

# Dissolved organic carbon and nitrate flows in two micro-watersheds with different land uses

## Fluxos de carbono orgânico dissolvido e de nitrato em duas microbacias sob diferentes usos

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### Highlights

Cerrado micro-watershed shows lower water retention and higher peak flow.  
Land use modifies the dissolved organic carbon (DOC) and nitrate (NO<sub>3</sub>) flows.  
Increments of DOC and NO<sub>3</sub> on rainy days are higher in the agricultural watershed.

### Abstract

The impacts of human activities on watersheds can change the quality and possible uses of water resources. In this context, we evaluated the flows of dissolved organic carbon (DOC) and nitrate (NO<sub>3</sub>) in the surface waters of two micro-watersheds with different anthropic impacts. Water samples were collected from two micro-watersheds with different land uses (regenerated savanna and an agricultural site) from January 2014 to April 2015. In the rainy season, the samples were collected every 15 days, while in the dry season, samples were collected every 30 days. An automatic sampler in the stream collected the composed samples. Water flow was monitored with a sensor that measured the hydraulic load on the sill of the triangular spillway installed in each micro-watershed. The DOC and NO<sub>3</sub> contents were analyzed via UV-Vis spectrophotometry. The flow was estimated based on the DOC and NO<sub>3</sub> flows and concentrations. The DOC concentrations were higher in the Cerrado micro-watershed; however, there was no difference in NO<sub>3</sub> concentrations. In both watersheds, there was a significant increase in the concentrations of DOC and NO<sub>3</sub> on rainy days, with more pronounced in the agricultural watershed. The DOC and NO<sub>3</sub> flows were higher in the micro watershed with Cerrado vegetation on days with rain; while on days without rain, there was no difference.

**Key words:** Land use. Hydrology. Hydrological monitoring. Nutrient flow.

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## Resumo

Os impactos das atividades humanas realizadas sobre microbacias hidrográficas podem alterar a qualidade e os possíveis usos do recurso hídrico. Neste contexto, o objetivo deste estudo foi avaliar os fluxos de carbono orgânico dissolvido (COD) e nitrato ( $\text{NO}_3$ ) nas águas superficiais de duas microbacias hidrográficas com diferentes impactos antrópicos. Para tal, foram realizadas coletas de amostras de água em duas microbacias com uso distintos do solo, Cerrado regenerado e uma área agrícola, no período de janeiro/2014 a abril/2015. As amostras de água foram coletadas quinzenalmente no período chuvoso e mensalmente no período seco. As amostras compostas foram coletadas com amostrador automático instalado nos cursos d'água. O monitoramento da vazão foi realizado através de um sensor de nível que registrou a carga hidráulica vertente na soleira do vertedouro triangular instalado em cada uma das microbacias. Os teores de COD e de  $\text{NO}_3$  foram analisados em um espectrofotômetro UV-Vis. O fluxo foi estimado com base nos valores de vazão e das concentrações de COD e  $\text{NO}_3$ . As concentrações de COD são maiores na microbacia Cerrado, porém não houve diferença nas concentrações de  $\text{NO}_3$ . Em ambas as microbacias há incremento significativo da concentração de COD e de  $\text{NO}_3$  nos dias com chuva, sendo este incremento maior na microbacia agrícola. Os fluxos de COD e de  $\text{NO}_3$  são maiores na microbacia com vegetação de Cerrado na presença de chuva, porém não houve diferença na ausência de chuva.

**Palavras-chave:** Uso e ocupação do solo. Hidrologia. Monitoramento hidrológico. Fluxo de nutrientes.

## Introduction

Studies on water quality changes in micro-watersheds are crucial to understand the impacts of different land uses on the aquatic environment. In the last decades, the Cerrado biome has been extensively modified by humans, with considerable impacts on its hydrological dynamics.

The increasing use of chemical fertilizers in this region has largely compromised water quality, with excessive nutrients reaching aquatic systems near cropping areas.

Of these, nitrate is considered a major pollutant, affecting water quality and (Galloway et al., 2008).

When nitrogen is applied to soil in the form of ammonia ( $\text{NH}_3$ ), it is rapidly transformed into ammonium ( $\text{NH}_4^+$ ) and, when not used by crops or microorganisms, is subsequently. Nitrate form weak chemical bonds with soil

colloids, and when not absorbed by plants, it is lost via soluviation or denitrification processes (Kaiser, Reinert, Reichert, Streck, & Pellegrini, 2010).

