic (BER). The system ran in an automated way and in sequential batches, each reactor running in parallel with others. The system operation was divided into two operational phases. In each phase, the type of electrodes used was alternated. At the first phase, as electrodes, brushed stainless steel n° 430, was used with a size of 13x14 cm, a thickness of 1.0 mm, a density of 7.8 g/cm³, and a resistivity of 0.06 μΩm. In the second phase of operation, the electrode applied was the pure graphite plate with density of 1.75 g/cm³, resistivity of 7.5 μΩm, size of 13 x 14 cm, and thickness of 2.7 mm. The automatic operation of the system was possible due to an Arduino microcontroller. This device had the function of controlling the operation of the cycles in the sequential batches, as well as the duration times of each batch phase, the filling and discharge of the reactors, activation of the aerators and the electric source. An Arduino Mega 2560 R3 was used due to the cost of acquisition and to fit the technical needs.

2.2. Experimental design

Due to the large variation in the domestic wastewater composition, synthetic wastewater (SW) was used to simulate domestic sewage pretreated by an anaerobic treatment. For this purpose, the effluent of a local study (Achaval, 2014) was chosen. Achaval (2014) developed a prototype of a compact wastewater treatment plant for single-family residential units. For the preparation of SW, the methodology used was that proposed by Torres *et al.* (1996) and used by Moura (2011) and Michelan (2006). For this research, an SW was used with approximately 265 mg COD. L⁻¹. The inoculum used in the first and second phase was the mixed liquor collected and transported from the return well of a biological aerobic reactor of the Sewage Treatment Plants of Brasília - South and North, respectively.

Figure 1. Schematic diagram of the experimental apparatus.

Figure 2. Photograph of the complete experimental system installed in the laboratory.

For the beginning of reactors operation, the inoculum was brought and introduced in the BR and the BER reactors, transferring the amount of 1.08 liters per reactor, equivalent to 30% of the useful volume of each reactor. During the day, the system was operated in two batches of three hours of reaction and one hour of sedimentation, totaling 4 hours of duration for each batch. During the night, only a single batch was carried out for 12 hours, with 11 hours of reaction and 1 hour of sedimentation. The filling and disposal steps were performed quickly at the beginning and end of each batch. This operation model was chosen to prevent system

interruptions caused by failures in the electric power supply, which would result in the system reboot. In addition, the prototype operation model tried to reproduce a single-family sewage treatment system, in which during the night period the inlet flow is small or non-existent, resulting in a long duty cycle at night. Each system operational phase was fixed in 25 days. Every 5 days a different electric current was applied. Each application of electrical current band started on Monday and ended on Friday. There were no electric current applications on weekends. Each band of electric current applied weekly was called the “experimental condition”. Therefore, a different experimental condition was applied each week of operation, which corresponds to the electric current ranges previously defined for the experiment. The current ranges were suggested according to the electric source specifications and the literature studied. Concerning the current variation in the reactors and the operation of each cycle, it is possible to understand the operation model in Tables 1 and 2.

Table 1. Operation of Biological Reactor (BR), Electrolytic Reactor (ER) and Bio Electrolytic Reactor (BER).

Stages of the cycle	Duration	Electric Current (A)	Aeration	Electrical Current (status)		
		BR, ER e BER		BR	ER	BER
Filling	1 min	-	On	Off	Off	Off
Reaction	180 min	0.001 a 0.100	On	Off	On	On
Sedimentation	60 min	-	Off	Off	Off	Off
Disposal of effluent	2 min	-	Off	Off	Off	Off

Table 2. Experimental conditions, current ranges and currents densities applied to the system during the operational phases.

Experimental Conditions	Current ranges (A)	Current densities (A/m ²)
1° Condition	0.001	0.0549
2° Condition	0.005	0.2747
3° Condition	0.010	0.5494
4° Condition	0.050	2.7473
5° Condition	0.100	5.4945

2.3. Sampling and analyses performed on the effluent

Samples were collected daily and weekly. The sample collection was carried out in the effluent inside the reactors (mixed liquor) and in the treated effluent. Sampling was performed in a composite manner, starting from the first cycle of the day. Simple samples were collected each cycle, and at the end of the day, the samples were homogenized and transferred to a specific bottle and refrigerated until the next day. This procedure was carried out from Monday to Thursday; on Friday, the samples collected were acidified to retain their characteristics until the analysis performed on Monday. All analysis were performed according to the standards established by the Standard Methods for the Examination of Water and Wastewater (APHA *et al.*, 2005). The analysis performed and the method followed for each one can be visualized in Table 3.

Table 3. Analysis performed on mixed liquor (ML) and effluent (EF).

Parameter to be analyzed	Point of collect	Weekly frequency	Method
Chemical demand for oxygen	EF	Daily	Colorimetric
Total suspended solids	ML / EF	1	Gravimetric
Turbidity	EF	2	Nephelometric Method
Dissolved oxygen	ML	Daily	Oximeter
Count of microorganisms	ML	2	Method of Madoni (1994)

As in the activated sludge system, the low illumination condition is not favorable for the development of the phytoplankton community; it was chosen to observe only the zooplankton community. Zooplankton monitoring was performed by the identification and counting of protozoa and metazoan, always in duplicate, presented in the mixed liquor of the BR and BER reactors. For each experimental condition, two micro fauna analysis corresponding to the beginning and the end of the electric current application in the reactors were performed. For identification and counting of zooplankton, the methodology proposed by Madoni (1994) was followed. In order to characterize the zooplankton community present in the mixed liquor of the BR and BER reactors, the proper systematics for typical microorganisms of activated sludge systems was used, as proposed by Canler *et al.* (1999) and Madoni (1994). For this characterization, microorganisms were identified up to the gender level, and were grouped into eight groups as follows:

- Amoebas: Represented by the naked amoebas and testae amoebas of the *Arcella* genus;
- Annelids: Represented by order oligochaeta, *Aelosoma* genus;
- Crawling ciliates: Represented by *aspidisca*, *chilodonella*, *drepano monas*, *Trachelophyllum* and *Trochilia* genuses;
- Free swimming ciliates: Represented by the genera *Colpidium*, *Tetrahymena* and *Litonotus*;
- Attached Ciliates: Represented by *Epistylis*, *Vorticella*, *Podophrya*, *Tokophrya* and *Acienta* genuses;
- Big flagellates: Represented by *Peranema* genus;
- Nematode: Represented by *Nematodes* genus; and
- Rotifers: Represented by *Digononta* e *Monogononta* orders;

To facilitate the results exhibition in the graphs, the groups of organisms were abbreviated. Table 4 shows abbreviation subtitles.

Table 4. Subtitle of the names of the groups of microorganisms.

Organism	Abbreviation
Amoebae	AM
Annelids	AN
Crawling ciliates	CC
Free swimming ciliates	FSC
Attached Ciliates	AC
Big Flagellates	BF
Nematodes	NE
Rotifers	RO

3. RESULTS AND DISCUSSION

The use of electrodes of different materials promoted changes in the characteristics of treated effluents. The first phase (with stainless steel) lasted four weeks due to operational problems. The second phase (with pure graphite) ran normally under the predicted conditions during five weeks. Table 5 shows the weekly COD removal efficiency values obtained at the three points analyzed during the two operational phases.

Table 5. Weekly medium COD removal efficiency values, obtained at the three points analyzed during the two phases of the experimental system operation.

	COD Removal Efficiency (%)					
	1° phase (with stainless steel)			2° phase (with pure graphite)		
	BR	ER	BER	BR	ER	BER
1° Week	90.44	81.35	78.09	95.10	67.60	98.20
2° Week	82.01	53.90	89.69	87.40	70.30	85.70
3° Week	85.14	65.88	80.77	79.20	73.20	81.00
4° Week	78.35	77.07	72.45	83.70	90.20	80.90
5° Week	-	-	-	82.00	92.20	88.20

BR = Biological reactor, ER= Electrolytic reactor, BER= Bioelectrolytic reactor.

It was noticed that the BER best performance in the first phase was reached on the second week of operation, while in the second phase the best performance occurred in the first week. In the first phase, reactive electrodes of stainless steel were used, so it is probable that, due to the electrolysis process, part of the iron that comprises this electrode has been lost in the water, added to the sludge and precipitate, avoiding that it was discarded with the effluent. According to Al-Shannag *et al.* (2013), when iron is used in the electrocoagulation process, the formation of Fe (III) ions is favorable. In a basic environment, simultaneous formations of ferric hydroxide/oxides are also expected. Precipitates of formed Fe (III) hydroxides have a coagulating character better than Fe (II) hydroxide, because Fe (OH)₃ is more stable than Fe (OH)₂, so the removal efficiency increases. It is important to note that the samples submitted to the COD tests were aerated during the batch reaction phase. This suggests that the effluent from the ER and BER reactors contained only Fe³⁺, which does not interfere in the COD test because these cations are in their maximum oxidation state. In the second phase, where graphite electrodes were used, the optimum efficiency value was reached in the first week. This can indicate that, with a current equivalent to 0.001 A, the microorganisms may not have been affected abruptly and current may have acted on a positive threshold for the micro fauna. However, in the first and second phases, during the weeks of operation, there were oscillations in COD removal performances, suggesting that the microorganisms present in the BER reactor may have been affected by the variations and increasing of the electric current.

Regarding the ER reactor in the first phase of operation, the best COD removal performance occurred in the first week. In the second phase, it occurred in the last operational week. The result obtained in the first phase was unexpected, because in the electrolytic treatment the higher the electric current applied, the greater the treatment efficiency. However, considering the usage of stainless steel electrodes in this phase, it is known that due to the oxidation of the electrode, an iron release occurs which may have caused interference in the COD analysis.

In the second phase, it was noticed that, with the increase of the electric current during the weeks of operation, the ER reactor's COD removal efficiency increased, confirming the premise of the electrolytic treatment. The COD reduction in the ER reactor increased with current density. This can be explained by the increase in the release of metallic ions with the increase in current density, which improves COD reduction. (Senthilkumar *et al.*, 2012). The BR reactor showed similar behavior in the two phases of operation. There were fluctuations in the removal values and a pattern of decreased performance was noted over the weeks of operation. This fact suggests an affectation of the microbial community in the BR reactor probably due to operational factors.

The overall COD removal performance in the first phase of the four weeks of operation were 84%, 70% and 80% of COD removal in BR, ER and BER reactors, respectively. In the

second phase, overall performance removal of the five weeks of operation was 85%, 79% and 87% in the reactors BR, ER and BER, respectively. The best performance was obtained in the second phase of operation, and BR and BER reactors presented similar results. The performance achieved by the BR reactor in the first and second phases, 84 and 85% of COD removal, was lower than that demonstrated by Santos (2005), who worked with Sequential Batch Reactor (SBR) under different sludge ages in bench scale for domestic wastewater treatment and reached 90% in COD removal. The BR reactor performances in both phases also were lower than that achieved by Chonova *et al.* (2016), who evaluated the efficiency of biological treatment with a conventional activated sludge system for domestic wastewater treatment and reached 95.4% COD removal. Comparing this reactor with a full-scale plant, it was found that the BR reactor also had an efficiency lower than expected, since the activated sludge reactor of the Brasília South Sewage Treatment Plant obtained a COD removal of 92.4% in the year 2013 (CAESB, 2013). The oscillations in COD removal efficiencies in the BR reactor in the two phases can be attributed to the model of operation adopted in the system. During the night, only a single and long batch was carried out for 12 hours, with 11 hours of reaction and 1 hour of sedimentation. Therefore, it can be considered that the biomass was affected and the F/M ratio was reduced drastically overnight due to batch duration. According to Von Sperling (1997), when the F/M ratio is low, when the amount of food supplied to biomass is reduced, the endogenous respiration rate prevails in the system. In this case, in order to ensure the survival of microorganisms, oxygen consumption by the cells occurs to degradation of the substrate present in the bacterial cell itself.

On the other hand, the COD removal efficiency reached by ER reactor was 70% in the first phase. This result is close to that of the research of Rodrigues *et al.* (2001), who worked with iron and aluminum electrodes treating effluent from a UASB reactor and obtained COD removal efficiency of 74.74% in the reactors. This result also is compatible with the results obtained by Daghrir *et al.* (2014), who worked with bored doped diamond anodes treating effluent from domestic wastewater and obtained COD removal efficiency of 78.2%. Furthermore, the work of Al-Shannag *et al.* (2013) may be cited, who operated a system to enhance the COD and nutrient removal of a secondary clarifier of municipal wastewater, using electrocoagulation technique with an iron electrode, and obtained steady removal efficiencies of COD and nutrients exceeding 89%, surpassing the result of this research. In the second phase, the efficiency result of the ER reactor was higher than that of Rodrigues *et al.* (2001), very close to that found by Daghrir *et al.* (2014), and smaller than that found by Al-Shannag *et al.* (2013). Briefly, in the second phase, due to the characteristics of the electrodes, since they were inert, it was noticed that the increase of the COD removal occurred with the increase of the electric current, according to the rules of electrolysis. In the integrated biological and electrolytic process, Senthilkumar *et al.* (2012) obtained 80% COD removal, the same result reached by the BER reactor in the first phase and lower than that of the second phase, which was 87%.

Regarding the efficiency of Total Suspended Solids (TSS) removal, in the first phase 93% was removed in the BR, 47% in the ER and 95% in the BER reactor. In the second phase, the results were 84% in the BR, 38% in the ER and 76% in the BER reactor. It can be stated that the highest values of TSS removal were reached in the decantation of the BR and BER effluents. This was possibly due to the optimum sedimentation of the effluent and due to the fact that the reactors have biological solids inside them. In the first phase, there was the aggregation of dissolved iron in the BER biomass, promoting the retention of part of the solids inside the reactor. In the ER, the mean removal was lower during the two phases, which may have been caused by the accumulation of material from the SW inside the reactor.

The turbidity results obtained in the treated effluents of the reactors indicate that the values corresponding to the effluent from the biological reactor were similar in the two phases, 9 and

8 NTU (Nephelometric Turbidity Unit), for the first and the second phase. However, in the first phase of operation, the highest discrepancy between turbidity values of the effluents was observed, 5 and 40 NTU for the ER and the BER, and in the second phase, the values were 13 and 11 NTU. The discrepancy between the values of the first and second phases can be attributed to the use of reactive electrodes and to their rapid wear. Concerning dissolved oxygen (DO) concentrations in the reactors, it was verified that the values were above 2 mg.L⁻¹, the minimum concentration recommended by the USEPA (1993). In the first phase, the DO concentration remained at 5.75 mg.L⁻¹ O₂ ± 0.73 in the BR and 6.14 mg.L⁻¹ O₂ ± 0.29 in the BER reactor. In the second phase, these values were 5.99 mg.L⁻¹ O₂ ± 0.27 and 5.95 mg.L⁻¹ O₂ ± 0.30 in the BR and the BER reactors, respectively. Therefore, aeration conditions were maintained for the development of heterotrophic aerobic biomass in the reactors.

The first phase of operation lasted four weeks. Consequently, four experimental conditions were applied in this phase, because each week represented an experimental condition. Stainless steel electrodes were used. In each week of operation two micro fauna analysis corresponding to the beginning and the end of the current application in the BR reactors and the BER were carried out. In the second stage of operation, pure graphite rigid electrodes were used. This phase lasted five weeks, where five experimental conditions were applied. However, the quantitative and qualitative characterization of the biomass began to be performed from the second week of operation, which represents the second experimental condition applied.

During the first operational phase, there were significant variations in the reactors' micro fauna. At the beginning of the first week of operation, corresponding to the first experimental condition, it was observed that in the two reactors, BR and BER, there was little variety of group of organisms (Figure 3). However, *Monogononta* and *Digononta*, organisms of the rotifers class, presented themselves as dominant species. At the end of the period, an increase in micro fauna diversity and in the ciliates relative frequency was noticed, accompanied by a reduction in the abundance of rotifers.

Figure 3. Microorganisms relative frequency in BR and BER of the third week of operation corresponding to the third experimental condition applied. The labels of the organisms are in accordance with Table 4.

In the beginning of the second experimental condition, at the first phase, the crawling ciliates appeared with the highest relative frequencies in the BR and BER reactors (Figure 4). At the end of the period, it was noticed that species diversity was affected in the two reactors, but the BR was more affected. It was also noticed that the amoebas became dominant in the BR and BER reactors. During this experimental condition, at the second phase, it was noticed at the beginning that the dominant groups were rotifers and attached ciliates, in the BR and BER reactors. However, at the end of this period the attached ciliates, a dominant species at the

beginning of the week, gave way to the rotifers and swimming ciliates, which predominated in the BER. This behavior was similar in the two reactors, so this fact was not due to the electric current application.

Figure 4. Microorganisms' relative frequency in the BR and BER during the second week of operation, corresponding to the second experimental condition applied. The labels of the organisms are in accordance with Table 4.

During the third experimental condition, at the first phase, at the beginning it was observed that the two reactors presented similar diversity (Figure 5). At the end of this period, no affectation of the microorganisms was observed with respect to their diversity, being that the predominant class in the reactors was the attached ciliates of the genus *Epistylis*. In the third experimental condition, at the second phase, a decrease in the number of species of microorganisms presented in the BER reactor was observed. The BR reactor remained stable in relation to the diversity of groups present. At the end of this period, there was a recovery of the reactors with the development of species and with the increase of the microorganisms diversity.

