

Short Communication

Physical, chemical and microbiological characterization of the soils contaminated by iron ore tailing mud after Fundão Dam disaster in Brazil

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ABSTRACT

The collapse of the Fundão dam in Mariana – Minas Gerais, in the Iron Quadrilateral, considerably modified the Carmo riverbank. This study aimed to perform a physical, chemical, and biological characterization of the soils contaminated by iron ore tailing mud after the dam disaster. From the riverbank to uncontaminated soil, three sites were sampled: site I (sandy sediment), site II (iron ore tailing mud) and site III (pasture soil). Analysis were performed to characterize soil physical features and soil fertility, fumigation-extraction method was applied to measure soil microbial biomass C, and to evaluate the photobiotic organisms' diversity by a growth experiment in a controlled chamber, for the samples collected in three sites. The results showed that site I presented quartz as the main mineral; site II presented both quartz and hematite; and site III presented greater diversity in the mineralogical composition. Site III showed greater fertility, except for the phosphorus and iron content that was higher in site II. The sites I and II have higher values of metabolic quotient and lower microbial carbon/organic carbon ratio, unlike site III. The results indicated that after one year of the dam failure the microbial activity was disturbed, as the nutrients and carbon concentration from the sites were decreased due to the presence of iron ore tailing mud in comparison to unaffected soil from the same area. However, a community of phototrophic microorganisms has been observed with a predominance of cyanobacteria, which may be important to enhance the development of this initial stage of succession.

1. Introduction

Considering the soil importance in maintaining the ecosystem equilibrium, soil microbial attributes has been used to diagnose and monitor impacted areas, as it is reported by a large number of research works (Martins et al., 2018; Niemeyer et al., 2012; Quadros et al., 2016). In general, a microbial population is an indicator of the soil quality, which is essential for a better comprehension of the soil properties. This is due to the sensibility of the soil microorganisms' population, which is easily affected by anthropogenic or natural disturbances in an ecosystem (Turco et al., 1994; You et al., 2019). Soil microbial biomass carbon, basal respiration and nutrients, such as soil carbon, nitrogen and phosphorus, are examples of soil microbial and chemical attributes

frequently used (Niemeyer et al., 2012; Quadros et al., 2016). According to Anderson and Domsch (2010), for a proper understanding of these soil attributes, it is necessary to comprehend the relationship among microbial biomass, energy transfer and carbon demand. The microbial metabolic quotient (qCO_2) may reflect this relationship, since it correlates the efficiency of energy use with the development of the microbial community (Zhang et al., 2008; Anderson and Domsch, 2010). In impacted regions, soil microorganisms under stress may be metabolically less efficient, as they have to invest more energy to maintain cells, resulting in an increase in CO_2 released per unit of microbial biomass (Brumme et al., 2009; Anderson and Domsch, 2010). In ecological terms, this means that a high qCO_2 reflects a high maintenance carbon demand, and if the soil system of the impacted area cannot replenish the carbon,

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which is consumed during respiration process, the microbial biomass must decline and ecological succession process might be significantly affected (Anderson and Domsch, 2010). In addition to soil microbial attributes, biocrusts is also important in the ecological restoration process (Rao et al., 2009; Wang et al., 2009; Bowker et al., 2017). Biocrusts include a variety of soil surface communities dominated by many different combinations of phototrophic and heterotrophic microbes, and cryptogams (e.g., mosses, lichens). They are exceptionally effective in accumulating nutrient reserves and important for the ecological succession process (Bowker et al., 2017).

On November 5th, 2015, Brazil experienced one of the largest environmental disasters in the country's history when an iron mine dam (Fundão Dam) failed in the municipality of Mariana, Minas Gerais State (MG) (Agurto-Detzel et al., 2016). The devastation impacted approximately 1469 ha of natural vegetation and 90% of the riparian habitats of the Fundão, the North Gualaxo and Carmo Rivers. The flow of tailings buried aquatic and riparian nursery habitats, eliminating most of the regenerative capacity of aquatic and terrestrial ecosystems. The thick tailings deposit layer can extend recovery time of soil biophysical structure and biodiversity in affected area from decades (Fernandes et al., 2016). Therefore, given the essentialness of the soil in the ecosystem equilibrium and recovery, the soil microbial attributes from the Doce River Basin area should be deeply studied. Although there has been scientific reports on how the affected region is regenerating

(Cordeiro et al., 2019; Dias et al., 2017; Fernandes et al., 2016; Neves et al., 2016; Santos et al., 2019; Cruz et al., 2020), there is none regarding the soil microbial activity and phototrophic organisms diversity.