Apart from nitrate, dissolved organic carbon (DOC) is another important parameter to evaluate the effect of anthropization on water resources. Generally, DOC in the watercourse stems from biological or physical-chemical processes, such as dissolution and soluviation of particulate organic matter or the decomposition of animals and plants (Creed, Webster, & Bourbonniere, 2013). It can interfere with the acidity of aquatic ecosystems (Eshleman & Hemond, 1985) and increase metal transport, resulting in the formation of organic complexes which can enhance the toxicity of metals in aquatic biota. Beyond that, the DOC represents almost 50% of the carbon flow exported to the oceans and is one important compound of the carbon global cycle, making it closely connected with climatic

and environmental changes (Smith, Kaushal, Beaulieu, Pennino, & Welty, 2017).

To understand the dynamics of DOC and  $\text{NO}_3$  in micro-watersheds, monitoring is extremely important. In this context, we evaluated the flows of DOC and  $\text{NO}_3$  in two micro-watersheds from the Das Mortes River.

## Materials and Methods

This study was performed in Campo Verde, which is located in the south-eastern part of the state of Mato Grosso. The climate is tropical savanna climate, with a rainy season between October and April and a dry season between May and September. Average annual precipitation is 1.800, with an average temperature ranging from 18 to 24°C (Meister, Nóbrega, Rieger, Wolf, & Gerold, 2017; Nóbrega et al., 2017).

We selected two micro-watersheds with different land uses (regenerated Cerrado and agricultural site) typical for the study area. The micro-watershed used for agricultural purposes (M-AGRO) covered an area of 135,4 ha and was located in the Santa Luzia Farm (S 15°44'16,9" and W 55°21'42,5"), which cultivates soybean (summer harvest) and corn (winter harvest) in succession. The fertilizers are applied onto the soil without incorporation.

The Cerrado micro-watershed (M-CER) had secondary Cerrado vegetation (more than 20 years of regeneration) and covered an area of 78 ha. It was located within the Rancho do Sol Farm (S 15 ° 47'42, 7" and W 55 ° 20'16, 6").

The Table 1 shows the soil characteristics of the two studied micro-watersheds.

**Table 1**  
**Physical and physical-hydric characterization of the soil attributes in the analyzed micro-watersheds, Campo Verde - MT, Brazil**

| Attributes | Depth (cm) | M-CER |      | M-AGRO |       |
|------------|------------|-------|------|--------|-------|
|            |            | Mean  | *SD  | Mean   | SD    |
| Clay (%)   | 00-10      | 12.12 | 5.95 | 56.76  | 9.52  |
|            | 10-20      | 10.49 | 2.62 | 52.84  | 7.49  |
|            | 20-40      | 11.31 | 0.77 | 56.18  | 10.84 |
|            | 40-60      | 11.88 | 1.19 | 60.36  | 5.19  |
| Silt (%)   | 00-10      | 2.44  | 2.24 | 15.59  | 6.21  |
|            | 10-20      | 1.89  | 1.61 | 21.21  | 7.81  |
|            | 20-40      | 1.27  | 0.48 | 18.45  | 5.91  |
|            | 40-60      | 1.87  | 0.91 | 19.60  | 5.05  |
| Sand (%)   | 00-10      | 85.45 | 8.01 | 27.64  | 14.24 |
|            | 10-20      | 87.62 | 4.02 | 25.95  | 12.10 |
|            | 20-40      | 87.42 | 1.02 | 25.38  | 13.43 |
|            | 40-60      | 86.24 | 0.83 | 20.05  | 3.47  |