Figure 5. Microorganisms' relative frequency in the BR and BER during the third week of operation, corresponding to the third experimental condition applied. The labels of the organisms are in accordance with Table 4.

Still referring to the first phase of operation, in the fourth experimental condition, the attached ciliates were the dominant species, but in the BER, also amoebas and nematodes were found (Figure 6). The rotifers and annelids disappeared in the BER, whereas in the RB the relative frequencies of these organisms increased, a fact that reinforces the possibility of the electric current influencing the microorganisms. The reduction of rotifers and annelids in BER may be indicative of damage to the system performance once these organisms represent satisfactory aeration conditions and effluent quality. During the fourth experimental condition,

at the second phase, the predominance of rotifers and annelids in both reactors was observed. At the end of the period, it was noticed that the rotifers were dominant in the BER. However, there was an annelids community increasing, represented by *Aelosomas*.

Figure 6. Microorganisms' relative frequency in the BR and BER during the fourth week of operation, corresponding to the fourth experimental condition applied. The labels of the organisms are in accordance with Table 4.

Finally, at the beginning of the fourth experimental condition, it was observed in the BER a diversity reduction of microorganisms (Figure 7). At the end of this period, the BER showed the same amount of species present at the beginning of this experimental condition for the attached ciliates. At the beginning of the fifth and final experimental condition evaluated, the annelids were the dominant group in the two reactors. At the end of the period, the BER reactor showed the highest reduction in the diversity of microorganisms presented in the biomass of this reactor, with only two species, rotifers and annelids, which can be associated to the negative effect of the electric current on the BER. It is suggested that there was interference of the electric current in the microbial metabolism in the first and second phase of operation. However, it cannot be stated to what extent electrostimulation of microorganisms positively or negatively affected the biomass.

Figure 7. Microorganisms' relative frequency in the BR and BER of the fifth week of operation, corresponding to the fifth experimental condition applied. The labels of the organisms are in accordance with Table 4.

In the first stage of operation, the formation of an iron hydroxide precipitate increased according to the increase of the applied electric current. Therefore, it is hard to make inferences from the COD values, since it is known that this analysis was impaired mainly in the last experimental condition with the highest electric current value tested. It can be suggested that the experimental conditions imposed on the BER reactor contributed to affect the zooplankton community. It can be affirmed that part of the oxygen that was being supplied to this reactor was consumed in the iron oxidation. Thus, it is probable that the aerobic degradation of organic matter and the nitrification process itself have been affected, reaching anoxic conditions in the system.

According to Thrash and Coates (2008), studies indicate that electrical stimulation can negatively affect growth and microbial activity; however, using appropriate currents, it is possible to promote microbial metabolism. Wei *et al.* (2011) demonstrated that the heterotrophic bacterial community was not significantly affected when the applied current density was less than 6.2 A/m², but was partially inactivated at current densities above 12.3 A/m². The current densities applied in the first and second phase of operation in this study (0.0549, 0.2747, 0.5495, 2.7473 and 5.4945 A/m²) were lower than 6.2 A/m². Therefore, it would be expected that in the studied system there would be no immediate inactivation of microorganisms, a process that occurred gradually with the increase of the applied electric current. According to Matsunaga *et al.* (1992), electrical stimulation can cause negative effects on bacteria, directly oxidizing intracellular constituents without destroying their membranes, leading to cellular inactivation. High electrical current can also cause irreversible permeability of the cell membrane and subsequent release of essential cytoplasmic constituents (Drees *et al.*, 2003). In addition, the literature indicates that electrolysis generates products derived from abiotic reactions on the electrode surface that may influence microbial metabolism, such as hydrogen peroxide, which may inhibit microbial metabolic activities (Thrash and Coates, 2008).

However, during the system operation, it was verified that the bio electrolytic reactor obtained similar values in terms of COD removal, and sometimes it was higher than the biological reactor. This may indicate a positive interaction between the zooplankton community in the bio electrolytic reactor and the electrical current application. However, it was not possible to state through this work that this interaction is beneficial for microorganisms.

4. CONCLUSIONS

The differences between COD removal in the bio electrolytic process during the two phases came from the different application of materials of the electrodes and due to the different origins of the inoculum used. The best results were achieved with rigid pure graphite electrodes, suggesting that non-reactive electrodes are more indicated for the process. According to the microbiological analysis, it was possible to identify that the zooplankton community was progressively affected due to electric current application in the bio electrolytic reactor during system operation. This fact was confirmed by the drastic reduction of diversity and relative frequency of the zooplankton community, indicating a possible electric current interference in the microbial metabolism during operation. With the design of the bio electrolytic reactor proposed in this research, this reactor did not reach sufficiently high organic matter removal efficiency when compared to the efficiency of the biological reactor to justify its application. However, even in this configuration (electrolytic and biological treatment in the same vessel), it is expected that such behavior can be improved when treating an effluent containing a toxic or recalcitrant compound.

As for the electrolytic reactor, the results suggest that, even under very low electrical currents, it was possible to achieve high COD removal rates under the conditions studied. It

should be remembered that one of the criticisms of the electrolytic reactor treating sewage is that its energy consumption is supposedly very high.

In order to subsidize future works on the electrolytic and bio electrolytic treatment applied to the treatment of sewage, some recommendations are suggested. For example, a BER test in separate reactors, with the electrolytic treatment preceding the biological reactor and vice versa. In addition, it is also proposed to evaluate the bio electrolytic reactor efficiency in order to promote the degradation and removal of difficult compounds to treat.

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Sensitivity of hydrological connectivity in a semiarid basin with a high-density reservoir network

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ABSTRACT

Water reservoirs, in general, have been considered to be the major cause of reduction of downstream hydrological connectivity in channels. Therefore, this study analyzed the sensitivity of hydrological connectivity in the Orós Reservoir hydrographic basin by using the ResNet model, designed to simulate the processes involved in fluvial hydrological connectivity in environments with a high density of reservoirs. The analysis of hydrological connectivity was performed with the model ResNetM, which simulated hydrological processes and considered hydrological connectivity between the reservoirs, according to the criteria established in this research. To identify the main elements that affect hydrological connectivity, sensitivity analysis (SI) was performed of some input parameters of the model. The sensitivity analysis indicated that the modification of the topology of the reservoir network was the variable that presented the highest sensitivity to hydrological connectivity, with a sensitivity value of 1.07, followed by the runoff coefficient, which obtained a sensitivity of 0.8. The modification of the rainfall and of the reservoir storage capacity, showed an intermediate sensitivity, with values of 0.46 and 0.45, respectively. On the other hand, the parameters of potential evaporation and transmission loss showed the lowest sensitivity, obtaining values of 0.19 and 0.01, respectively. In conclusion, the runoff coefficient and the reservoir network (change in the reservoir number of the network) were the parameters evaluated with the highest sensitivity of hydrological connectivity. Thus, the alteration of the landscape by man provides significant changes in river navigation between the reservoirs in the basin.

Keywords: climate changes, hydrological modeling, sensitivity analysis.

Sensibilidade da conectividade hidrológica em uma bacia semiárida com uma rede de alta densidade de reservatórios

RESUMO

Os reservatórios de água, em geral, têm sido considerados a principal causa de redução da conectividade hidrológica a jusante nos canais. Portanto, este estudo tem como objetivo analisar a sensibilidade da conectividade hidrológica na bacia hidrográfica do reservatório de Orós, utilizando o modelo ResNet, desenvolvido para simular os processos envolvidos na

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conectividade hidrológica fluvial em ambientes com alta densidade de reservatórios. A análise da conectividade hidrológica foi realizada com o modelo ResNetM, que simulou processos hidrológicos e considerou a conectividade hidrológica entre os reservatórios, de acordo com os critérios estabelecidos nesta pesquisa. Para identificar os principais elementos que afetam a conectividade hidrológica, foi realizada a análise de sensibilidade (SI) de alguns parâmetros de entrada do modelo. A análise de sensibilidade indicou que a modificação da topologia da rede de reservatórios foi a variável que apresentou maior sensibilidade à conectividade hidrológica, com um valor de sensibilidade de 1,07, seguido do coeficiente de escoamento, que obteve uma sensibilidade de 0,8. A modificação da precipitação pluviométrica e da capacidade de armazenamento do reservatório mostrou uma sensibilidade intermediária, com valores de 0,46 e 0,45, respectivamente. Por outro lado, os parâmetros de potencial evaporação e perda de transmissão, apresentaram a menor sensibilidade, obtendo valores de 0,19 e 0,01, respectivamente. Conclui-se que o coeficiente de escoamento e a rede de reservatórios (mudança no número do reservatório da rede) foram os parâmetros avaliados com a maior sensibilidade de conectividade hidrológica. Assim, a alteração da paisagem pelo homem, proporciona mudanças significativas na navegação fluvial entre os reservatórios da bacia.

Palavras-chave: análise de sensibilidade, modelagem hidrológica, mudanças climáticas.

1. INTRODUCTION

In the northeast region of Brazil, classified as a semi-arid region, the construction of reservoirs has been the main decision adopted by water resource managers and by the civil population in order to try to solve the problem of water scarcity in the region over time (Malveira *et al.*, 2012; Toledo *et al.*, 2014). On average, there is one severe drought every decade, which sometimes lasts for many years (Araújo and Bronstert, 2015) - for example, from 2012 to 2015 (Marengo *et al.*, 2017). This reservoir construction policy often occurs without any control or use of technical-scientific knowledge. As a result, a “high-density reservoir network” is produced, with predominantly small- and medium-sized reservoirs, which turns out to be a complex system to manage (Araújo and Piedra, 2009).

Reservoirs have been considered to be the major cause of changes to water flow dynamics in water bodies, thus provoking disruption of hydrological connectivity. Hydrological connectivity in channels or rivers has been evaluated by analysis of water flow continuity in their riverbeds. For this purpose, a minimum flow rate index is used to characterize such hydrological connectivity (Brozovic *et al.*, 2011; Fryirs, 2012; López-Vicente *et al.*, 2013; Garbin *et al.*, 2019). This flow can be measured by fluvimetric methods, by injection dilution gauging (Burke, 2009) and also by simulations in hydrological models (Döll *et al.*, 2003; Hanasaki *et al.*, 2010; Malveira *et al.*, 2012; Sun *et al.*, 2015). Lane *et al.* (2009) highlight that hydrological models have a very important role in hydrological connectivity, as they enable temporal variation and structural connectivity to be checked.

A hydrological model can be defined as a tool of mathematical representation of water flow rate and its interactions with the atmosphere and the terrestrial surface; in other words, representation of hydrological processes that occur in a water basin. This mathematical representation uses a series of parameters, which simulate the characteristics of the environment and modeled physical processes; often, it requires previous calibration and/or sensitivity analysis of these parameters. (Yang *et al.*, 2015; Silva *et al.*, 2015).

Sensitivity analysis enables the identification and definition of the input elements of a model that cause the greatest disturbance in the performance of the output results, that is, how much the output values are affected by changes in input values (Loucks and Van Beek, 2005). Sensitivity analysis helps users to define which elements should be estimated with more

precision or accuracy, because these are of great relevance for the processes being evaluated. Kruk *et al.* (2009) noted that knowledge of the sensitivity of the elements of a hydrological model is extremely important to direct field research and to select parameters for future research.

In this context, the general objective of the present study was to perform sensitivity analysis of hydrological connectivity in the water basin of Orós Reservoir by using the ResNet model, designed to simulate the processes involved in fluvial hydrological connectivity in environments with a high density of reservoirs.

2. MATERIALS AND METHODS

The study area is the Orós Reservoir Basin, which is nestled at the Alto Jaguaribe Basin (BAJ). The basin is located in the southeast portion of the State of Ceará, in 24 South zone, between the 292000 and 508300 m abscissas, and the 9180000 and 9409000 m ordinates, of the rectangular plane UTM for *datum* WGS – 1984. It has a drainage area of 24211 km², which corresponds to 97% of BAJ and 16% of the State of Ceará, approximately. The climate of the basin, according to Köppen classification, is Bsh, characterized as a steppe's climate, with an average annual temperature of 28°C. Annual precipitation ranges between 500 and 1000 mm, with an average of 751 mm. It occurs mostly from January to May, with marked irregularity in time and space. Annual potential evaporation in the study area is in the order of 2200 mm. Geologically, it is formed mainly by Precambrian crystalline basement rocks (81% of its territory) and, on a smaller scale, by sedimentary deposits (19%).

The dominant superficial runoff is Hortonian, which generates from 10 to 70 mm annually. The typical superficial runoff coefficient ranges from 5 to 12%. The main water source of Orós Reservoir Basin is based on the reservoirs spread out in the basin. Toledo *et al.* (2014), based on satellite images, identified 4717 public and private reservoirs in the basin in 2011 (Figure 1), with a total water storage capacity of 3646 hm³. Eighteen reservoirs of this High-density Reservoir Networks (HdRN) are considered to be strategic for water resource management of the basin; they are monitored by the Companhia de Gestão dos Recursos Hídricos do Estado do Ceará (COGERH) (Water Resources Management Company of the State of Ceará).

Figure 1. Localization and spatial distribution of the 4717 identified reservoirs, in 2011, in the hydrographic basin of Orós dam (BHAO).

The hydrological model chosen for the present study was the Reservoir Network Model – ResNetM, designed by the study group *Grupo de Estudos Hidrosedimentológico do Semiárido – Hidrosed*, to simulate the hydrological processes and enable the study of hydrological connectivity in the semiarid environment, while approaching a simplified water balance for each reservoir. The model has the advantage of requiring few input data and low processing time, even when considering the thousands of reservoirs in the basin. This enables intensive studies of its parameters, e.g., sensitivity analysis (Peter *et al.*, 2014).

In the ResNetM model, the dense reservoir network is simulated as a tree, while respecting the location of each reservoir. In this tree, the reservoirs are the vertices or nodes and the connections between them are the branches, with a focus on complex network theory. The reservoirs located at the extremity show only one downstream reservoir and the other “intermediary” reservoirs are spread in the basin; there may be many upstream reservoirs, but only one downstream reservoir. The branches correspond to the draining networks of the basin, which connect the reservoirs. Their direction is determined according to the topography of the terrain, and for this reason, it does not enable the formation of cycles between vertices or reservoirs.

The model begins the interaction between the reservoirs by computing the superficial runoff generated in the sub-basin of each reservoir. Then, the resulting runoff is transferred to the reservoir, where water balance is performed. The flow generation and water balance system begins by the most upstream reservoirs of the network; the flow is transferred in the form of cascades between the sub-basins to the most-downstream reservoir, namely, the Orós Reservoir, considered to be the output vertex of the system, when there is no other downstream reservoir.

The hydrological connectivity of the reservoirs is characterized by the presence of a fluvial flow throughout the whole path of the river between the reservoir and the downstream mouth, whether it is a mouth, a river (affluence), or a new reservoir. In order to possibly acknowledge that the reach was connected, the flow rate could not be lower than a minimum reference flow rate (Speir *et al.*, 2016). A minimum reference flow rate of 1.0 L s^{-1} is adopted, according to Art. 6º, subsection III, of Resolution 707/2004 from National Water Agency - ANA. Therefore, whenever the flow rate in the upstream reservoir and in the mouth of the reach exceeded 1 L s^{-1} , hydrological connectivity of the reach was acknowledged.

Once the hydrological connectivity criteria have been established, the ResNet model can check the Hydrological Connectivity Indicator (HCI) which occurred in the basin. The HCI is estimated by the ratio between the quantity of connected reservoirs (number of hydrologically connected reaches – NHCR) and the product of the total number of reaches in the basin and the number of days of the simulation (Equation 1).

$$HCI = \frac{NHCR}{TN \times ND} \quad (1)$$

Where:

NHCR is the total number of hydrologically connected reaches (-); TN is the total number of reaches in the basin (-) and ND is the number of simulated days (days).

The input parameters considered in the sensitivity analysis of the ResNetM model were rainfall (P), potential evaporation (Ep), superficial runoff coefficient (CR), transmission loss coefficient (K), storage capacity of the reservoirs (CA) and reservoir network (Rn). The evaluated output variable was the Hydrological Connectivity Indicator (HCI).

The sensitivity of each parameter was estimated by the Sensitivity Index of Nearing *et al.* (1990) (Equation 2), which represents the normalized change generated in the output to a

normalized change in data input. In this way, the furthest from zero IS is, the more sensitive the model is for the input parameter, regardless of whether this value is positive or negative.

$$IS = \left(\frac{Out_{max} - Out_{min}}{Out_{med}} \right) / \left(\frac{Int_{max} - Int_{min}}{Int_{med}} \right) \quad (2)$$

Where:

IS is the sensitivity index of the input parameters; Int_{max} and Int_{min} are the maximum and minimum values used in the variation of the input parameter; Out_{max} and Out_{min} are the output values for Int_{max} and Int_{min} , respectively; Int_{med} and Out_{med} are the averages of input and output values, respectively.