The study aimed to perform a physical, chemical and biological characterization of the soils contaminated by iron ore tailing mud after Fundão Dam disaster and was conducted on Carmo riverbank one year after the disaster, where three different sites were identified and classified according to the distance from the river. The following questions were raised: (i) what are the physical and chemical characteristics of the soil in these sites affected by mud (iron ore dam tailing)?; (ii) are there differences in soil microbial attributes between the sites?; and (iii) what is the photobiotic organism's diversity in these sites?

2. Material and methods

2.1. Location and characterization of the study area

The samples were obtained from the southern end of the Espinhaço Range, located in the Minas Gerais State, southeastern Brazil, in the Iron Quadrilateral region on the banks of the Carmo River (20°16'31.24"S and 43°0'54.57"W), a tributary of the Doce River located in the municipality of Barra Longa. The failure of the Fundão dam caused a drag of 34 million cubic meters of iron ore tailings along the Doce River and its

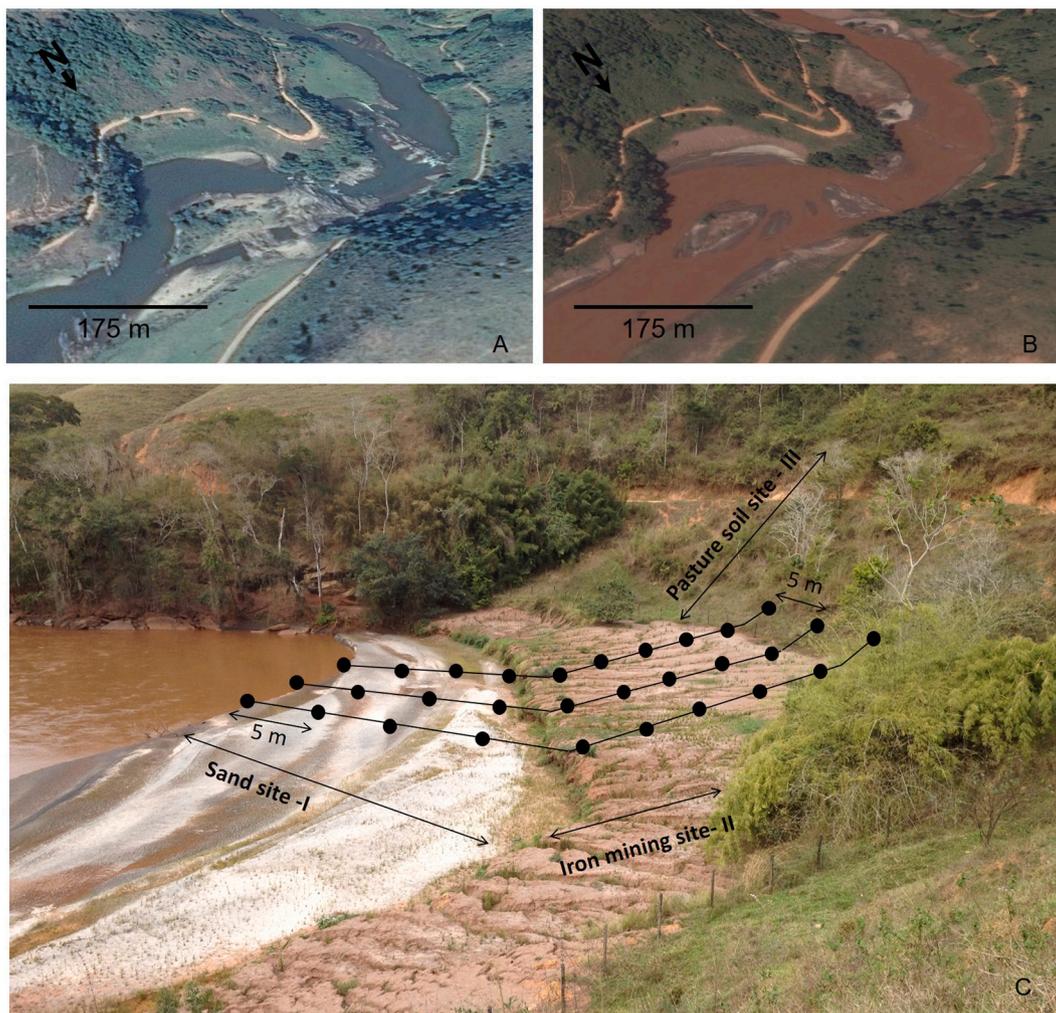


Fig. 1. Study area on the banks of the Carmo River before (A) and after (B) the dam failure. Sampling of the experimental area in the site I (constituted by sandy sediment of the river bed and with absence of riparian vegetation), II (area defined by a slope approximately 2 m high, consisting primarily of iron ore tailing mud, where the riparian vegetation was covered) and III (further away area from the river, but with degraded soil due to grass cover used for grazing livestock) on the edge Carmo River (C).