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|  |       |       |       |       |       |
|--|-------|-------|-------|-------|-------|
| Microporosity (%)                            | 00-10 | 18.09 | 6.29  | 48.39 | 4.55  |
|  | 10-20 | 15.24 | 4.56  | 43.62 | 5.52  |
|  | 20-40 | 15.87 | 5.11  | 41.84 | 5.92  |
|  | 40-60 | 16.88 | 6.11  | 44.66 | 4.23  |
| Macroporosity (%)                            | 00-10 | 31.16 | 4.13  | 12.08 | 5.70  |
|  | 10-20 | 30.57 | 5.29  | 13.80 | 4.24  |
|  | 20-40 | 27.02 | 4.74  | 16.10 | 5.55  |
|  | 40-60 | 25.17 | 6.10  | 12.19 | 3.71  |
| Total porosity (%)                           | 00-10 | 49.26 | 3.87  | 60.47 | 5.96  |
|  | 10-20 | 45.81 | 2.06  | 57.42 | 4.04  |
|  | 20-40 | 42.89 | 3.01  | 57.94 | 5.73  |
|  | 40-60 | 42.05 | 0.94  | 56.86 | 4.87  |
| Field capacity (%)                           | 00-10 | 16.47 | 5.97  | 39.55 | 4.69  |
|  | 10-20 | 13.42 | 4.67  | 36.44 | 5.34  |
|  | 20-40 | 14.72 | 4.44  | 35.11 | 5.77  |
|  | 40-60 | 15.63 | 5.23  | 36.77 | 4.04  |
| Hydraulic conductivity (mm.h <sup>-1</sup> ) | 00-10 | 531.1 | 206.9 | 54.5  | 72.5  |
|  | 10-20 | 498.4 | 331.2 | 185.0 | 151.6 |
|  | 20-40 | 515.3 | 291.1 | 92.0  | 145.3 |
|  | 40-60 | 407.7 | 260.3 | 52.0  | 78.1  |
| Total organic carbon (%)                     | 00-20 | 0.94  | 0.62  | 2.60  | 0.26  |
|  | 20-40 | 0.60  | 0.43  | 1.47  | 0.20  |
| Total nitrogen (%)                           | 00-20 | 0.05  | 0.03  | 0.14  | 0.03  |
|  | 20-40 | 0.04  | 0.02  | 0.09  | 0.01  |
| Slope (%)                                    |       | 5.2   | 29.1  | 1.68  | 8.3   |

SD: standard deviation.

### *River flow monitoring*

Monitoring of the river flow started in January 2014 and ended in March 2015. In the rainy season, from November to April, data collection and water sampling were performed every 15 days, while in the dry season, we sampled every 30 days.

For river flow monitoring, we installed metal triangle spillways on the control section of the micro-watersheds, which contained a

multiparameter probe Hydrolab, DS5X model, to continually monitor the piezometric head above the spillway sill. The probe estimates the piezometric head of the water channel as a function of hydrostatic pressure.

The probes were installed at 2 m upstream of the spillway, where the water level is lower, to avoid interference with the piezometric head. Water level was measured in 10-min intervals.

### Calculation of river flow and specific river flow

To converse the outflow water level height above the sill into river flow, we used the Kindsvater-Shen equation (Equation 1) and its calibration adjustment functions (Equations 2 and 3):

$$Q = \frac{8}{15} C_e \sqrt{2g} \tan \tan \left( \frac{\theta}{2} \right) h e^{\frac{5}{2}} \quad (1)$$

$$Kh = 0.001 [\theta(1.395\theta - 0.05734) + 4.135] \quad (2)$$

$$C_e = \theta(0.02286\theta - 0.05734) + 0.6115 \quad (3)$$

Here, Q is the spillway outflow ( $\text{m}^3 \text{s}^{-1}$ ),  $C_e$  is the effective discharge coefficient to the triangle spillway (dimensionless),  $\theta$  is the spillway angle (radian), h is the water level above the spillway sill, Kh is the adjustment factor of the outflow, and he is the effective water level outflow (h+Kh).

Based on the specific river flow, we compared the two micro-watersheds. is the specific river flow is the quantity of water produced by the drainage area and can be calculated as follows (Equation 4):

$$Q_e = \frac{Q}{A}, \quad (4)$$

where  $Q_e$  = specific river flow, in  $\text{L s}^{-1} \text{ha}^{-1}$ ; Q = river flow in  $\text{L s}^{-1}$ , and A = watershed drainage area in ha.

### Precipitation monitoring

In each micro-watershed, we installed three pluviometers with a tipping bucket, which recorded the precipitation height every 10 minutes. In this way, based on the precipitation data and on the analyzed water variables, we evaluated the changes in the micro-watershed during rain events.

### Water sampling

At the control section of each micro-watershed, we installed an automatic water sampler (BL2000®, Hach-Lange GmbH), which contained 24 1-L bottles. Automatic sampling was performed simultaneously with water level measurements to represent basis flow or flow during rain. The chosen interval was 14.4 hours for each extraction of 200 mL, with five samples over 72 hours to fill a 1-L bottle.