In order to make changes to the values of input parameters, scenarios were created to describe possible alterations that can occur at a particular time in the system. Therefore, each alteration in the input parameter values of the model gave rise to a scenario (*Ceteris Paribus* principle), which was simulated with the same time period of the current system of the basin (1991 to 2011). These alterations have the following characteristics, the reference rainfall height was reduced by 5, 10, 15, 20, 25, 30, 35, 40, 45 and 50%, and also increased by 5, 10, 15 and 20%. These values were based on studies which evaluated the effects of climate change in rainfall until 2100 in Brazilian Semiarid (Krol *et al.*, 2011). The potential evaporation of the scenarios were set up to increase the Ep reference value by 1, 5, 7, 10 and 15%; these values were based on Krol *et al.* (2011). Although there was a lack of scientific evidence for a possible decrease of Ep in the region, a reduction in Ep values was simulated in the same proportions of the increase (-1, -5, -7, -10 and -15%), thus generating another 10 scenarios.

- **Runoff Coefficient (RC):** in the variation of RC, the scenarios were arranged by multiplying the specific RC reference values from the 18 sub-basins of the strategic reservoirs by the factors of 0.10; 0.25; 0.50; 0.75; 1.25; 1.50; 1.75 and 2.00, which represent the possible changes in the landscape from soil use and occupation.
- **Transmission Loss Coefficient (k):** changes in the reference value of transmission loss were made multiplying the K reference value by the factors of 0.10, 0.25, 0.50, 0.75, 1.25, 1.50, 1.75 and 2.00, in order to simulate a possible variation in water abstraction from rivers, as a result of anthropic factors and/or predicted climate changes for the region in this millennium (Krol *et al.*, 2011);
- **Reservoir storage capacity (Ca):** simulated by varying +5, +25, +50, +75, +100, -5, -25, -50 and -70%. As a result, the strategic reservoirs did not suffer variations in their storage capacity;
- **Reservoir Network (Rn):** sensitivity of hydrological connectivity to the presence of thousands of reservoirs was calculated by modifying the reservoir network topology. The reservoirs from Class 1 ($< 0.1 \text{ hm}^3$), Class 2 ($0.1 - 1 \text{ hm}^3$) and Class 3 ($1 - 3 \text{ hm}^3$) were gradually excluded from the network, and three “artificial” networks were generated, namely, Rn1, Rn12 and Rn123, respectively.

To evaluate the behavior of the scenarios and determine the effect of the dense reservoir network on hydrological connectivity, investigations were made regarding the ratio between the HCI of the scenarios and the total volume stored in the basin, as well as the volume stored in the Orós Reservoir.

3. RESULTS AND DISCUSSION

Sensitivity analysis (Sensitivity Index – SI, Table 1) of the hydrological connectivity demonstrated high sensitivity ($SI > 0.80$) to variation in the reservoir network ($SI = 1.07$) and

to runoff coefficient ($SI = 0.84$) and moderate sensitivity ($SI = 0.40$ to 0.80) to variation in storage capacity of non-strategic reservoirs ($SI = 0.45$) and rainfall alteration ($SI = 0.46$). However, hydrological connectivity in Orós Reservoir Basin has low sensitivity to potential evaporation ($SI = 0.19$) in the reservoirs and even lower to transmission loss in rivers and streams ($SI = 0.003$).

Table 1. Sensitivity Index (SI) of hydrological connectivity for the main input parameters of ResNetM model.

Input parameter	Reference input value	Interval of values		SI
		Input	ICH (%)	
Rainfall (mm)	1.88	0.94 - 1.97	4.94 - 7.25	0.46
Potential evaporation (mm month ⁻¹)	207.8	176.6 - 209.9	7.03 - 6.57	0.19
Runoff coefficient (-)	0.03	0.003 - 0.06	2.17 - 8.20	0.84
Transmission loss (km ⁻¹)	0.027	0 - 0.054	6.79 - 6.76	0.01
Reservoir storage capacity (hm ³)	0.73	0.18 - 1.46	8.69 - 5.48	0.45
Reservoir network (-)	4717	53 - 650	4.00 - 5.84	1.07

The variations of parameters k and P resulted in alteration of HCI in a “stable” rhythm, resulting in a linear tendency (Figure 2), in which a low slope refers to the axis of abscissas. The low sensitivity of hydrological connectivity to evaporation (Ep) can be explained by the period in which the highest HCI values occurred, in the rainy season of the basin. Consequently, in the rainy season, the atmospheric abstractions decrease and thereby volumetric losses in the reservoirs are small, hence not enough to significantly affect the HCI of the basin. In the case of transmission loss (k), the low sensitivity of hydrological connectivity is due to the short extension of the reaches. The thousands of reservoirs present in the basin break the draining network in short reaches (average size of 1.89 km), and consequently, water losses in these reaches become small: on average 5.1% of fluvial flow rate.

Initially, it was thought that amongst the “natural” elements, rainfall would promote the largest variations in the HCI of Orós Reservoir Basin. However, this moderate influence of rainfall over hydrological connectivity can be explained by the type of modification performed in rainfall. Rainfall variations altered the height captured directly by the reservoirs and the runoff volume in sub-basins, yet it did not influence the number of days with rainfall, and it modified the runoff coefficient to an even lesser degree. This coefficient varies according to the presence of previous rainfall, regardless of height.

It is believed that high sensitivity of hydrological connectivity to variation in runoff coefficient (CR) is linked to the process of (Hortonian) runoff generation in the region. In the process of Hortonian runoff, rainfall intensity must surpass infiltration capacity for runoff to occur. In long hillslopes, the runoff generated upstream can re-infiltrate (Medeiros et al., 2010; Safeeq and Fares, 2012). It has already been seen in other studies that the average annual rainfall below the average indicates conditions of low hydrological connectivity when compared to the average and temporarily well distributed rain years (Fryirs, 2012). Since connectivity is understood as the transfer of matter between two distinct topographical points, the identification of the connectivity patterns in a river basin allows the investigation of the cumulative impacts of small reservoirs of water resources (Lopes and Pinheiro, 2015).

In this way, soil infiltration capacity, represented inversely by the runoff coefficient (CR), becomes determinant in the process of superficial runoff re-infiltration, thus influencing directly the flow that reaches the draining network and, consequently, the hydrological connectivity of the basin. For example, when the CR parameter was increased by 50%, the possibility of superficial runoff re-infiltration decreases. The repercussions of such lower re-

infiltration was that the runoff volume which reached the draining network increased and, consequently, the HCI value was 13% higher than the reference HCI. When the CR was reduced in the same magnitude (-50%), the probability for the superficial runoff to re-infiltrate increased, thus reducing the runoff volume, and the HCI decreased by 29%, when compared with the reference value. That can be influenced by several factors; a study conducted by Rodrigues et al. (2013), observed changes in surface runoff as a consequence of the partial removal of vegetation by thinning; while in other events the average leaflet depth was higher in the preserved micro basin. Those results revealed that the practice of caatinga thinning presented modifications, presented values of drained slides inferior to those of the preserved micro basin; it was verified, however, that the maximum discharges in the thinned micro basin were superior to those of the preserved basin.

Figure 2. Values of hydrological connectivity index (HCI) obtained in BHAO, as a function of input parameters variation from ResNetM model for the period of 2001 to 2011.

In the case of variation in the reservoir network topology, the high sensitivity of hydrological connectivity confirms that anthropic intervention in the environment has been significantly interfering in the fluvial flow regime of a basin. As simulated, after removal of thousands of small- and medium-sized reservoirs of the reservoir-dense network of Orós Reservoir Basin, the HCI was significantly reduced (41% compared with the reference HCI). This indicates that the small- and medium reservoirs, even in the presence of minor fluvial floods, are capable of producing an effluent flow (sangria) enough to overcome the transmission losses (small reaches) and connect with the downstream reservoir. However, without the thousands of reservoirs, the minor floods normally are not capable of generating hydrological connectivity between the reservoirs.

The prominent roles of rainfall, reservoir network topology and superficial runoff properties over hydrological connectivity have been addressed in other hydrological studies as

well as in ecological ones (Callow and Smettem, 2009; Hall *et al.*, 2012; Long and Pavelsky, 2013). Bracken and Croke (2007) found that, in the scale of a hydrographic basin in semiarid areas, hydrological connectivity usually requires high intensity rainfall uniformly distributed throughout the basin, while slope connectivity can be initiated by short-lasting rainfall, or by less intense events. However, the authors have warned that each hydrographic basin has a spatial base pattern in terms of hydrological connectivity, which depends, primarily, on the runoff generating areas and on the rainfall-runoff relation.

In regard of the influence of the dense-reservoir network topology, Jackson and Pringle (2010) argue that the thousands of small reservoirs alter the hydrological connectivity of terrestrial landscape in innumerable ways. They hold from 81 to 98% of sediments and nutrients that are affluent to these reservoirs; they block or prevent the longitudinal movements of aquatic organisms; they transform lotic habitats into lentic habitats; and they alter the temporal series of downstream flows.

In regard of superficial runoff, Bracken and Croke (2007) point out that, in semiarid and arid environments, hydrological connectivity occurs differently compared with humid and temperate areas because Hortonian is the most common type of runoff. As a general rule, hydrological connectivity in semiarid and arid environments is harder to achieve and occurs less frequently than in temperate and humid areas. This difference in hydrological connectivity between dry and temperate/humid areas is due to many factors, for example, precedent conditions of soil humidity.

A good correlation ($R^2 = 0.74$) was found after an analysis of the relation between the volume of water stored in the basin and the HCI (Figure 3). One can notice that, when modifying the HCI, the volume stored in the basin follows the same tendency; that is, when the HCI decreased, the volume stored in the basin decreased as well; when the HCI increased, the stored volume increased. However, when the HCI is lower than 5.5%, the volume stored in the basin reduces more rapidly. This shows that the volume stored in Orós Reservoir Basin is more sensitive ($SI=1.83$) to a reduction of the reference HCI than to an increase ($SI=0.26$). The results confirmed that hydrological connectivity can be a good indicator of water availability in semiarid hydrographic basins.

An evaluation of the influence of HCI variation over the behavior of the volume stored in Orós Reservoir, showed that the influence of natural elements is different from the influence of anthropic elements (Figure 3 A and B). The reduction in HCI caused by one of the natural elements being analyzed - for example, superficial runoff (CR) or rainfall (P) - would cause a significant reduction in the volume stored in Orós reservoir ($SI=1.45$). In other words, there would be lower water availability in the reservoir.

The reduction or increase of HCI caused by changes to one of the anthropic elements being analyzed, that is, the increase or reduction in storage capacity of the reservoirs or alteration in the reservoir network topology, did not entail expressive changes in the stored volume of Orós Reservoir, yielding a sensitivity index (SI) of only 0.21. For example, by doubling the storage capacity of non-strategic reservoirs, the HCI decreased by 20% ($HCI = 5.5\%$), and the stored volume in Orós Reservoir would be reduced by only 1.3%, when compared with the reference volume (1520 hm^3), thus resulting in a sensitivity index (SI) of only 0.06.

Thereby, if the storage capacity of small and medium reservoirs spread out in Orós Reservoir Basin increased by 100%, the volume retained by them would not significantly affect the volume stored in Orós Reservoir. Or yet, if the 4664 reservoirs were removed, thus forming, the reservoir network denominated Rn123, the HCI would fall to 4% (59% less). However, the stored volume in Orós Reservoir would increase to 280 hm^3 . This increase is considered to be very small ($IS=0.09$), because the direct catchment area of Orós Reservoir would increase from 996 km^2 to 12340 km^2 after the removal of these thousands of reservoirs (Figure 4).

Figure 3. Highlighted: Relation between the Hydrological Connectivity Index (ICH) with the stored volume in the hydrographic basin of Orós dam (BHAO), according to the simulation of the scenarios in the ResNetM model, for the period of 1991 to 2011. In figures A and B: Relation between the hydrological connectivity index (ICH) and the volume stored in Orós dam, according to the variation of the natural (A) and anthropic (B) elements of major relevance for the hydrological connectivity of BHAO.

Figure 4. Relation between the direct catchment area and the average stored volume of Orós dam, obtained in the simulation of the current BHAO System and of the reservoir network variation (Ra).

The low influence of the HCI, calculated by changing the reservoir dense network topology over the stored volume in Orós Reservoir, was a surprising result. However, this is evidence that the volume of Orós Reservoir has low sensitivity to the presence or absence of the thousands of small- and medium-sized reservoirs built upstream over the years ($SI = 0.12$). Thus, one can affirm that the spatial distribution of water performed by the high-density

reservoir network (HdRN) of Orós Reservoir Basin has not been causing any significantly negative impact over the stored volume and, consequently, over the water availability of Orós Reservoir, if the superficial runoff and rainfall conditions inside the basin do not decrease.

The expansion of water-resource development has been a threat to the natural flow regime and the ecological integrity of rivers and streams in the Brazilian semi-arid region. Efforts to manage and conserve the natural flow regime of these systems is hampered by limited scientific information on processes that lead to high natural variability in the water flow regime (Maltchik and; Medeiros, 2006; Santos *et al.*, 2017). Although there is a consensus that the main impacts of reservoirs are a decrease in river discharges, peak flows due to water abstraction from reservoirs and water loss, impacts on low river flow may vary from basin to basin due to many types of reservoirs and their different uses, a factor that makes it difficult to anticipate the estimation of hydrological connectivity even using several types of indicators (Habets *et al.*, 2018). However, there is still a lack of data on the flows of these waterways, which increases the need to apply hydrological models to this estimation; studies being developed in this estimation are important to carry out this management (Maltchik and Medeiros, 2006; Santos *et al.*, 2017).

4. CONCLUSIONS

Based on the analyses performed in the present study, it can be concluded that the runoff coefficient and the reservoir network (change in the reservoir number of the network), were the parameters evaluated with the highest sensitivity of hydrological connectivity. Thus, the alteration of the landscape by man provides significant changes in river navigation between reservoirs in the basin. In the case of the topology of the reservoir network, the presence of thousands of small- and medium reservoirs is not broken, keeping the reservoir network connected even during small river floods.

The reduction of the reservoir network also provides a reduction in the hydrological connectivity of the basin; it also provides a reduction of spatial availability of water inside the basin, besides not significantly altering the inflow to the strategic reservoir of the basin, the Orós Reservoir, considered the main discharging body of the basin in this work.

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Selection of variables in the definition of a water quality index for the Brazilian Federal District

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ABSTRACT

Water Quality Indices (WQIs) are based on a small number of variables that establish categories for water quality. This method presents some problems with the evaluations, since WQIs do not consider the multiple uses of the water or differences in regional characteristics. The present study proposes a regional and more appropriate variable group for the definition of a Water Quality Index for the Brazilian Federal District, based on water quality monitoring of surface waters in the region. The monitoring was carried out monthly between August 2016 and July 2017, at three points on the Jardim River, Sobradinho Stream and Sarandi Stream, where 21 variables were evaluated. Among physical-chemical and microbiological analyses, the regional characteristics of these water bodies were noted, as well as their differences, and the main anthropogenic interferences that generate changes in water quality. In the urban area, the disposal of treated sewage from the wastewater treatment plant of Sobradinho was highlighted as the main anthropic interference, and agricultural activities stood out in the rural region of the study area. The impacts in the urbanized area were of greater weight. Thus, based on the results of the Principal Component Analysis (PCA), two groups of variables were selected, one representative of urban areas and the other of rural areas, to compose two probable water quality indices (WQIs) for the Federal District.

Keywords: Brazilian Federal District, regional variables, surface water, WQI.

Seleção de variáveis na definição de um índice de qualidade de água para o Distrito Federal

RESUMO

Os Índices de Qualidade da Água – IQAs, baseiam-se em um pequeno número de variáveis que estabelecem categorias para a qualidade da água. Esse método apresenta alguns problemas quanto às avaliações, pois os IQAs não consideram os usos múltiplos da água, bem como as diferenças nas características regionais. O presente estudo tem como objetivo propor um grupo

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de variáveis regional e mais apropriado para a definição de um Índice de Qualidade de Água para o Distrito Federal, baseado no monitoramento da qualidade de água de rios da região. Foi realizado monitoramento mensal entre agosto de 2016 a julho de 2017, em três pontos do Ribeirão Sobradinho, do Córrego Sarandi e do Rio Jardim, onde foram avaliadas 21 variáveis. Dentre análises físico-químicas e microbiológicas, foram observadas as características regionais desses corpos hídricos, bem como suas diferenças, e as principais interferências antrópicas que geram alteração na qualidade da água. Destacou-se na área urbana como principal interferência antrópica o descarte do esgoto tratado da estação de tratamento de esgoto de Sobradinho, e as atividades agropecuárias na região rural da área de estudo. Os impactos da área urbanizada foram de maior proporção. Dessa forma, baseado nos resultados da Análise de Componentes Principais – ACP, foram selecionados dois grupos de variáveis, sendo um representativo para a áreas urbanas e outro para áreas rurais, para compor dois prováveis índices de qualidade da água para o Distrito Federal.

Palavras-chave: água superficial, Distrito Federal Brasileiro, IQA, variáveis regionalizadas.

1. INTRODUCTION

Knowledge of the environmental characteristics of a given region is a basic tool for its sustainable development. In this context, studies on natural resources are included, and water appears to be one of the most important (Saad *et al.*, 2007). In conflicts over water use, there is a pressing need for the establishment of tools capable of responding with agility and confidence to questions related to water use in Brazil. The monitoring and evaluation of surface water quality are key factors for the appropriate management of water resources (ANA, 2018). Monitoring water quality should be seen as a means of verifying its potability for human consumption. Several economic activities carried out in Brazil, such as agribusiness, can generate negative socio-environmental impacts for water resources (Medeiros *et al.*, 2016).

Another important anthropic activity that generates environmental impact is the disposal of waste in water systems, as well as the use and occupation of the soil. These aspects have a direct relation with water quality, thus reaffirming the need to monitor water quality in a way that observes the characteristics of the water resource in the face of anthropic impacts (Costa and Ferreira, 2015).

The Brazilian Federal District (FD) is the Brazilian Federative Unit of the country with the third-lowest surface water availability per capita per year. In recent years, the Federal District has been experiencing strong population growth. In seven years (2010-2017), the total FD population grew 18.2%, more than double the national average (8.1%) (IBGE, 2017). Population growth in the FD does not only affect water consumption, but also leads to the inadequate use of natural resources, which implies environmental impacts. Thus, effective and rapid methods to monitor water quality are often necessary for the FD (Bilich and Lacerda, 2005).

In Brazil, the main tools related to the monitoring of water quality are the Water Quality Index (WQI) proposed by the São Paulo State Environmental Company (CETESB) (Zagatto *et al.*, 1999; ANA, 2018), and the maximum values allowed by Resolution 357/05 of Brazilian Environment Council (CONAMA, 2005). Although these tools are important, they do not consider the multiple uses of water or regional characteristics, respectively (Passos *et al.*, 2018). WQIs aim to give a unique value to the quality of the water body, so the formulation and use of indices have been strongly defended by agencies responsible for the supply and control of water pollution. Since data on water quality are collected through sampling and analysis, the need arises to translate the results in a way that is easily understood (Abbasi and Abbasi, 2012).

It is worth noting that Brazil, as a country with a continental dimension and different

regional conditions, with wide climatic and geomorphological variation, possessing seven biomes, three ecotones and 79 ecoregions (Arruda *et al.*, 2008), exemplifies how necessary it is to know the characteristics of water (Oliveira-Filho *et al.*, 2014). This is because the impacts of any activity on water bodies can only be measured if there is prior knowledge of the natural conditions of the resource.

Variable selection is the first and most important step in the composition of any WQI. In an attempt to reduce subjectivity in the selection of variables, statistical analyses such as Principal Component Analysis (PCA) have been widely adopted (Abassi and Abassi, 2012). For Bollman and Marques (2000), the use of statistical calculations in the selection of variables aims to reduce uncertainties and increase the applicability of the indices. In this context, the present work aims to select more appropriate variables with the natural configuration of the Brazilian Federal District (FD), by means of PCA, to compose a water quality index for FD water bodies. According to Lemos (2011), water quality is determined by its physical, chemical and microbiological characteristics; however, quality is a very broad term and has as its basis the intended use. As in the present study there was no specific direction for the use of water, the water in its natural condition was understood as being of standard quality. Therefore, the interruption or modification of the natural conditions was considered to represent a change in the natural quality, making it possible to characterize variables of regional importance.

2. MATERIALS AND METHODS

2.1. Study Areas and Sample Collection

The Federal District is located in central Brazil, with an area of 5,789.16 km² (Barbosa, 2010), representing the soil diversity of the Cerrado (savanna) region. It is in the Central Plateau ecoregion, with a significant geoenvironmental variety (Arruda *et al.*, 2008), reaching altitudes of up to 1,300 meters (Bilich and Lacerda, 2005). About 85% of FD soils are represented by the classes of Red Latosol, Yellow Red Latosol and Cambisol (Campos and Silva, 2000). The climate is characteristic of the Cerrado biome, with well-defined seasons: rainy summer and dry winter. In April, the dry season begins, ending around September; from October to March higher rainfall rates can be observed (Muniz *et al.*, 2011).

In the Federal District, the São Bartolomeu Basin is the one with the largest extension, covering approximately 50% of the total area, representing 2,864.05 km² (Barbosa, 2010). The sub-basins belonging to the São Bartolomeu River have significant importance in the activities of rural and urban areas. Its area consists of rural training and agricultural activities that correspond to approximately 70% of the area, and urbanization corresponds to approximately 4% of the area (Ferreira, 2006; Sena-Souza *et al.*, 2013). Although urbanization is present in a smaller part of this river basin, it presents considerable impacts, since the disorderly urban expansion from the 90's generated environmental degradation in the area (Barbosa, 2010).

In order to identify the characteristics of the surface waters of the Federal District and to qualify the variables, one river and two streams were examined: Jardim River, from the Rio Preto Basin; and Sobradinho and Sarandi Streams, from the São Bartolomeu Basin (Figure 1). The fact that there was a natural area or an area under use, whether urban or agricultural, was considered for the selection. The fact that there was both natural or areas under human use, either under urban or agricultural, it was a factor taken under consideration for sample selection.

The water bodies selected in the São Bartolomeu River Basin were Sobradinho and Sarandi Streams, an urban and rural water body, respectively. Sobradinho Stream rises in the Canastra hill and is the main water body of the Hydrographic Unit of Ribeirão Sobradinho. It presents an altimetry amplitude of approximately 350m and average annual temperature around 22°C. In relation to land use and occupation, this region is marked by intense subdivision, which has increased significantly since the 1990s (Zoby and Duarte, 2001). Sarandi Stream is a tributary

of the right bank of the Mestre d'Armas River, both belonging to the Hydrographic Unit of the Upper São Bartolomeu River, located in the north of the Federal District, with a drainage area of approximately 30 km². The use and occupation of the soil of this area is characterized by agricultural activities and land allotments (Assis *et al.*, 2013; Carvalho, 2005).

Figure 1. Location of the study area.

The Rio Preto Basin includes, besides the Federal District (12.94%), the states of Goiás (22.01%) and Minas Gerais (65.05%), so it is defined as a federal river. In the Federal District, this hydrographic basin corresponds to approximately 23% of the total area, with drainage of 1,343.75 Km² (Barbosa, 2010). Its use is mainly for agriculture, and it is responsible for about 80% of the agricultural production of the Federal District. The Rio Preto Basin is a major contributor to the São Francisco River, which is an important source of energy generation, irrigation and supply of human needs (Oliveira-Filho and Parron, 2007).

The Jardim River comprises a total drainage area of 141.8 km². It is located in the Hydrographic Unit of the Upper Jardim River, east of the Federal District, framed in the Rio Preto Basin, which is an affluent of the Paracatu River, a considerable tributary of the São Francisco River (Castro *et al.*, 2016). It is a predominantly rural area with intense agricultural activity (Vieira *et al.*, 2018), used for the cultivation of several crops, leaving few areas of its natural vegetation bordering the river (Lima, 2010).

The collection points were defined based on the representativeness of the point in the river and the safety of sampling. Previous studies in the basins also helped to define the sampling sites (Muniz *et al.*, 2011; Pires *et al.*, 2015), and the surveys carried out at the sites contributed information about the characteristics of the areas and the main polluting activities of the region (ANA, 2012). The springs were selected to observe the natural characteristics of the rivers, without direct anthropogenic interference. The other two points were aimed at assessing the impact of different human activities on the river. Samplings occurred monthly between August 2016 and July 2017, with 12 samplings from each point in each water body, totaling 108 samples, 36 from each river.

In the Sobradinho Stream, the collection points were denominated: P1, Sobradinho Spring (15°37'59" S and 47°46'21" W); P2, upstream of WWTP (wastewater treatment plant) effluent release (15°38'37" S and 47°48'44" W); P3 downstream of WWTP effluent release (15°39'59"

S and 47°48'32" W). In the Sarandi Stream, collection points were described as: P1, Sarandi Spring (15°35'43.0" S and 47°44'46.4" W); P2, Sarandi bridge (15°35'10.9" S and 47°43'58.7" W); P3, Sarandi downstream (15°35'33.9" S and 47°42'17.1" W). The collection points of the Jardim River were denominated: P1, Jardim Spring (15°43'51.2" S and 47°34'41.8" W); P2, dam upstream (15°45'54.9" S and 47°35'34.80" W); P3, Estanislau confluence upstream (15°49'44.3" S and 47°33'28.9" W).

In the water body, about 40 cm from the river bank, the properly prepared 300 ml polyethylene bottles were dipped into the water to a depth of approximately 30 cm. After collection, the vial was stored in a cool box under refrigeration and protected from light until it reached the laboratory for analysis. For determination of total coliforms and *Escherichia coli*, samples were collected in appropriate sterile containers containing sodium thiosulfate, 0.1 mg/100 mL sample. All samples were subjected to physical, chemical and microbiological analysis. Samples destined for ionic analysis by chromatography were filtered through a hydrophilic membrane, consisting of mixtures of cellulose esters with 0.45 µm porosity and frozen until the moment of analysis.

2.2. Laboratory and Statistical Analysis

For each sample, 21 variables were analyzed: 19 physicochemical and 2 microbiological variables: temperature (TEMP), dissolved oxygen (DO), pH, electrical conductivity (EC), total dissolved solids (TDS), turbidity (TURB), total hardness (TH), total dissolved phosphorus (TDP), sodium (Na⁺), potassium (K⁺), ammonium (NH₄⁺), calcium (Ca²⁺), magnesium (Mg²⁺), fluoride (F⁻), chloride (Cl⁻), nitrite (NO₂⁻), nitrate (NO₃⁻), phosphate (PO₄³⁻), sulfate (SO₄²⁻), total coliforms and *Escherichia coli* (*E. coli*).

The variables temperature and dissolved oxygen were obtained in the field with the use of a portable multiparameter meter HQ40d Hach, and the other analyses were carried out in the Embrapa Cerrados Water Chemistry Laboratory in compliance with the Brazilian Association of Technical Standardization (ABNT, 1992) and the 22nd edition of Standard Methods for the Examination of Water and Wastewater - SMEWW (APHA, 2012).

In the statistical analyses, the data matrix of Sobradinho Stream, Sarandi Stream and Jardim River were submitted to descriptive statistics (mean and standard deviation), in order to synthesize the data to have an overview of the results.

Principal Component Analysis (PCA) was used to rewrite multivariate data (Lattin *et al.*, 2011). With this analysis, it was possible to observe similar variables that can have the same meaning in terms of water quality. PCA provides a smaller view of the data that are in high dimensions, and explains as much of the data variance as possible in its first components (axes). The results of the PCAs of this study helped to select variables that contributed to the greater variability in water quality for the proposal of a water quality index for the DF. The statistical treatment of the data was carried out using software R version 3.4.2 (R Core Team, 2017). In the PCA analysis, the 'vegan' packages were used (Oksanen *et al.*, 2017) and 'FactoMineR' (Le *et al.*, 2008).

3. RESULTS AND DISCUSSION

3.1. Physical and Chemical Analyses

The data of the 19 physical-chemical variables obtained in this study show a significant difference between the water quality of Sobradinho Stream and Sarandi Stream and Jardim River (Table 1). The change in the values of P1 (spring) to P3 (downstream of the WWTP) of Sobradinho Stream occurred in more variables and in a greater proportion than in the other two rivers. Observing the averages of each physicochemical parameter of Sobradinho Stream (Table 1), it is possible to verify that the TDS increased about 90 times from P1 to P3, the hardness increased by about 70 times, ammonium showed an increase of about 50 times from the source

to the P3, the conductivity increased approximately 40-fold, the chloride had a magnification of about 25 times, total dissolved phosphorus and nitrate increased approximately 20-fold and potassium and turbidity also increased by about 15 and 10 times, respectively from P1 to P3.

The TDS is the junction of dissolved chemical compounds in water; its main sources are agricultural activities, point sources such as WWTP and industrial outflow (Parron *et al.*, 2011), and this would explain its considerable increase in P3, due to the WWTP point of Sobradinho. The same applies to the increase in hardness, which indicates contamination in the water, since it is a natural characteristic of it to present extremely low values in environments without direct anthropogenic interference (Aquino *et al.*, 2009). The main ions that provide hardness in water are calcium and magnesium, which are often interconnected with sulfate ions (Di Bernardo and Dantas, 2005).

The variables total dissolved phosphorus (TDP), potassium (K^+) and ammonium (NH_4^+) follow the same pattern, with the highest values found in P3 and the lowest values in P1. These variables extrapolated the values allowed in P3 in several months (CONAMA, 2005). According to Parron *et al.* (2011), high values of these variables may indicate contamination of raw sewage, transport of fertilizers and industrial effluents.

Chloride (Cl^-) is strongly related to the discharge of sanitary sewage, since human excreta contain a high amount of this element (WHO, 2017), which explains its increase in P3. However, even though P3 shows the highest value for this parameter, it is still lower than the maximum values allowed by CONAMA, which is 250 mg.L^{-1} . In some months, P3 Nitrate NO_3^- exceeded the values stipulated by CONAMA Resolution 357/2005, with the majority of these months in the dry period. It is believed that the reduction in water volume increased NO_3^- . The NO_3^- and Cl^- are considered relevant ions in the indication of contamination by urban effluents (Muniz *et al.*, 2011).

The study by Muniz *et al.* (2011) in Sobradinho Stream also showed considerable changes in the water quality as it approached the urban area. Urbanization is strongly connected with the decrease in water quality (Silva *et al.*, 2009), with P3 being within an urban environment. As in this study, Muniz *et al.* (2011) found the highest DO values at the spring and their lowest values at P3. CONAMA Resolution N° 357/2005 states that Class I fresh water must not have DO of less than 6 mg.L^{-1} , Class II must not be less than 5 mg.L^{-1} and Class III not less than 4 mg.L^{-1} ; that is, the minimum found in the creek is below the value determined for Class IV of the CONAMA resolution framework, in which it indicates that the DO should not be less than 2 mg.L^{-1} . This is probably due to the location of P3, which is downstream of Sobradinho's WWTP outflow, at a distance of approximately 600 meters (INPE, 2018). The bacteria that are present in large amounts in the WWTP effluent use oxygen in their respiratory processes during the stabilization of organic matter, which may contribute to the reduction of DO levels in water (Alves *et al.*, 2008). The electrical conductivity (EC) showed a considerable increase downstream of the WWTP. Because this parameter is related to the presence of ions in the water body, the more inorganic compounds are present in the solution, the greater its conductivity (APHA, 2012).

Slightly acidic values and low pH variation were also observed. The slightly acidic pH is a characteristic of Cerrado waters, which reproduce the acidity of the soils of this biome (Pires *et al.*, 2015). Sarandi Stream and Jardim River presented fewer variables with large changes between P1 and P3 (Table 1). In Sarandi, only nitrate, turbidity and TDS increased considerably, even though in much lower proportions than in Sobradinho. The nitrate increased about 6 times and the turbidity about 4 times, and the TDS almost doubled. In the Jardim River, in addition to the TDS, which also almost doubled from P1 to P3, nitrate increased by approximately 23 times, regarding the mean of P1 to the mean of P3, while fluoride increased about 5 times, and the potassium, calcium, magnesium and conductivity almost doubled.

Table 1. Descriptive statistics of the physical and chemical variables analyzed at the collection points of Sobradinho Stream, Sarandi stream and Jardim river.