Flow sampling during rain was activated by the increase in water level via a hydrostatic level sensor (FD-01, Profimess), which was connected to the automatic sampler to drive the water sample each time when the watercourse level changed by 5 cm. Each sampling event lasted for 5 minutes, filling a 1-L water bottle in 30.

Each bottle was filled during one program, which means that as soon as the second program was activated, the automatic sampler selected one bottle different bottle of that one that was being filled during the first program. Prior to collecting the bottles for laboratory analysis, we downloaded information related to the sampling time and program.

Samples were collected every 10 days and packed in a cool box with ice, according to the recommendations of the National Sample Collection and Preservation Guide (Agência Nacional de Águas [ANA], 2011).

### Preparation and analysis of water samples

The DOC and  $\text{NO}_3$  analyses were performed in the ecophysiology laboratory of the Federal University of Mato Grosso. For this, we used 200 mL of the water samples,

which were filtered through 0.45- $\mu\text{m}$  fiberglass filters without organic resin (Whatman, with a diameter of 47 mm), so that only the dissolved carbon organic fraction was analyzed.

To speed up filtration, we used a Millipore filtration system with a vacuum pump. After filtration, the samples were analyzed using a UV-Vis spectrophotometer (Spectrolyser; Scan, Austria) at an absorption spectrum of 200 to 750 nm. The  $\text{NO}_3$  and DOC concentrations were calculated based on the absorbance spectrum of each sample, using the ANAPro software.

#### *Calculation of dissolved organic carbon (DOC) and nitrate ( $\text{NO}_3$ )*

The DOC and  $\text{NO}_3$  flows represent the transported quantity of these substances to the micro-watershed outlet. The flow was obtained by means of the concentrations of DOC,  $\text{NO}_3$  and the specific river flow, using the following equation:

$$\text{Fluxo}_{\text{COD ou NO}_3} = C_{\text{COD ou NO}_3} \times Q_e, \quad (5)$$

where the flow is represented in  $\text{g ha}^{-1} \text{s}^{-1}$ , C is the concentration of DOC and  $\text{NO}_3$ , in  $\text{g L}^{-1}$ ; and  $Q_e$  is the specific flow, in  $\text{L s}^{-1} \text{ha}^{-1}$ .

#### *Statistical data analysis*

To determine the relationship between water quality parameters in the dry and rainy seasons, we performed Pearson's correlation analysis. The correlation coefficients were obtained at a significance level of 5%, using the statistical software IBM® SPSS®17.

## **Results and Discussion**

Figure 1 A-B shows the distribution of the rain events for both micro-watersheds. The total amount of precipitation during this study was about 1.300 mm, with distinct dry and rainy seasons. In the M-CER, we measured 74 days with rain, while the M-AGRO received rain on 82 days.

In both watersheds, the dry season occurred between April and September; isolated rain events accounted for 5.4 and 8.3% of the annual precipitation at the M-AGRO and M-CER watersheds, respectively.

#### *Specific river flow in the micro-watersheds*

Figure 1 C-D shows the hydrograph with the specific river flow variation ( $Q_e$ ) on days with and without rain for M-AGRO and M-CER. Several factors, such as precipitation, can modify the river flow behavior in a micro watershed. As the pluviometric patterns of both watersheds were similar, without intervention with watercourses, the differences in the hydrological behavior are related to the soil characteristics of the micro-watersheds (Table 1).

Menezes, Prado, Silva, Mansur and Oliveira (2009) stated that the higher river flows found in the rainy season are because the water inflow into groundwater is higher than the quantity lost to the atmosphere. The authors highlight that in the dry season, the groundwater recharge is affected due to the low quantity of precipitation and the greater water use by plants, in addition to higher evapotranspiration, resulting in a lower base flow.



We noted a difference between the mean values of specific flow between the watersheds as well as the measured flow during days with and without rain. In the micro-watershed M-CER, the specific river flow increased by around 78% in the days with rain, while in the M-AGRO, this increase was only 57%.