Variables	Sobradinho Stream						Sarandi Stream						Jardim river					
	M			SD			M			SD			M			SD		
	P1	P2	P3	P1	P2	P3	P1	P2	P3	P1	P2	P3	P1	P2	P3	P1	P2	P3
TEMP °C	22.75	21.36	21.40	1.26	1.24	0.93	21.48	23.78	22.64	1.46	1.29	1.41	21.65	21.86	22.18	1.20	1.39	1.16
DO (mg L ⁻¹)	5.56	5.65	3.04	1.07	0.77	1.89	7.65	7.15	6.86	0.54	0.23	0.39	7.17	7.35	7.25	0.66	0.35	0.37
pH	5.23	6.45	6.80	0.54	0.25	0.48	6.15	5.96	5.56	0.45	0.59	0.47	5.94	5.95	5.98	0.49	0.49	0.50
EC (µS/cm)	4.57	62.80	170.4	1.61	22.47	77.96	3.88	5.02	5.88	0.81	0.71	1.21	3.98	7.57	7.85	1.03	1.64	4.10
TDS (mg L ⁻¹)	1.81	33.26	90.01	0.73	6.48	36.70	1.37	1.89	2.38	0.44	0.36	0.60	1.89	3.14	3.29	1.74	0.76	1.93
TH (mg L ⁻¹)	0.40	16.25	70.08	0.76	10.28	88.52	2.83	2.50	3.21	1.70	1.31	1.50	3.83	4.83	4.33	2.08	3.33	2.71
TURB (UNT)	3.80	13.44	35.59	5.57	13.74	33.70	2.63	3.63	10.11	1.94	2.29	15.53	6.12	12.55	9.67	4.68	13.26	6.42
TDP (mg L ⁻¹)	0.024	0.023	0.396	0.02	0.04	0.27	0.002	0.002	0.001	0.001	0.001	0.001	0.007	0.002	0.018	0.001	0.001	0.05
Na ⁺ (mg L ⁻¹)	0.29	0.43	0.74	0.12	0.64	0.98	0.15	0.19	0.15	0.15	0.20	0.14	0.50	0.44	0.55	0.30	0.17	0.19
K ⁺ (mg L ⁻¹)	0.17	3.44	15.92	0.26	2.90	11.53	0.17	0.33	0.22	0.09	0.23	0.20	0.32	0.26	0.58	0.32	0.17	0.82
NH ₄ ⁺ (mg L ⁻¹)	0.71	16.06	32.20	0.47	8.42	19.72	0.81	1.40	1.03	0.23	0.72	0.74	1.28	1.71	3.09	1.24	3.30	6.86
Ca ²⁺ (mg L ⁻¹)	3.12	9.55	12.74	2.15	4.47	1.95	1.46	1.56	1.78	0.23	0.21	0.39	1.67	2.40	3.00	1.46	2.08	4.21
Mg ²⁺ (mg L ⁻¹)	0.23	0.94	1.74	0.16	0.37	0.43	0.30	0.29	0.26	0.06	0.07	0.10	0.30	0.49	0.57	0.21	0.21	0.49
Fl ⁻ (mg L ⁻¹)	0.02	0.05	0.18	0.03	0.09	0.10	0.03	0.02	0.01	0.05	0.04	0.02	0.01	0.03	0.05	0.02	0.03	0.05
Cl ⁻ (mg L ⁻¹)	0.96	6.78	22.76	2.07	3.22	13.72	0.47	0.69	0.64	0.19	0.31	0.38	0.57	1.15	1.79	0.31	2.05	4.26
NO ₂ ⁻ (mg L ⁻¹)	n.d	n.d	12.81	0.01	n.d	29.68	0.03	n.d	n.d	0.09	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
NO ₃ ⁻ (mg L ⁻¹)	0.79	6.02	14.99	1.70	3.56	24.89	0.06	0.08	0.37	0.09	0.14	0.33	0.08	0.49	1.83	0.08	1.10	5.52
PO ₄ ³⁻ (mg L ⁻¹)	n.d	n.d	2.25	n.d	n.d	2.43	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	0.09	n.d	n.d	0.30
SO ₄ ²⁻ (mg L ⁻¹)	n.d	0.15	0.20	n.d	0.36	0.49	n.d	n.d	n.d	n.d	n.d	n.d	n.d	0.01	0.01	n.d	0.02	0.02

M = Mean / DP = Standard Deviation / P1 = Point 1/ P2 = Point 2/ P3 = Point 3/ TEMP = Temperature/ DO = Dissolved oxygen / EC = Electrical conductivity / TDS = Total dissolved solids / TH = Total hardness / TURB = Turbidity / TDP = Total Dissolved Phosphorus/ n.d = non detected.

In the rivers of the rural area the parameter with the greatest variation was nitrate. This increase can be explained by the agricultural activity of the region (Aquino *et al.*, 2009). Both rivers presented values lower than those found in Sobradinho Stream, and the anthropogenic interference is different. This was because in Sobradinho Stream, the wastewater treatment plant (WWTP) was observed as clearly being the main source of water quality changes, whereas in Sarandi Stream and Jardim River, which are located in rural environments, the main interference is agriculture and livestock. The widespread agricultural activity in the Jardim River region, which leads to intense use of agrochemicals, can cause water contamination (Muniz *et al.*, 2011), which is the possible explanation for the increase of nitrate at P3 of this river.

A common feature observed in the three monitored water bodies is the low values detected in their springs for the hardness, and it is natural for these waters to always have low hardness (Aquino *et al.*, 2009; Muniz *et al.*, 2011; Oliveira-Filho *et al.*, 2014); thus, it is believed that when this parameter changes, resulting in higher values, it signals the point at which anthropic interference occurs.

3.2. Total Coliforms and *E. coli*

In the results of the microbiological analyses of Sobradinho Stream, high values were observed for Total Coliforms (TC) at P2 and P3 for all samples taken. With the exception of P2 in June, all samples were detected with values $> 2,419.6$ MPN (Most Probable Number) / 100 mL. According to CONAMA Resolution No. 357/2005, total coliforms are acceptable up to the value of 5,000 MPN / 100 mL. Also according to this standard, Class III waters used in recreation of secondary contact should not exceed the limit of 2,500 MPN / 100 mL in 80% or more of at least 6 samples, and for the watering of animals raised in confinement the limit is 1,000 NMP / 100 mL in 80% or more of at least 6 samples. *E. coli* showed the lowest values at P1 and the highest values at P3, as well as TC. P1 and P2 presented acceptable values for the use of secondary-contact recreation, as well as for watering animals in confinement. In contrast, P3 exceeded all limits determined by CONAMA throughout the *E. coli* sampling for the use of the stipulated activities. The main explanation for the high values of TC and mainly of *E. coli* at P3 of Sobradinho is the release of WWTP effluents. The presence of *E. coli* in the water bodies means there is fecal contamination through these microorganisms (Silva and Bringuel, 2007).

At P1 of Sarandi Stream the TC value was the highest among the three rivers. In some months, the P2 of both Sarandi and Jardim presented values $> 2,419.6$ MPN / 100 mL, and in Jardim this value was found more frequently at P2 and Sarandi at P3. The values found in these two water bodies are within the allowed limits (CONAMA, 2005).

The *E. coli* amounts found in these water bodies are probably associated with animal feces (cattle ranching) and a small amount of untreated sewage (Aquino *et al.*, 2009; Muniz *et al.*, 2011).

CONAMA Resolution No. 357/2005 (CONAMA, 2005) was used throughout the research, with the minimum and maximum values stipulated as the basis for qualifying each parameter.

3.3. Principal Component Analysis

Before the principal component analyses (PCA) took place, Kaiser-Meyer-Olkin index (KMO) and Bartlett Sphericity Test (BST) measurements were carried out. For Hair *et al.* (2009), KMO values greater than 0.5 to 1.0 are considered acceptable for PCA application. The result of the KMO index obtained in the first study matrix was 0.79 and the BST was considered significant (<0.000 , $p < 0.05$).

Principal component analysis (PCA) was performed on the total set of normalized data (21 variables) for the 9 sampling points in order to observe the dispersion diagram of the three water bodies (Figure 2).

Figure 2. Dispersion diagram of water bodies sampled in main components 1 and 2.

It is possible to observe how the general characteristics of Sobradinho differ from Sarandi and Jardim, and how the latter two are similar. The probable explanation for this is that Sobradinho Stream is an urban water body, with a specific source of pollution provided by Sobradinho's WWTP, while the similarity between Sarandi and Jardim is possibly due to the fact that both are in rural areas, with agriculture and livestock as a source of anthropogenic contamination.

After the result of the dispersion diagram (Figure 2), showing the difference between Sobradinho and the other two rivers, a PCA was generated from Sobradinho Stream and a PCA with Sarandi Stream and Jardim River together (Table 2), in order to observe the ordered variables.

For the Sobradinho Stream data set ($KMO = 0.82$ and $BST = p < 0.000$) and the Sarandi Stream and Jardim River ($KMO = 0.77$ and $BST = p < 0.000$) the first two components (PC1 and PC2) were selected, because they presented eigenvalues > 1 , based on the Kaiser criterion, and the total percentage explained between 70 and 90% according to the criterion adopted by Jolliffe (Kaiser, 1958; Jolliffe, 2002). In studies that use PCA in water quality assessment, the first two or three major components generated account for much of the variation in the original data. In most cases these components are sufficient to describe the entire data matrices without significant loss of information (Simeonov *et al.*, 2003). A Varimax normalization was applied as a rotation method in the analysis of the main components for better interpretation of the results. For Sobradinho Stream, the two components explained 75.06% of the original data variation, while in Sarandi Stream and Jardim River the total variation explained by the three components was 80.77% (Table 2).

Loads greater than 0.75 were established to consider the variable with an important load in the main component. Liu *et al.* (2003) classified values of absolute load > 0.75 , $0.75-0.5$ and $0.5-0.3$ as strong, moderate and weak, respectively. For the Sobradinho Stream data set, the variables DO, EC, TDS, potassium, ammonium, magnesium, fluoride, chloride, phosphate and *E. coli* presented loads above 0.75 for PC1 and the sodium and sulfate variables likewise in PC2. In the data set of Sarandi Stream and Jardim River, TDP, potassium, ammonium, calcium, magnesium, chloride, nitrate and phosphate presented strong loads in PC1, while EC, TDS and sulfate had loads > 0.75 in PC2 (Table 2).

Table 2. Loadings of the main components, eigenvalues, percentage of variation and total percentage explained by the monitoring of the sampled water bodies.

Variables	Sobradinho Stream		Sarandi Stream and Jardim River	
	PC1	PC2	PC1	PC2
Temperature (TEMP)	-0.397	-0.391	-0.008	-0.000
Dissolved Oxygen (DO)	-0.832	0.021	-0.126	-0.116
pH	0.743	0.118	-0.014	0.316
Electrical Conductivity (EC)	0.918	0.047	0.248	0.873
Total Dissolved Solids (TDS)	0.965	-0.013	0.232	0.831
Total Hardness (TH)	0.756	0.069	0.228	0.528
Turbidity (TURB)	0.636	0.347	0.006	0.065
Total Dissolved Phosphorus (TDP)	0.321	0.337	0.934	-0.170
Sodium (Na ⁺)	0.093	0.869	0.042	0.165
Potassium (K ⁺)	0.881	-0.218	0.876	-0.096
Ammonium (NH ₄ ⁺)	0.852	-0.342	0.958	-0.120
Calcium (Ca ²⁺)	0.721	0.253	0.948	-0.034
Magnesium (Mg ²⁺)	0.904	0.114	0.891	-0.001
Fluoride (F ⁻)	0.813	-0.261	0.392	0.104
Chloride (Cl ⁻)	0.928	-0.245	0.961	-0.074
Nitrite (NO ₂ ⁻)	0.448	-0.248	-0.038	-0.050
Nitrate (NO ₃ ⁻)	0.456	-0.294	0.973	-0.103
Phosphate (PO ₄ ³⁻)	0.798	-0.168	0.939	-0.160
Sulfate (SO ₄ ²⁻)	-0.019	0.946	0.007	0.780
Total Coliforms (TC)	0.599	0.359	0.093	0.132
<i>Escherichia coli</i> (<i>E. coli</i>)	0.801	0.404	0.152	0.471
Eigenvalue	9.610	2.400	7.199	2.494
% of Variation	60.06	15.00	59.99	20.78
% Explained total	75.06		80.77	

3.4. Proposal of variables for a regional WQI

The differences observed in all the analyses of this work demonstrate the necessity of two separate groups of variables to constitute the FD Water Quality Index, one being an index for rivers in the urban environment and the other for rivers in the rural environment. For the proposal of these two groups of variables in the formation of WQIs for FD, the results of the PCA of Sobradinho Stream (Table 2) and PCA of Sarandi Stream and Jardim River together (Table 2) were considered, and the data of the physical and microbiological analyses (Table 1).

Electrical conductivity is a very significant variable in the analysis of water quality, since it can warn about high levels of many inorganic variables in the rivers. Besides the inorganic ions, it also shows good correlation with other variables. The conductivity showed a correlation with several inorganic ions in Sobradinho Stream, which can be interpreted as a warning for river pollution. This parameter was incorporated in the quality index of the urban and rural rivers, since its high values can signal the presence of possible contaminations and impacts undergone by rivers (Wanick *et al.*, 2011) and mainly modifications in their mineral concentration (CETESB, 2009).

The TDS were also ordered in all PCAs, but the correlation between TDS and conductivity is high, so that only the conductivity will be proposed for the WQI of FD. Chloride was also selected in all PCAs, and is part of the proposal in the group of variables for urban and rural

WQIs. Calcium was selected in the rural area, since this variable presented high loading and being very sensitive to changes in the rural environments studied.

Potassium, ammonium and magnesium variables were also ordered in the PCA of Sobradinho Stream, and of Sarandi Stream and Jardim River. These variables presented changes from P1 to P3 in the analyzed water bodies, and they were therefore suggested for the urban and rural WQIs of the FD.

Phosphate and sulfate appeared in low amounts in all water bodies in this study. The selection of them by PCA must have occurred because they presented a variation. Indeed, at many points their presence was not detected, and at some points in a few months, they were found. However, because they were detected in a few months and in low amounts throughout the monitoring, phosphate and sulfate are not proposed in the urban and rural WQIs of FD. Nitrate and total dissolved phosphorus were selected only in the PCA of the rural water bodies, and both variables are thus suggested in the rural WQI.

The variables that were selected only in the PCA of Sobradinho Stream were DO, total hardness, sodium, fluoride and *E. coli*. DO presented a negative correlation with several variables, but it was important in the evaluation of the urban water quality of FD.

The total hardness had an increase of about 70 times from P1 to P3 in this river. The water hardness, according to the results of the monitoring of this study, is low in natural waters, such as at a spring, but with the increase of anthropic intervention the values tend to get higher, making this parameter a sign of contamination in the water body. Sodium and fluoride are significant variables, since they may present increased values in water due to effluents from wastewater treatment plants (CETESB, 2009; Parron *et al.*, 2011).

E. coli is the main indicator of fecal contamination and the possible presence of pathogenic organisms (Brasil, 2017); the presence of this bacterium in high concentrations leaves water unfit for various activities (Silva and Bringuel, 2007). Although *E. coli* was not ordered in the PCA of the rural rivers, but it is an important microbiological parameter, as mentioned above, which meant that it was also incorporated in the rural WQI, mainly due to the potential presence of livestock in those areas.

Thus, among the 21 initial variables analyzed, it was proposed to select 10 variables for the definition of a WQI for urban areas, and 9 variables for a WQI for rural areas of the Brazilian FD. The variables selected for the urbanized regions and the rural regions in the FD are shown in Table 3, in comparison with the original variables from CETESB-WQI (Zagatto *et al.*, 1999).

Table 3. Comparison among variables from original WQI CETESB and proposed by the present study for urban WQI and rural WQI to be defined for FD.

WQI CETESB	Urban WQI	Rural WQI
Dissolved Oxygen	Dissolved Oxygen	Electrical Conductivity
pH	Electrical Conductivity	Total Dissolved
Turbidity	Total Hardness	Phosphorus
Biological Oxygen Demand	Sodium	Potassium
Total Nitrogen	Potassium	Ammonium
Total Phosphorus	Ammonium	Calcium
Total Residues	Magnesium	Magnesium
Temperature	Fluoride	Chloride
<i>Escherichia coli</i>	Chloride	Nitrate
	<i>Escherichia coli</i>	<i>Escherichia coli</i>

4. CONCLUSIONS

The data obtained in this study from the monitoring of the rivers and the characterization of the water quality variables evidenced the differences between the water bodies.

Sobradinho Stream is a water body that is considered urban, because it crosses the town of Sobradinho and receives effluent from the wastewater treatment plant of this town, which considerably changes the quality variables. Sarandi Stream and Jardim River are considered rural water bodies, since both run mainly through an agricultural area. The main non-point source of changes in the water quality of these surface waters is the agricultural activities present near the watercourses.

Thus, based on the regional characteristics of the FD and on quality as meaning the maintenance of the natural condition, the present study evidenced the urgent need to establish two different WQIs for FD water quality. One can be exclusively for the definition of an urban WQI, composed of: DO, electrical conductivity, total hardness, sodium, potassium, ammonium, magnesium, fluoride, chloride and *E. coli*; and the other for the construction of an eminently rural WQI, consisting of electrical conductivity, total dissolved phosphorus, potassium, ammonium, calcium, magnesium, chloride, nitrate and *E. coli*. The definition of the weights for each parameter may be objects of other studies, when there is a need to define the respective use of waters.

The use of these variables divided into two groups could minimize water quality conflicts and help with planning and framing issues aiming at better management of water resources in the Brazilian Federal District.

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Analysis of the effect of impermeability of urban soils on the infiltration of rainwater in the city of Recife, PE

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ABSTRACT

The process of urbanization interferes in the elements of the hydrological cycle, altering the infiltration, flow, and evaporation of rainwater. Several methods and tests exist for analyzing this hydrological cycle that aim to hydrodynamically characterize the soil of a locality. However, the collection and field trials can be expensive and time consuming. Because of these high costs, it is important to look for methods that save time and money. One such method is to perform simulations of water flow in the soil, using computational models such as Hydrus 1-D, in order to explain the water balance of a region. The results of these simulations showed that 355.18 mm.m⁻² of the total 385.02 mm.m⁻² of precipitation was able to infiltrate, indicating that the soil of the region has a high infiltration capacity, due to its high sand content. However, of the 228,000 m² studied, only 38,760 m² are unpaved soil. This shows that the soil at the location studied would be able to infiltrate most rainwater without the occurrence of flooding, if more than only 17% of the land area were permeable soil. This conclusion can be extrapolated to other areas surrounding this neighborhood and to other large urban centers, which have similar characteristics.

Keywords: Hydrus-1D, infiltrability, soil hydrodynamics.

Análise do efeito da impermeabilização dos solos urbanos na infiltração de água pluvial na cidade de Recife-PE

RESUMO

O processo de urbanização interfere nos elementos do ciclo hidrológico, alterando as características de infiltração, escoamento e evaporação da água oriunda da chuva. Para analisar este ciclo hidrológico há diversos métodos e ensaios que visam caracterizar hidrodinamicamente o solo de uma localidade. Entretanto, a realização de coletas e ensaios de campo são onerosos e morosos. Por conta destes altos custos, é importante buscar métodos que visem economizar tempo e dinheiro. Uma das maneiras, é realizar simulações do fluxo de água no solo, mediante o uso de modelos computacionais como o Hydrus 1-D, afim de explicar o balanço hídrico de uma região. Os resultados dessas simulações apontaram que dos 385.02 mm que precipitaram 355.18 mm infiltraram, mostrando que o solo da região possui uma grande capacidade de infiltração, o qual é composto majoritariamente por areia, entretanto, dos

228000 m² estudados apenas 38760 m² são de solo não pavimentado. Estes resultados mostram que o solo do local estudado seria capaz de infiltrar a água pluvial sem gerar alagamentos, caso não houvesse apenas 17% de solo não impermeabilizado. Além disso, esta conclusão pode ser extrapolada para as áreas que circundam este bairro e também para grandes centros urbanos, uma vez que eles possuem características semelhantes.

Palavras-chave: dinâmica da água no solo, Hydrus-1D, infiltrabilidade.

1. INTRODUCTION

Water is a natural resource of great relevance to anthropic activities in many areas. Its cycle is indispensable for the maintenance of the passive balance between urbanization and nature. The processes that make up the hydrological cycle, from infiltration to evapotranspiration to surface runoff, are dependent on the physical environment in which they are found (Tucci, 2007).

The urbanization process interferes with the elements of this hydrological cycle, modifying the environment through deforestation, degradation of natural areas, as well as other damages, such as impermeabilization of the soil, lack of an ideal urban drainage system, and pollution of the catchment and outflow network, all related to the growth of population (Corte, 2015).

Soil is a vital part of the environment. It influences the distribution of plant species and provides a habitat for a large number of organisms. It also functions as a filtration system between the atmosphere and the earth for surface water and chemicals. Soil is relevant to the destiny of society because of the goods, resources, and services that it provides to humanity (Soares, 2018).

The water inside the soil exerts forces that influence the state of tension of the soil, modifying its stability, among other pertinent edaphic attributes (Carvalho and Almeida, 2018). Moisture has a great influence on the physical behavior of the soil, especially with regard to the dynamics of water caused by external agents, such as rain. The greatest interactions occur in the topmost surface layers of the soil, where the highest water flow rates can be observed in two directions: descending (infiltration) and ascending (evaporation). Beyond these vertical movements of water, the accumulation of water on the surface can also occur when the precipitation rate is greater than the infiltration rate. When this happens, water will pool on the surface of the soil, possibly giving rise to surface runoff (Simůnek *et al.*, 2013).

With urbanization, the consequent impermeabilization of the soil is a current and recurrent problem for aquifers and groundwater sources, especially in large urban centers, because the water supply comes from water that infiltrates from the surface into the lower layers of the soil. There is great variability in the amount of time it takes for this process to occur completely, from days up to years (Cavalcanti, 2013).

According to Tucci (2004), a large urban concentration is considered to be a problem that favors flooding because of the improper use of land and the occupation of space, as well as through the insufficient management of urban drainage. Lack of planning and undue land occupation in the metropolitan area of Recife, together with high rainfall levels and variations in high tide, cause numerous floods.

There are many methods and tests that can be used to hydrodynamically characterize the soil of a locality. Because field experiments and laboratories are expensive and time-consuming, many researchers have used numerical and analytical methods that make it possible to characterize the soil with great efficiency (Martinez and Silva, 2010). Zeng *et al.* (2018) used software to simulate a variety of possible events, obtaining results that match closely with reality.

Researchers like Mujovo (2014) have used computational models, such as Hydrus-1D, to

perform hydrodynamic characterization of soils in urban areas, in order to control urban drainage by simulating infiltration trench models. This model has proven to be an excellent tool in decision making for drainage control techniques.

The use of these computational models has already become a reality in the scientific environment, because beyond modeling and predicting the objects of study accurately and precisely, they also save time and financial resources. This study estimated the water infiltration capacity of the soil using computational modeling of water flow by computational models such as Hydrus 1-D in a central and representative region of the city of Recife, the Madalena district, with data parameters of precipitation, insolation, and soil granulometry.

2. MATERIALS AND METHODS

The study area, in the Madalena neighborhood, is one of the main corridors connecting downtown Recife with the suburbs, through which can be accessed places such as the Federal University of Pernambuco and the Integrated Passenger Terminal. In addition, it contains several large shopping areas, hosts the Benfica campus of the University of Pernambuco and the Ademar da Costa Carvalho football stadium. It also contains many residential units and some government offices.

The area shown in Figure 1, having approximately 228,000 m², indicates the location studied containing the points where 27 samples were collected. In areas with natural soil cover, which make up 17% of the total study area, samples were collected for the purpose of laboratory tests. The points chosen were public squares, partially wooded and without large impermeabilized areas, as well as grassy areas with few trees making up part of the property of a local supermarket.

Much of this section of the Capibaribe River has a retaining wall separating the mangroves from the roads that border it. This wall was constructed to prevent the roads from being affected by the water during periods of high tide. Therefore, the physical properties of the superficial layers of the unpaved soil along the riverside are not influenced by the transport of fluvial sediments.

Figure 1. Area demarcated for study.

Most of the city of Recife is composed of flat relief (Silva, 2018). The delimited region varies gradually between 0 and 1 m, with a small slope of 0.0025 m.m⁻¹. Therefore, the sediments present in the unpaved soil are not leached.

Hydrus 1-D, used for modeling soil water dynamics, was constructed from a 300 cm deep layer, subdivided every 10 cm for contour conditions. The vegetation present at the collection sites was not considered in the simulations, because the crowns of the trees did not cover a large area and the root system was deep with no significant undergrowth along the ground. Urban drainage was also not considered in the simulations, because the absence of maintenance on the network present in this area has resulted in insufficiency of the drainage system.

The simulations were performed using version 4.17 of the HYDRUS-1D model (Simunek *et al.*, 2013), which numerically solves the equation of one-dimensional water flow in porous media having variable saturation (Equation 1).

$$\frac{\partial \theta}{\partial t} = \frac{\partial}{\partial z} \left(K \frac{\partial h}{\partial z} \right) \quad (1)$$

Where h is the matric potential, t is the time, z is the depth, and K is the hydraulic conductivity of the soil.

In the simulation, the hydraulic model proposed by van Genuchten (1980) was used, where the soil water retention and hydraulic conductivity curves are expressed by Equations 2 and 3:

$$\theta(h) = \theta_r + \frac{\theta_s - \theta_r}{(1 + (\alpha h)^n)^{1 - \frac{1}{n}}} \quad (2)$$

$$K(\theta) = K_s \left(\frac{\theta - \theta_r}{\theta_s - \theta_r} \right)^{\frac{1}{2}} \left[1 - \left(1 - \left(\frac{\theta - \theta_r}{\theta_s - \theta_r} \right)^{\frac{1}{1 - \frac{1}{n}}} \right)^{1 - \frac{1}{n}} \right]^2 \quad (3)$$

Where θ_r and θ_s are the residual and saturated volumetric humidity, respectively, α and n are the shape parameters, and K_s is the saturated hydraulic conductivity. The values of θ_r , θ_s , α , and n , which are required input values for the Hydrus 1D model, were obtained through the use of pedotransfer functions (PTFs), proposed by Barros *et al.* (2013), and the K_s values were estimated using the proposal by Cosby *et al.* (1984). The values of these parameters are found using Equations 4, 5, 6, 7 and 8, shown below:

$$\alpha = 10^{0.93 - 1.60G} \quad (4)$$

$$n = 1.53 + 0.33A - 0.39G \quad (5)$$

$$\theta_r = 0.13 - 0.15A + 0.36G \quad (6)$$

$$\theta_s = 0.55 - 0.23A - 0.12G \quad (7)$$

$$K_s = 7.05556 \times 10^{-6} \times 10^{(-0.6 + 0.0126A - 0.0064G)} \quad (8)$$

These take into account the contribution of each component of the soil, where A is the sand content (kg kg^{-1}) and G is the clay content (kg kg^{-1}).

The soil granulometry was obtained using the NBR 7181 method (ABNT, 2016). This standard determines that the samples be sieved to separate the fractions of sand (with a diameter greater than or equal to 0.075 mm) from the fractions of silt and clay (with diameter smaller than 0.075 mm). This finer material must then be subjected to a sedimentation process, thereby obtaining the clay and silt quantities present in each of the 27 samples collected (Density Method).

To carry out the water simulation, the atmospheric parameters of wind speed, maximum and minimum temperatures, insolation time, precipitation, and relative humidity were used as contour conditions. The atmospheric data were obtained from the National Institute of

Meteorology (INMET) database at the Curado station, where the meteorological station sensors are read and verified by an observer at predefined time intervals.

In order to estimate the daily reference evapotranspiration, the Penman-Monteith model was used, which is described by the FAO and makes use of input parameters such as precipitation, maximum and minimum temperatures, relative humidity, wind speed, and insolation time. In this case, the volume of accumulated precipitation (Figure 2A) was approximately 2264.8 mm.m^{-2} , with two major rains having values of 81.6 mm and 73.0 mm, and an average rainfall of 5.72 mm. Over the seven months analyzed, there were 63 days on which no precipitation occurred, with the period between January 1 and March 3 being the longest without any rainfall greater than 10 mm.

The maximum and minimum temperatures (Figure 2B) were at their lowest levels during the months of greatest rainfall, that is, from May to July. It can also be observed that the periods with the greatest amount of sunlight, shown in Figure 2E, are during the months of January and February, which were the least rainy and had the lowest relative humidity (Figure 2D).

Figure 2. **A** accumulated rainfall values, **B** maximum (black) and minimum (blue) temperature, **C** wind speed, **D** relative humidity and **E** daily sunshine.

3. RESULTS AND DISCUSSION

3.1. Hydrodynamic Parameters

Knowing the hydraulic conductivity or the infiltration capacity of the soil is very important because, according to the physical characteristics of each soil type, the infiltration of rainwater will be of different form and intensity, and this will affect the capacity of the soil to favor water percolation to the water table (Souza and Rodrigues, 2014).

According to Giachetti *et al.* (2000), soils with low or intermediate saturated hydraulic conductivity values may be clayey or silty, while sandy soils usually have high Ks values. The soil studied is characterized as being mostly sandy with a saturated hydraulic conductivity of $147.75 \text{ cm.day}^{-1}$, even for the areas near the banks of the Capibaribe River, which are approximately 15 m away.

Another important factor to be considered in studies such as this is the composition of the soil, since, according to Oliveira *et al.* (2013), the water infiltration potential depends directly on the sand and clay content. In addition, they also stated that the rate of infiltration of water into clayey soils is usually lower than the rate of infiltration into sandy soils.

The soil studied was composed mainly of sand (83.82%) with a small portion of clay (3.83%) and the rest being silt (12.35%). The values of $\alpha = 0.0413 \text{ cm}^{-1}$, $n = 1.9446$, $\theta_s = 0.3857 \text{ cm}^3 \text{ cm}^{-3}$, and $\theta_r = 0.0408 \text{ cm}^3 \text{ cm}^{-3}$ were obtained from the soil composition and used as necessary input values for the Hydrus 1D model simulation of soil water dynamics.

3.2. Modeling

During the period under study, from January to July 2017, the amount of water that infiltrated into the unpaved soil was approximately 355.18 mm.m^{-2} , with near constant growth at some times of the year. The soil was found to have a high infiltration capacity, corroborating with the study by Coutinho (2011). However, due to the increasing amount of impermeabilized areas inherent to urbanization, which already cover 83% of the study area, the amount of water that can infiltrate into the soil has gradually diminished.

The rates of evaporation, infiltration, and runoff for the months of January to July 2017 originated from the simulations (Figure 3). In addition, these simulations also showed that, of the 385.02 mm.m^{-2} that precipitated onto the 17% of unpaved soil during this period, 355.18 mm.m^{-2} infiltrated and the rest either drained or evaporated, completing the area's water balance and showing the effectiveness of Hydrus-1D in modeling the water balance of the studied area.

Figure 3. Water balance from January to July 2017 generated using Hydrus-1D.

By comparing the rates of evaporation, infiltration, runoff and precipitation, it can be seen that, during the drier months of January and February, the evaporation curve (yellow) accompanies the precipitation curve (black). On January 14, for example, the water evaporation levels are consistent with precipitation levels. It is possible to observe that the rate of infiltration for this day is similar to the rates of evaporation and precipitation, but it reflects the accumulation of precipitation from the previous two days (Figure 3).

During the rainiest months, June and July, the infiltration curve (red) accompanies the precipitation curve (black). On June 6, for example, it can be observed that practically everything that infiltrated was related to the rain that fell on that day, while the amount of water that evaporates is related to the amount of water that did not infiltrate during the preceding days. Thus, it is possible to infer that if the paved area were smaller than its current 83%, the locality would not suffer from constant flooding, because the soil present in the Madalena neighborhood allows for high rates of infiltration, agreeing with the study by Souza and Rodrigues (2014), which shows that soils with mostly sandy textures allow water to infiltrate more efficiently.

The month of July had the lowest evaporation rate during the period studied and it presented the highest precipitation values, more than 81 mm, as can be observed in Figure 2A. This is due to the fact that, on the rainiest days, the amount of solar radiation reaching the surface is usually lower, which reduces the amount of evaporation taking place. It also needs to be taken into account that, in the wetter months, the relative humidity is higher, increasing the vapor pressure and reducing evapotranspiration rates. During the drier months of January and February, the highest evaporation rates were observed, approximately 6 mm.day^{-1} , as well as the highest temperatures (36.8°C) and lowest rainfall rates (Figure 2B), in line with the results obtained by Ismael Filho *et al.* (2015).

Due to the heavy rainfall in the months of June and July, there is an increase in the amount of runoff because the few permeable areas saturate rapidly. In this case, in addition to rainfall, the presence of the Capibaribe River must be taken into account, which reduces the depth of the water table and consequently decreases the amount of water needed to saturate the permeable areas.

As its saturated hydraulic conductivity K_s is $147.75 \text{ cm day}^{-1}$, the soil present in the Madalena neighborhood should be able to absorb all rainwater without generating significant flooding. However, as only 17% of the soil present in the locality is permeable, the scenario ends up reversed, with numerous floods occurring throughout the year, before even considering the interaction between the amount of residual water present in the soil and its infiltrability. It can therefore be said that in months where there is greater rainfall, as well as lower evapotranspiration rates and radiation balance, the Madalena neighborhood is susceptible to constant flooding, corroborating the study by Moreira (2014). That author goes even further in his analysis, noting that even following the construction of the Tapacura dam complex to control floods, poor occupation and use of soil in the region has intensified flooding events.

However, before the process of urbanization took place in Recife (or in the Madalena neighborhood), historical records from 1632 already indicated the occurrence of floods in the region (PE-AZ, 2011). The results obtained can be extrapolated to the areas surrounding the Madalena neighborhood, which make up the central zone of Recife, making it possible to explain flooding in other districts of the city, in addition to those in Madalena.

Just as in the Madalena neighborhood, where only 17% of the soil remains unpaved, other neighborhoods in the city of Recife (and in other large cities) also have few areas favorable to the drainage of rainwater. Many important neighborhoods of the city are therefore susceptible to flooding, especially in the months when the river level is higher and evaporation rates are lower. In addition to hampering access by residents, this flooding also disrupts the flow of through-traffic, considering the important avenues that cut through neighborhoods.

4. CONCLUSION

The soil present in the unpaved areas has a high water infiltrability capacity, since the data obtained through the simulations show that 355.18 mm.m⁻² of the 385.02 mm.m⁻² total precipitation was infiltrated, or 92.25%. This was expected due to the high sand content in its composition, containing 83.82% sand, 12.35% silt, and 3.83% clay. The remaining amount of water from this total precipitation either formed surface runoff or evaporated.

Flooding is more frequent during the months of June and July, which have the highest pluviometric indexes and the lowest sunshine indexes. Like the Madalena neighborhood, other localities also suffer from the same flooding problem inherent where there are high amounts of impermeable soil. If the impermeable area were not so high, 83%, it would be difficult for the study area to suffer constant flooding, because the natural soil below the impermeable portion is sandy.

Compensatory urban drainage techniques, such as permeable pavement, infiltration trenches, rain gardens, green roofs, and retention and detention basins, have been proposed to assist the existing drainage system in minimizing flooding and in large urban centers. Some of these techniques are already being used in Recife, such as green roofs, which are mandatory and governed by Municipal Law 18.112/2015 (Recife, 2015).