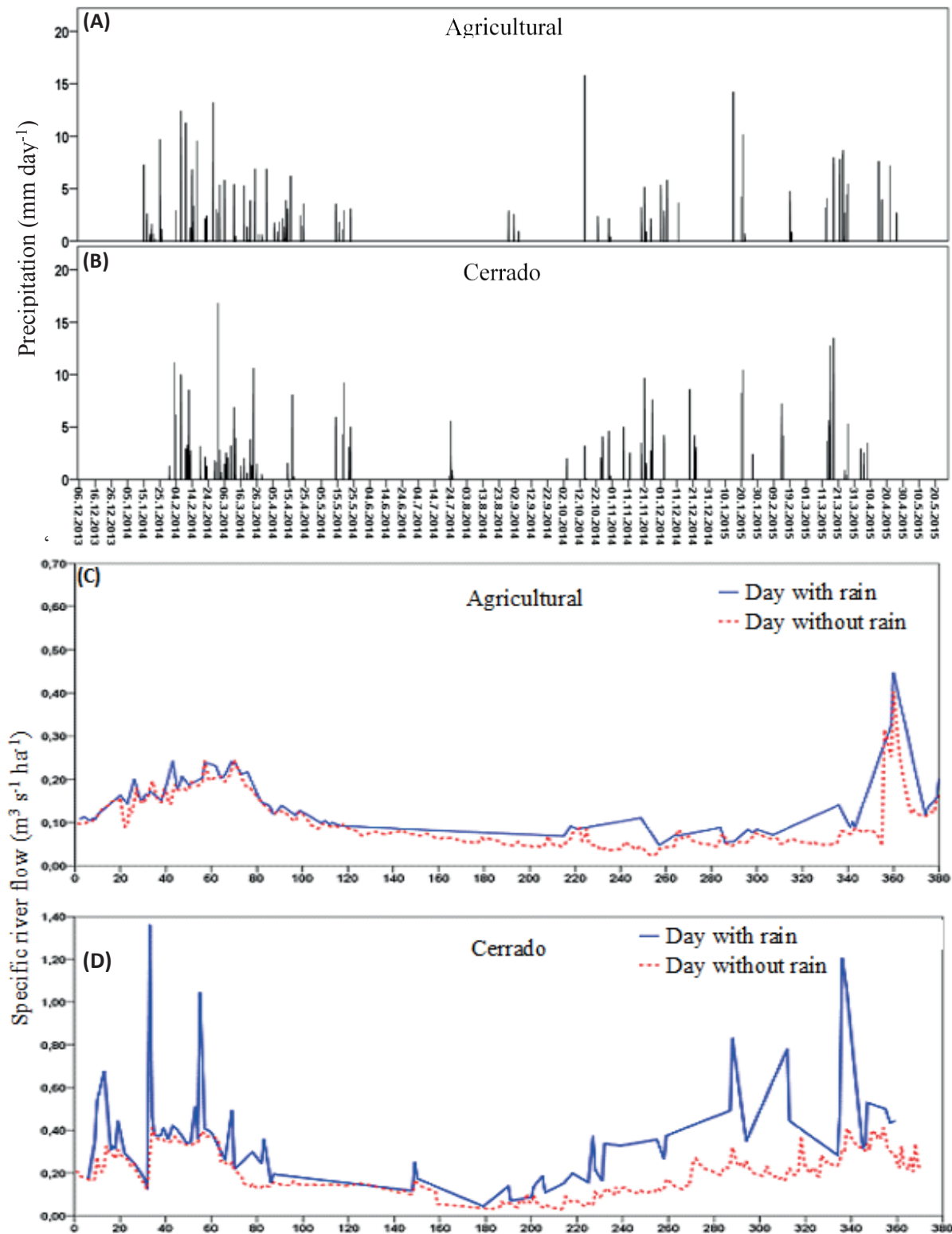
The higher increase in the specific river flow in the M-CER can be related to the sandy soil, with a sand content of more than 85%, a high hydraulic conductivity, a low water retention capacity, and high permeability and slope (Table 1). In this way, during rain events, water percolates more easily through the soil profile, also because of the higher slope in this watershed (Table 1) and the subsurface and base flow get the watercourse in lower time, which resulted in the difference between the base flows and peak between the micro-watersheds.

Generally, micro-watersheds with pasture and native vegetation are characterized by sandy soils, with good permeability and water infiltration (Bocuti, Amorim, Santos, Di Raimo, & Pereira, 2019). The M-CER, despite having a soil with high infiltration capacity, had a low water retention capacity, allowing water infiltration and percolation; as a result, water transportation to the watercourse is increased, which led to a 285% higher river flow on days with rain (Figure 1-D).

### *DOC and NO<sub>3</sub> concentrations and flow in the micro-watersheds*

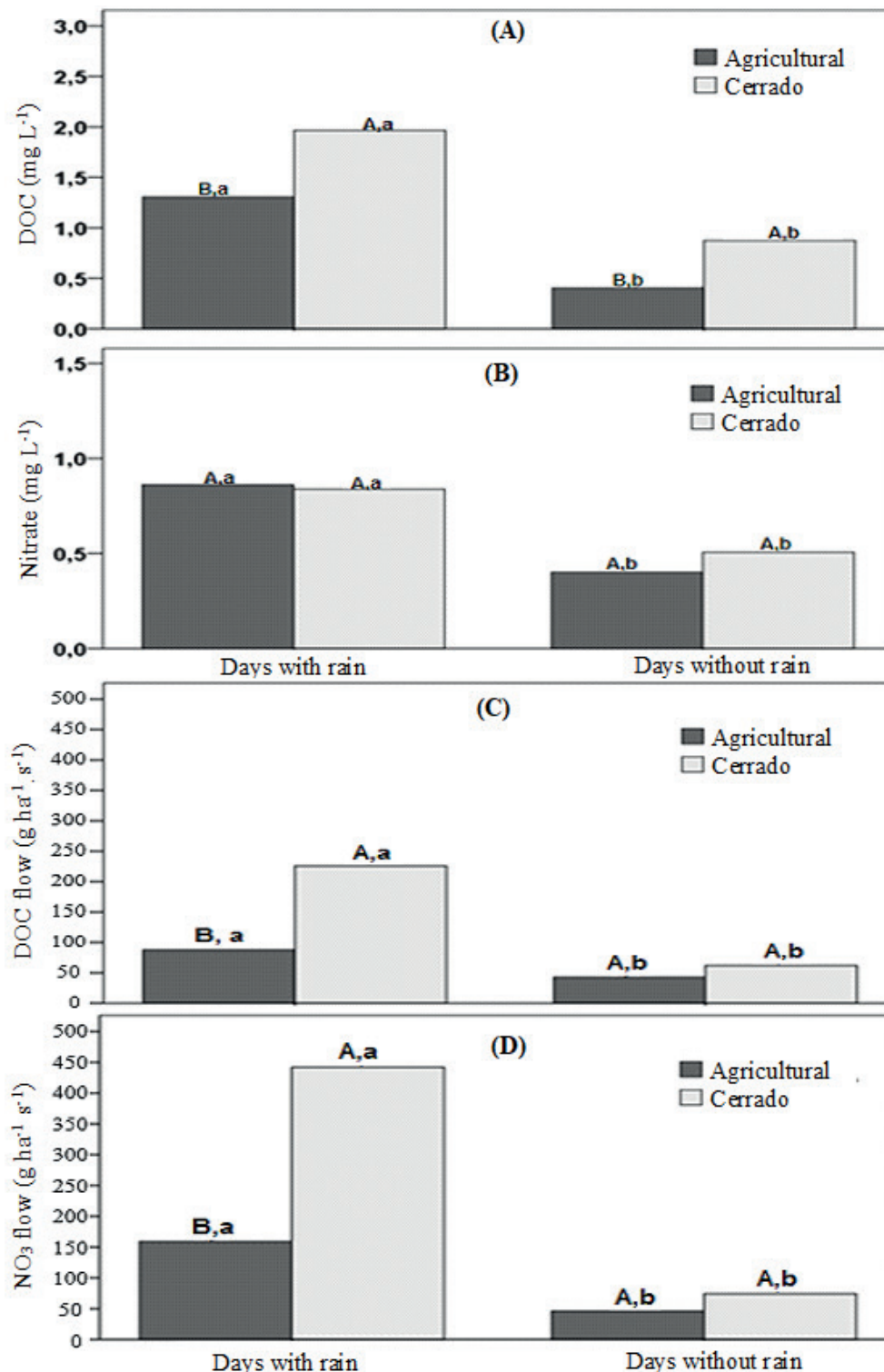
The average DOC and NO<sub>3</sub> concentrations, measured in the surface water of the micro-watersheds in the periods with and without rain, are shown in Figure 2 A-B. The obtained values in this study are according to the range of values observed by Toledo and Nicolella (2002).