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Post-treatment of landfill leachate using rice husk ash as adsorbent medium

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ABSTRACT

Sanitary landfill is the most disposal method for urban solid waste. However, in the process of organic matter degradation, a dark-colored liquid with high potential for polluting is produced, necessitating an efficient treatment before its discharge into watercourses. Several technologies can be used in the treatment, such as biological and physico-chemical systems. The activated carbon produced from agricultural residues - rice husk ash (RHA), appears as an alternative treatment, mainly for color reduction in landfill leachate. This study determined the best operational condition for the slurry treatment using a fixed-bed column. The column was filled with activated RHA and support material (sand). The column heights studied were 5 cm (C1) and 20 cm (C2) of adsorbent. The column with 20 cm of RHA and sand intercalated with the coal layer showed better efficiency in removal of the chemical oxygen demand (COD), with COD removal efficiency of 88.91% and rupture time of 120 min. The removal of apparent color was 87.40% with the rupture time of 40 min and true color removal of 86.64% with rupture time of 50 min. Activated rice husk ash was shown to be promising for post-treatment of the leachate with substantial efficiency.

Keywords: agricultural waste reuse, basic activation, fixed-bed column.

Pós tratamento de lixiviado de aterro sanitário utilizando cinza de casca de arroz como meio adsorvente

RESUMO

O aterro sanitário é o método mais geral de disposição de resíduos sólidos urbanos. Porém, no processo de degradação da matéria orgânica é produzido um líquido de coloração escura de alto potencial poluente sendo necessário tratamento adequado antes de seu lançamento nos corpos de água. Diversas tecnologias podem ser utilizadas no tratamento, como sistemas biológicos e método físico-químico. O carvão ativado produzido a partir de resíduos agrícolas, cinza de casca de arroz (CCA), surge como alternativa para o tratamento, principalmente para a redução de cor em lixiviados de aterro. Este trabalho determinou a melhor condição

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operacional para o tratamento de chorume utilizando uma coluna de leito fixo. A coluna foi preenchida com CCA ativada e material suporte (areia). As alturas de coluna estudadas foram 5 cm (C1) e 20 cm (C2) de adsorvente. A coluna com 20 cm de CCA e areia intercalada com a camada de carvão apresentou melhor eficiência na remoção de carga orgânica (DQO), com eficiência de remoção de DQO de 88,91% e tempo ruptura de 120 min. A remoção de cor aparente foi de 87,40% com tempo de ruptura de 40 min e a remoção a cor verdadeira de 86,64% com tempo de ruptura de 50 min. A cinza de casca de arroz ativada mostrou-se promissora para pós tratamento de lixiviado com eficiência.

Palavras-chave: aproveitamento de resíduos agrícolas, ativação básica, coluna de leito fixo.

1. INTRODUCTION

Urbanization exploits natural resources and generates a large amount of Municipal Solid Waste (MSW). These wastes cause much environmental damage, and highlight the need for adequate treatment (Andrade and Ferreira, 2011). Among the most common techniques of treatment and final disposal of solid wastes are reduction, compaction, milling, animal feed, composting, anaerobic digestion, incineration, launching in sewage treatment systems and deposition in dumps and landfills. Among the techniques used, the landfill is an environmentally adequate disposal method commonly employed. However, leachate is formed from the percolation of rainwater through the wastes, and it that carries pollutant compounds with high levels of ammoniacal nitrogen (NH_3 and N), biochemical oxygen demand (BOD₅), oxygen demand (COD), color (humic substances), heavy metals and suspended solids (Mojiri *et al.*, 2014).

Due to its characteristics, the release of leachate without proper treatment can lead to contamination of surface and groundwater. Slurry treatment is therefore essential. Many technologies can be used in the treatment, such as biological systems and physico-chemical methods. Among the technologies, activated carbon has been used to remove several pollutants from the effluents, and it presents high efficiency in the removal of non-biodegradable compounds from the leachate (Hosseini *et al.*, 2015; Mojiri *et al.*, 2014).

Some studies involving only the adsorption process combined with biological reactors showed good efficiency in the reduction of the recalcitrant organic matter, color and other contaminants present in the leachate (Anju and Kani, 2017; Kasman and Ibrahim, 2016; Mojiri *et al.*, 2014). However, there are few studies using rice husk ash resulting from the boiler combustion process of the rice-processing companies, as well as the use of low-cost adsorbents in the treatment of real effluents.

Anju and Kani (2017) studied the removal of turbidity, oxygen demand and suspended solids from a landfill leachate using activated rice husk ash as adsorbent; however, the authors used rice husk as a previously washed precursor for the removal of impurities to subsequent carbonization and ash activation.

In this context, this study evaluated the use of activated rice husk ash as adsorbent product in a dynamic system for the post-treatment process of leachate from the landfill of the city of Candiota/RS, which receives wastes from 19 municipalities in the Southern region.

2. MATERIALS AND METHODS

The study was carried out at the Laboratory of Analysis of Water and Effluents, located at the Center of Engineering (CEng) of the Federal University of Pelotas (UFPel).

2.1. Leachate characterization

The leachate used was sampled from the landfill of the South Half at latitude -31, 56873500 and longitude -53, 73099600 in the municipality of Candiota of the State of Rio Grande do Sul

(RS). The effluent was collected by the technician responsible for the sanitary landfill in the last anaerobic lagoon and stored in a 25-liter container and sent to the Laboratory.

The effluent used in this study was characterized according to the parameters of Chemical Demand of Oxygen, pH, Apparent and True Color at the input and output of the fixed-bed system with active carbon of rice husk ash, according to the methodology described by APHA *et al.* (2005).

2.2. Activated carbon used

The activated carbon used was from rice husk ash (RHA) from a rice-processing industry located at Pelotas/RS. The company uses the rice husk for generating thermal energy, conducting the firing in a grid-type reactor at a temperature of 800°C.

The activation of RHA was performed with sodium hydroxide as activating agent. In this process, 600 g of the raw material was separated, to which was added 600 g of sodium hydroxide dissolved in 2 liters of distilled water with a ratio of 1:1 (NaOH/ash). The mixture was maintained in contact for 24 hours, then it was washed with distilled water in order to achieve neutrality and to remove the reagents and impurities. Afterwards, the materials were dried in an oven at 110°C for 24 hours.

The concentration of the activating agent was established based on the study by Muniandy *et al.* (2014) with a difference in impregnation time. The methodology chosen for the ash-washing process was based on the work of Le Van and Luong Thi (2014), who also produced activated carbon from rice husk ash.

2.3. Characterization of pulverized activated carbon

The physical characterization was performed using the methodologies described by the Brazilian Technical Standards Association (MB 3414/1991, MB 3413/1991 and MB 3412/1991). The main characteristics of activated carbon ash used in the test were: humidity of $2.37 \pm 0.2\%$, apparent specific mass of $0.25 \pm 0.004 \text{ g.cm}^{-3}$, methylene blue of $10,05 \pm 0,70 \text{ mg.g}^{-1}$ and granulometry (% passing mass) of $38.8 \pm 2.35\%$, $80.2 \pm 1.64\%$ and $97.4 \pm 0.05\%$ in sieves of n°100, n°200 and n° 325, respectively.

The morphological and elemental composition of the precursor surface (RHA) and activated carbon was mapped with the Scanning Electron Microscopy (SEM) system coupled to an EDS system. The analyses were carried out using a scanning electron microscope (JEOL JSM 6610, Japan). The samples were metalized with gold. A 15 kV voltage acceleration was used and magnification ranges from 50 to 2500 times. The morphology and elemental composition was carried out at the Laboratory of Unitary Operations of the Chemical Engineering Course of the Federal University of Rio Grande (FURG). The EDS spectrum for raw rice husk and activated carbon are shown in Figures 1 and 2.

Figure 1. ESD spectrum for raw Rice Husk Ash (RHA).

Figure 2. EDS spectrum for chemically activated carbon with NaOH.

Figure 1A shows the formation of a material with a structure of interstices more evident, suggesting the appearance of the porous structure. From Figures 1B and 2B, the majority of silicon and oxygen can be seen, still presenting in its composition sulfur, carbon, calcium, magnesium, sodium, aluminium, phosphorus, chlorine, silicon oxide and potassium.

2.4. Adsorption Assays in Fixed-Bed Column

For the down flow adsorption tests, a column of glass was adjusted with a length of 30 cm, internal diameter of 5 cm, and the flow was maintained by a prototype with a peristaltic pump developed at the laboratory.

Two tests were carried out in the column of activated carbon of rice husk ash called C1 and C2. The C1 test presented a bed height of 5 cm and C2 presented a bed height of 20 cm, corresponding to adsorbent masses of 24 and 95 grams, respectively. The experiments were performed in triplicate for each treatment.

The system was placed the lower base of the column in wire screens, in order to retain particles which are eventually flow in the liquid and can obstruct or decrease the operating capacity of the system. A layer of sand was then placed over the screen, as a support layer for the adsorbent, whose particles were compacted with a little shaking. Subsequently, a layer of coal was introduced, which also was compacted. Finally, a layer of pebbles was added to the upper base to facilitate distribution of the flow through the adsorbent layer, thus minimizing the effects of forming preferred paths during its operation.

In the 20 cm bed height column, the sand was distributed in the base and in the core of the column, to act as a support and diffuser for liquid within the bed.

The beginning of the test occurred when the system was completely filled with the sample and the flow rate adjusted. Therefore, the first few minutes of effluent collection was discarded. Subsequently, samples were taken at intervals of 10, 20, 30, 40, 50, 60, 90, 120, 150, 180, 210 and 220 minutes, in order to evaluate the concentration at the output of the column. The analyzed parameters were COD and color. The effluent was repressed by a pump with a flow rate of 8 mL.min⁻¹. All assays were performed at room temperature.

2.5. Fixed-bed Column data analysis

The adsorption capacity of the column was determined by the following Equation 1:

$$q = \frac{C_0 Q}{m_s} \int_0^t \left(1 - \frac{C}{C_0}\right) dt \quad (1)$$

Being, q = the adsorption capacity of the adsorbent (mg g⁻¹); C_0 = initial concentration of the fluid in the input (mg L⁻¹); C = concentration of the fluid at the output of the column (mg L⁻¹);

Q = volumetric flow rate of the solution (L min^{-1}); t = time (min); m_s = dry mass of the adsorbent (g).

Removal efficiency was calculated by the following Equation 2:

$$\%R = \frac{(C_0 - C)}{C_0} * 100 \quad (2)$$

Being, $\%R$ = Removal percentage; C_0 = initial concentration of the fluid in the input (mg L^{-1}); C = concentration of the fluid at the output of the column (mg L^{-1}).

The time of the *Breakthrough* was obtained by the curve inclination of the adsorbate concentration at the output of the bed, which consists of the ratio of C/C_0 against the time. The break point was considered from $(C/C_0) \geq 0.3$ for C1 and $(C/C_0) \geq 0.1$ for C2 and saturation point $(C/C_0) \geq 0.8$ for C1 and C2. In practice, the industries work with a useful column volume, which means that the rupture and saturation time can be decided according to need or the output concentration can be equivalent to a reference value established by current legislation (Karunarathne and Amarasinghe, 2013). The performance of RHA activated at different bed heights was analyzed in the Statistica® program, Version 7.0. The parameters were adsorption capacity for the rupture and saturation times for the analysis of chemical oxygen demand; real and apparent color were performed with a completely randomized design and the results were treated by Analysis of Variance (ANOVA) and, when significant, the Fischer test was performed ($p < 0.05$).

3. RESULTS AND DISCUSSION

The results were obtained by the curves considering the height of the bed of 5 cm (C1) and 20 cm (C2) and initial organic matter concentration of $4538.61 \pm 190.82 \text{ mg.L}^{-1}$ COD, pH 8.14, apparent color of $176000 \pm 1885.62 \text{ UC}$ and true color of $16600 \pm 942.81 \text{ UC}$ (Figures 3 and 4). The parameters t_r and t_s were calculated (Table 1), along with the adsorption capacity, residual concentration of COD and color. The t_r refers to the time of rupture or time required for the adsorbate to be detected at the exit of the bed and t_s refers to the time required for bed exhaustion.

Figure 3. Rupture curves for C1 (bed height of 5 cm) and C2 (bed height of 20 cm) treatments for the removal of organic matter (COD).

Figure 4. Rupture curves of apparent color and true color removal for C1 and C2.

Table 1. *Characteristics of the C1 and C2 tests for COD and apparent color removal.

Parameters	COD		Apparent color		True color	
	C1	C2	C1	C2	C1	C2
Bed Height (cm)	5	20	5	20	5	20
Flow (mL min ⁻¹)	8	8	8	8	8	8
t _r (min)	10	120	20	40	30	50
q _r (mg g ⁻¹)	10,23 ^a	40,77 ^{ab}	79,77 ^{acd}	54,81 ^{bc}	110,5 ^d	60,56 ^{bc}
**Parameters residual _r	1467,89	503,145	5633,3	2216,67	5550	2216,7
t _s (min)	40	220	210	220***	210	220***
q _s (mg g ⁻¹)	13,55 ^a	11,57 ^a	289,33 ^g	220,15 ^{ef***}	242,67 ^{cf}	207,80 ^{e***}
Parameters residual _s	3522,013	3914,37	13466,67	5716,67*	13133,33	5383,33***

*Means followed by the same letter are not significantly different at the 95% confidence level (Fischer's test). **Parametres residual: Cod (mg.g⁻¹); Color (UC). ***values obtained for the total operating time.

Figures 3 and 4 show that the increase in height of bed positively affected the efficiency of removal of organic load and leachate color, since the career time of the rupture of the C2 treatment was greater than that of the C1 (Table 1). The C2 treatment had shown a removal efficiency up to 86.64% of the true color, and 87.4% of the apparent color and 88.91% of the COD. On the other hand, the treatment C1 showed a efficiency for removal of 66.56% of the true color, 67.65% of the COD and 67.99% of the apparent color. These values are compatible with other study developed by Kawahigashi *et al.* (2014), in which authors verified the effectiveness of adsorption with commercial granular carbon of coconut endocarp as post treatment of landfill leachate, removing around 45 to 76% of COD, 95 to 100% of apparent color and 94 to 100 of the true color under conditions of pH = 4.0, Fe³⁺ dose (mg Fe³⁺.L⁻¹), initial COD of 303 mg.L⁻¹ and height of bed of 9 cm.

Vieira (2014), in a similar study, found that with the increase of the height of the bed from 10 cm to 30 cm, an improvement in performance was shown, increasing the rupture time and the percentage of removal at this condition. The best condition found in this study was 30 cm of the bed height, yellow tartrazine dye concentration of 50 mg L⁻¹, rupture time of 88 min,

maximum column capacity of 108.0 mg g⁻¹ and the removal percentage of 85.9%, using the chitosan as adsorbent.

The behavior shown in our study corroborates with that found by Vieira (2014), with the variation of the bed height a factor with a great influence in the phenomenon of adsorption. In addition, the height of the bed, particle size, pore size, molecular weight and pollutant concentration are other parameters that altered the characteristics of the adsorption process.

Regarding the particle size, generally, materials with lower granulometry have a higher capacity for pollutant removal, i.e., adsorbent with particle size 0.297-0.841 mm results in 2 to 3 times greater capacity for removal than adsorbents with a particle size of 0.420-1.680 mm (Tauma, 2013). Thus, it can be affirmed that the particle size of the activated charcoal of rice husk ash showed a great capacity of removal influence on the organic matter and color of the landfill leachate.

Methylene blue index is a technique used to indirectly measure the pore size of the adsorbents; the materials with high methylene blue index indicate that the material analyzed is mesoporous (2 to 50 nm) (Kawahigashi *et al.*, 2014). In this perspective, the activated carbon of RHA presents elusive indication of material with mesopores, according to the value of index of methylene blue found of 10.05 mg.g⁻¹. The presence of pores of the coal used in the study can be observed in the SEM image presented in Figure 2.

According to Castilhos Junior *et al.* (2010), one of the main constituents of the recalcitrant organic matter of the leachate are the humic substances and fulvic acids that are high molecular weight substances. These substances are responsible for the coloration of leachates (Queiroz *et al.*, 2011). Thus, the high efficiency of the systems can be attributed to the large molecular weight of humic substances and fulvic acids that can easily be removed from the aqueous solution by adsorption through two effects: solubility and surface attraction. Also, the principle of destabilization due to particle collisions can be highlighted. This phenomenon occurs due to the high concentration of the contaminant in solution (Tauma, 2013), as shown in the characterization of the leachate with high staining, expressed as apparent and true color.

4. CONCLUSION

In summary, the study was satisfactory for the dynamic process of adsorption in a fixed bed. The study showed that the use of activated rice husk ash is a promising method for post-treatment of stabilized landfill leachate with regard to COD removal, apparent and true color. The experiment showed removal efficiency higher than 60% for the analyzed parameters.

The 20 cm of height of bed was the system that provided better performance and longer operation time in the leachate treatment. Further studies changing other parameters are highly recommended.

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Constructed wetlands as post treatment of a decanter digester followed by an anaerobic filter

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ABSTRACT

The amount of wastewater has increased due to the considerable expansion of higher education institutions. When wastewater is released without treatment or with inefficient treatment, it causes significant water pollution. In this context, this study evaluated the performance of Constructed Wetlands as a post-treatment of a decanter digester followed by an anaerobic filter, operated with effluent from the Federal University of Mato Grosso, Cuiabá Campus, Mato Grosso State. Four wetlands were constructed to develop the research: planted with *Typha domingensis*, planted with *Heliconia psittacorum*, planted with *Pontederia parviflora*, and not planted. The following physicochemical parameters were analyzed: Color, Turbidity, pH, Temperature, Total Solids, Total Dissolved Solids (TDS), Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Nitrogen, Sulfate, Nitrate and Nitrite. The results show an organic matter removal efficiency with mean removal results: 70% BOD₅ for TCW, HCW and PCW; and COD 76% for PCW. The mean removal of total phosphorus was 78% for PCW and 92% NO⁻², as macronutrients. Analysis of variance ANOVA shows significance in the results of TDS, SO₄ and COD removal.