Based on Figure 2-A, M-CER had higher DOC concentrations in the periods with and without rain, mainly because of its sandy texture and higher slope. These factors contribute to soluviation processes in the soil and the surface waters, which are intensified via rain.



**Figure 1.** Daily precipitation (Agricultural - A and Cerrado - B) and hydrograph of the observed daily specific river flow in the days with and without rain in the agricultural (C) and Cerrado (D) micro-watersheds.





**Figure 2.** Concentration and flow of dissolved organic carbon - DOC (A and C) and nitrate - NO<sub>3</sub> (B and D) in the surface waters of the Cerrado and agricultural micro-watersheds on days with and without rain. \*Different capital letters indicate a significant difference between micro-watersheds; different lowercase letters indicate a significant difference within the same micro-watershed.

Both watersheds had increased levels of DOC and  $\text{NO}_3$  on days with rain, which is in agreement with the findings of Ríos-Villamizar, Ríos and Waichman (2011) and Lopes and Magalhães (2010). The M-AGRO (DOC - 225% and  $\text{NO}_3$  - 90%) had higher increases than M-CER (DOC - 127% and  $\text{NO}_3$  - 66%), which can be explained by the differences in soil characteristics (Table 1). The soil in M-AGRO was more clayey, with an average clay content of approximately 53% in the 0-60-cm soil layer, a higher microporosity, an higher water retention capacity, and a lower permeability. During the rainy season, in some places, we observed groundwater outcropping, indicating an increased soil moisture.

Because of the higher clay content and microporosity, the soil of M-AGRO was less susceptible to the soluviation of solutes such as  $\text{NO}_3$  and DOC. However, due to its lower hydraulic conductivity and macroporosity (Table 1), resulting in lower permeability, this soil was more prone to runoff, which probably intensified the transportation of DOC and  $\text{NO}_3$  into the watercourse.

The increase in DOC concentration with the increase in precipitation is common in the rainy season. Andrietti et al. (2016) also observed an increase in DOC during the period of higher river flow, associated to a higher washing out of the surface layers through the runoff.

Countway, Canuel and Dickhut (2007) highlighted that erosion and runoff are the main sources of organic and inorganic compounds in watercourses. During transport, organic matter can be modified through biotic and abiotic processes, such as photo-oxidation, adsorption and desorption, microbial respiration, flocculation, precipitation,

and immobilization in the sediments, thus contributing to particulate and dissolved carbon.

In the M-CER, the lower increment of DOC and  $\text{NO}_3$ , on days with rain might be a result of lower runoff volumes.

According to Beltrame (1994), the physical soil characteristics, such as texture, porosity, and depth, influence rainfall infiltration capacity and runoff velocity, which affect erosivity processes.

The soil of M-CER had a higher sand content and a higher hydraulic conductivity (about 10 times more at the 0-10-cm-layer), with a higher macroporosity and a lower microporosity (Table 1). These characteristics result in a lower water retention, a higher permeability, and a higher infiltration capacity. Therefore, at rain events with the same intensity, the runoff volume is lower in M-CER, reducing DOC and  $\text{NO}_3$  inputs.

According to Beltrame (1994), vegetation coverage, apart from protecting the soil against the impacts of raindrops, promotes porosity and soil permeability, thereby reducing runoff volume and velocity. Because M-CER had a perennial vegetation, the effect of vegetation in this site was more pronounced when compared to M-AGRO with a more seasonal vegetation.

Combined with rain, DOC flows into the watersheds were higher. The M-CER received higher DOC inputs, both on days with and without rain (Figure 2-C).

Regarding the  $\text{NO}_3$  flow, although the levels were similar for days with and without rain,  $\text{NO}_3$  flow into M-CER was higher (Figure 2-D). Considering that the  $\text{NO}_3$  flow is obtained by the product of the specific river flow and

the concentration of the compound, the higher  $\text{NO}_3$  flows must be the higher observed specific river flow in M-CER.

## Conclusions

The micro-watershed with Cerrado vegetation had a higher specific flow and a higher increment on days with rain when compared to the watershed with agricultural production

The DOC concentrations were higher in the Cerrado micro-watershed.

There was no difference in the  $\text{NO}_3$  concentrations between the two micro-watersheds.

In both micro-watersheds, there was a significant increase in DOC and  $\text{NO}_3$  concentrations on days with rain.

On days with rain, the DOC and  $\text{NO}_3$  flows were higher in the micro-watershed with Cerrado vegetation.

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