Keywords: biotechnology, horizontal subsurface flow, macrophytes, wastewater.

Wetlands construídos como pós-tratamento de um decanto-digestor seguido de um filtro anaeróbio

RESUMO

Com a crescente expansão das Instituições de Ensino Superior (IES), aumentam-se a quantidade de águas residuais, quando a mesma é lançada sem tratamento ou com tratamento ineficiente contribuem de maneira significativa para poluição dos recursos hídricos. Neste contexto, o objetivo deste trabalho foi avaliar o desempenho de Wetlands Construídos (WC) como pós-tratamento de decanto-digestor seguido de filtro anaeróbio, operados com efluente da Universidade Federal de Mato Grosso Campus Cuiabá-MT, no que se refere à remoção de nutrientes e matérias orgânicas. Para o desenvolvimento da pesquisa foram construídos quatro wetlands, sendo eles: WCT - plantado com *Typha domingensis*, WCH - plantado com *Heliconia psittacorum*, WCP plantado com *Pontederia Parviflora*, e WCB - não plantado. Foram

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analisados os parâmetros físico-químicos de cor, turbidez, pH, temperatura, Sólidos Totais (ST), Sólidos Totais Dissolvidos (STD), Demanda Bioquímica de Oxigênio (DBO₅), Demanda Química de Oxigênio (DQO), nitrogênio total, fósforo, sulfato, nitrato e nitrito. Os resultados obtidos mostram uma eficiência de remoção de matéria orgânica com resultados médios de remoção: de DBO₅ 70% para WCT, WCH e WCP; e de DQO 76% para WCP. A remoção média de fósforo total 78% para WCP e NO⁻² 92%, como macronutrientes. A análise de variância ANOVA mostra significância nos resultados de remoção de STD, SO₄ e DQO.

Palavras-chave: águas residuais, fitorremediação, fluxo horizontal subsuperficial, macrófitas.

1. INTRODUCTION

Wastewater from universities (WU) contains pollutant loads that, if discarded without treatment, can degrade water resources. This occurs when the pollutants are in contact with different processes of water use at universities, leading to water contamination, and thus changing chemical, physical and biological patterns (Santín *et al.*, 2016; Freitas *et al.*, 2016).

Wastewater treatment plants (WTPs) are responsible for restoring water quality standards to return it as naturally as possible to watercourses (Meneses *et al.*, 2015). In order to reduce impacts caused to the environment by sanitary sewage at universities, the water goes through wastewater treatment procedures in WTPs that can efficiently remove the dissolved particles, suspended and organic loads that are added to the wastewater (Morrison *et al.*, 2016; Wagner and Da Costa, 2015).

The Federal University of Mato Grosso is located in the Barbado Stream Basin in the city of Cuiabá, Mato Grosso state, a tributary of the Cuiabá River, and has a decanter digester as wastewater treatment, which comprises wastewater storage tanks. These tanks have active sludge inside with bacterial colonies capable of degrading organic matter through anaerobic biological processes such as hydrolysis, acidogenesis, acetogenesis, methanogenesis and sulfidogenesis. This wastewater treatment system, although simple and cheap, is inefficient in terms of organic matter removal (Chernicharo, 2019).

The aim of a Constructed Wetland (CW) is to treat both raw and post-treatment wastewater, thus ensuring greater efficiency in the removal of organic fats and macronutrients. A CW can operate in two ways: with the flow free flow where the macrophytes are floating or emerging (Toscano *et al.*, 2015); or with subsurface flow (Rodríguez and Brisson, 2015).

CWs are commonly used in countries such as Chile, Spain and Canada (Vera *et al.*, 2013, Casas-Ledón *et al.*, 2017, Caselles-Osorio *et al.*, 2017), mainly in post-treatment, because they have plants that have the capacity to absorb macronutrients (N and P), which are released in the process of decomposing matter, as well as in the nitrification process that occurs in bacteria related to the roots of plants (according to Li *et al.*, 2015).

This study evaluated the efficiency of a CW as post-treatment of a decanter digester followed by an anaerobic filter on an experimental scale.

2. MATERIAL AND METHODS

The methodology was developed in three stages: I) designing and constructing the experiment in a pilot scale of the CW; II) monitoring the physico-chemical parameters of the WTP; and III) monitoring the physicochemical parameters and CW biomass growth.

The experiment was carried out at the WTP at the Federal University of Mato Grosso, Cuiabá Campus, located between the geographic coordinates 21S 600022.00m E and 8274119.00m S. The climate in the city of Cuiabá is characterized as continental tropical, with two distinct seasonal seasons, one rainy (October-March) and the other dry (April-September). It has an annual precipitation of 1.262 mm and an average temperature of 25, 91°C (INMET, 2018).

The wastewater treatment system is formed by a lift station that receives the WU, from the sanitary station of the university, and from the *Jardim América* neighborhood. It is then pumped to the WTP, which is comprised of a Decanter Digester, where the degradation of organic matter occurs anaerobically. This is followed by a biofilter, which retains part of the dissolved solids of the system, and then finally the Drying Bed (DB).

The treated effluent from the WTP was moved by a peristaltic pump to a reservoir of 1 m³ made of polyethylene, then by gravity to CWs, which were fed continuously in a subsurface form with controlled flows in each CW, at a flow rate of 50 liters / 10 days. The internal volume of the tanks used in the CW implantation was 0.45 m³, made of high-density polyethylene, with a surface length of 2.20 m and a depth of 1.84 m, a surface width of 0.71 m, and a bottom measuring 0.33 m, respectively. The substrate filling consisted of a sequence of filter layers: hand stone, washed sand and gravel N°. 02.

Four wetlands were constructed, with three different species of macrophytes in separate planted CWs and one unplanted CW; those that were planted had a density of 10 rhizomes per CW, divided into the following: one was planted with *Typha domingensis* (TCW), one planted with *Heliconia psittacorum* (HCW), one planted with *Pontederia Parviflora* (PCW), and one was not planted (NCW) in order to obtain the block of the treatment tanks (Figure 1).

1 Reservoir; 2 CW planted with *Typha*; 3 CW planted with *Heliconia*; 4 CW with *Pontederia*; and 5 CW not planted.
A) stone hand; B) sand; C) gravel N° 2

Figure 1. Constructed Wetland.

Sampling was carried out from October 2017 to February 2018, and six points were monitored at the WTP: Gross Effluent - Point 1 (P1) - Landfill and Treated Effluent - Point 2 (P2) - Biofilter Output, considering the flow of the system structure. The other four samples at the output of each CW, occurring over a period of 150 days, complied with the Hydraulic Detention Time (HDT) of 5 and 10 days. The samples were collected using collector tubes installed in each CW tank at the following sampling points: TCW, HCW, PCW and NCW.

Samples were analyzed considering the following parameters: Total Dissolved Solids - TDS (mg/l), Total Phosphorus - TP (mg/l), Sulphate - SO₄ (mg/l), Nitrate - NO₃⁻ (mg/l), Nitrite - NO₂⁻ (mg/l), Total Nitrogen - TN (mg/l), pH, Biochemical Oxygen Demand - BOD₅ (mg/l) and Chemical Oxygen Demand - COD (mg/l).

After collecting the concentration results of the TDS, TP, SO₄, NO₃⁻, NT, NO₂⁻, BOD₅ and COD parameters, they were submitted to Equation 1 to each CW to obtain the Removal Efficiency.

$$RE = \frac{\text{Conc}(P2) - \text{Conc}(CW)}{\text{Conc}(P2)} \times 100 \quad (1)$$

At where:

$\text{Conc}(CW) \neq 0$

2.1. Biomass

Every 10 days, four plants from each treatment were collected and separated into roots, leaves and stems. Afterwards, the roots were washed in running water and distilled to remove soil. The vegetal material was placed individually in paper bags, properly identified and then dried in a forced ventilation oven at 70°C for 48 hours. Finally, the material was weighed to calculate the dry mass of the leaves, roots and stems of each plant.

Statistical analyses of the results from the laboratory analyses were performed by the paired ANOVA test that evaluated the variance of the results, as well as the standard deviation between them. Significant differences in water quality between wet and dry plant species were evaluated using a completely randomized analysis of variance with significance of 5% and 10%. To prove the variance test, the correlation test was used to determine the relationship between the parameters.

3. RESULTS AND DISCUSSION

Dissolved Oxygen inside the WTP and CW varied between 0.91 and 7.24; the WTP output was lower than the standards established by CONAMA (Brazilian National Council for the Environment) due to the anaerobic system where the pH rises and DO decreases (Figure 2). The CWs had a higher oxygen insertion at the beginning of the experiment, decreasing after the month of January. This variation is related to plant respiration and biofilm development using DO in the decomposition of organic matter (Pelissari *et al.*, 2014).

Figure 2. Dissolved Oxygen Variation.

The results of pH and CW in the WTP varied between 8.95 and 6.81, within the range established by CONAMA (Figure 3), and the most alkaline result was recorded in the HCW in the HDT on October 10th, and the most acidic result was recorded in the TCW in the HDT also on October 10th. These values correspond to those of Caselles-Osorio (2017), who states that plant root growth can influence plant pH by acidifying the medium with carbon production due to respiration (Kadlec *et al.*, 1996; Vymazal, 2007).

Figure 3. pH variation.

The CW removals were calculated in relation to P1 following the research sample; therefore, the average removal efficiency of the TDS in the HDT of 5 days was highlighted by the PCW, with 51%. The others had close values, which were 48%, 50% and 46% for TCW, HCW and NCW, respectively. In the 10-day results, the efficiencies of all CWs rose. The PCW

had the best result, with a value of 73%, while the other TCW, HCW, NCW obtained 71%, 72% and 61%, respectively. These values show that there was increased efficiency in the TDS, similar to those of Sehar *et al.*, who obtained efficiency values between 42% and 74%, in their experiment at a 20-day TDH. In the present study, it was observed that the removal of TS is related to sorption in the filter medium, absorption by the plants (Figure 4) (Sezerino *et al.*, 2015).

Figure 4. Total Dissolved Solids efficiency.

Phosphorus in the CW had a great variation mainly in the months of November 2017 and February 2017, but the CW behaved efficiently in both concentrations. The average phosphorus efficiencies in the HDT of 10 days were 49%, 45%, 78% and 41%, for TCW, HCW, PCWP and NCW, respectively.

The P is part of the macronutrients that the plants absorb to use in their metabolic compositions, comparing the phosphorus concentrations with Abou-Elela and Hellal (2012) and Leto *et al.* (2013) who obtained a result of 62% removal and 47.9% with a concentration of 0.4 mg/l and 4.2 mg/l. The PCW obtained better removal results, while TCW, HCW and NCW obtained results of efficiency lower than both (Figure 5).

Figure 5. Phosphorus efficiency.

The mean sulphate efficiencies in 5 days of HDT were 60%, 59%, 65% and 35% for TCW, HCW, PCW and NCW, respectively; and in 10 days of HDT were 95%, 99%, 95% and 93% for TCW, HCW, PCW and NCW, respectively. These results show that in the planted CWs, there was higher sulphate removal efficiency compared to the non-planted ones. This may be related to oxygen in the available plant roots (Figure 6).

Figure 6. Sulphate efficiency.

The mean efficiency of NO^{-2} in the HDT of 5 days was: 68%, 57%, 61% and 66% for TCW, HCW, PCW and NCW, respectively; for 10 days, it was 92%, 88%, 89% and 78%, for TCW, HCW, PCW and NCW, respectively. The mean NO^{-3} removal in the HDT of 5 days was 66%, 66%, 67% and 57% for TCW, HCW, PCW and NCW, respectively; for 10 days it was 86%, 75%, 82% and 67% for TCW, HCW, PCW and NCW, respectively. The removal of NO^{-3} and NO^{-2} occurs due to the absorption of nutrients by plants and the release of oxygen forming N_2 (Sehar *et al.*, 2014). Maine *et al.* (2006) obtained results of 88% and 85% concerning the removal of NO^{-3} and NO^{-2} , in WC planted with *Typha domingensis* and *Panicum elephantipes* (Figure 7).

Figure 7. Efficiency of Nitrite and Nitrate.

The mean total nitrogen efficiency results in the HDT of 10 days were 52%, 42%, 34% and 31%, for TCW, HCW, PCW and NCW, respectively. The maximum removal in the PCW

was the one that stood out, as it had 60% in October 2017. Hsu et al. (2011) obtained a result of Total Nitrogen removal in a natural wetland which is close to the results of the present study. However, in an experiment similar to this one, Rodríguez and Brisson (2015) managed a removal of 97% (Figure 8).

Figure 8. Total Nitrogen Removal.

The BOD₅ obtained the mean efficiency for the HDT of 5 days, which was less than 50%. The CW that had the best mean efficiency was HCW with 45%, then PCW with 44%, TCW with 42% and NCW with 38%. However, the results of the 10-day analysis were the same for all planted CWs at 70%, and the NCW was 66%. The 5-day and 10-day HDT efficiency values, despite being effective in maintaining lower load effluent within the limits of CONAMA 357/05, these same results in the HDT of 5 days are lower than those of Sohslam *et al.* (2008); Vera *et al.* (2013) and Abou-Elala and Hellal (2012); and the efficiency result in the HDT of 10 days are equal to these authors. BOD₅ removal occurs due to deposition and filtration, while organic compounds were degraded aerobically and anaerobically by heterotrophic microorganisms, depending on the concentration of oxygen in the bed (Figure 9).

Figure 9. BOD₅ efficiency.

The mean COD removals for TCW, HCW, PCW and NCW were 42%, 34%, 47% and 29%, respectively, in the HDT of 5 days. In the HDT of 10 days, the values were close, ranging from 76% to 69%. The COD values were similar to those of Ansola *et al.* (2003), Caselles-Osorio *et al.* (2011) and Ebrahimi *et al.* (2013), who had mean values of efficiency of 79%, 75% and

72%. These studies found a greater efficiency in planted CWs due to the assimilation of the plant to organic materials and macro nutrients found in the wastewater (Figure 10).

Figure 10. COD efficiency.

3.1. Development of macrophytes

The macrophytes were planted in their young stage and accompanied by their growth in height (cm) and weight (g). The result shows the growth variation between plants from the young to the adult stages. Among the macrophytes, *Typha domingensis* and *Pontederia parviflora* evolved in the analyzed period; however, *Heliconia psittacorum* had a low adaptation to the wetland system implanted (Figure 11).

Figure 11. Total Nitrogen Removal.

The average height of the *Typha domingensis* plant in October 2017 was 35.2 cm, and in February 2018 it had an average size of 111.2 cm. In addition, *Heliconia psittacorum* obtained an average height in October 2017 of 3.36 cm, and in February 2018 it was 35.2 cm. The *Pontederia parviflora* species reached 23.55 cm at the beginning of the period, and at the end 35.6 cm (Figure 12).

Figure 12. Macrophyte growth.

The mean growth of the leaf of the *Typha domingensis* species was initially 43.05 cm, reaching 72.02 cm; compared to Lorenzen *et al.* (2001), *Typha domingensis* had similar growth, and in its experiment the final size was 97 cm. On the other hand, the species *Heliconia psittacorum* had an average increase of 6.74 cm, and at the end of the studied period reached 18.40 cm, compared to Cerqueira *et al.* (2008). These species showed considerable development, since species presented average growth of 97.1 cm. The average leaf length of the *Pontederia parviflora* started at 5.70 and ended at 9.45 cm. The biomass growth was measured in the *Typha domingensis* and *Pontederia parviflora* species, comparing the beginning and end period of 50 days. In the first plant there was an individual increase in its wet weight of 92.53 g and in the dry weight of 24.60 g, compared to the second. Regarding the *Pontederia parviflora* species, the individual wet weight was 76.36 g and the dry weight was 18.15 g. In this plant biomass analysis, we were not able to determine the weight of the *Heliconia psittacorum*.

3.2. Statistical Analysis

The static ANOVA test was performed for 5 and 10 days of HDT, with a significance of 5% for all parameters. The COD, SO₄ and TDS, both for 10 days of HDT, were significant at 10%, according to ANOVA.

4. CONCLUSION

To sum up the experiment, it was concluded that the wetlands present a greater concentration removal in the CWs (TCW, HCW and PCW) than in the non-planted ones. Among those planted, PCW was the one that most stood out in the removal of organic matter and macronutrients. It was the CW that obtained the highest result in 7 of the analyzed parameters in the HDTs of 5 days and 10 days.

Plant growth was fast, as the *Typha domingensis* and *Pontederia parviflora* species flowered at the end of five months, and also multiplied in individuals. However, *Heliconia psittacorum* showed a slower evolution with little productive adaptation.

The parameters such as COD, SO₄ and TDS had a significance of 5% in the ANOVA test, proving that HDT in the CW can be efficient in the removal of these pollutants even with the variation of the input load.

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