tributaries (Fig. 1A, B). The area not contaminated at the top of the Carmo River are rock outcrops of quartzite, itabirite, and phyllites, with soil classified by the WRB System as Ferralsols, Leptosols and Cambisols. Downstream, the riverbeds are formed out of eutrophic Ferralsols and Latosols (Foesch et al., 2020).

2.2. Field layout and sampling

The study area was separated in three sites (Fig. 1C): Site I) sandy sediment; Site II) iron ore tailing mud; and Site III) pasture soil (not contaminated). From the riverbank to the pasture soil, three sampling lines of 10 × 50 m (Fig. 1C) were drawn on the soil (area of 1200 m²). Samples were collected from 0 to 5 cm deep with an approximate mass of 500 g every five meters (15 samples for site I and II, and three samples of site III). The samples collected in each site were air-dried, crushed, homogenized, passed through 2 mm stainless steel sieves (Tyler series sieves) and stored at 7 ± 3 °C. The analysis of the microbial attributes was performed shortly after the collection.

Granulometric analysis of the fine silt and clay fractions were performed in triplicate using a Laser Granulometer (CILAS, model 1090). Mineralogical phases identification were performed by powder method of X-ray diffraction (XRD) using a diffractometer Shimadzu 7000 under the following operating conditions: Cu K α radiation (35 kV/40 mA), goniometer speed of 0.02° per step 2 θ , with 5 s per step, and collected from 5° to 80° 2 θ . Spectra interpretation was carried out by comparisons with standards contained in the PDF 02 database – ICDD 2003.

2.3. Soil organic carbon (SOC) and chemistry analysis

The quantification of oxidisable soil organic carbon content was determined by wet oxidation method developed by Walkley and Black (1934). The evaluated soil chemical properties were as follows: soil pH, exchangeable calcium (Ca²⁺) (extractor: KCl – 1 mol L⁻¹), exchangeable magnesium (Mg²⁺) (extractor: KCl – 1 mol L⁻¹), cation exchange capacity (CEC), sum of bases (SB), base saturation (BS), available phosphorus (P) (Mehlich 1), aluminum (Mehlich 1), micronutrients (Cu, Fe, Mn, Zn) (Mehlich1) (Claessen et al., 1997).

2.4. Microbial biomass carbon (MBC), basal respiration (BR) and metabolic quotient (qCO₂)

The procedures established by Vance et al. (1987) were followed. The method was developed by using chloroform fumigation and extraction with 20 mL K₂SO₄ (0.5 mol L⁻¹) and potassium dichromate digestion methods. A correction factor (K_c) of 0.33 was used to estimate microbial carbon (Silva et al., 2007). BR was determined in according to Jenkinson and Powelson (1976). The respirometry test was performed in the dark at room temperature for 144 h using a Bartha respirometer with KOH solution (10 mL, 0.1 mol L⁻¹). The specific respiration of the biomass (qCO₂), relation between CO₂ released (BR) and the microbial carbon (C_{mic}), was expressed in mg CO₂-C mg⁻¹ C_{mic} h⁻¹, and the ratio between the soil microbial carbon (C_{mic}) and the soil organic carbon (C_{org}), in percentage.

2.5. Evaluation of photobiotic organisms' growth on samples

For the photobiotic organism's growth experiment, 50 g of the samples were placed in a transparent container (Gerbox plate). The samples, in duplicates, were incubated in a germinating chamber with controlled temperature (25 °C) in an alternating regime of light and dark (photoperiod 12 h) for 63 days. The field capacity moisture was maintained throughout the experiment by weekly humidification. After incubation, the samples were analyzed and photographed on a Carl Zeiss photo-stereomicroscopic and Zeiss M37 photomicroscope using 1 mL of water aiming at the observation of the microorganisms.

2.6. Statistical analysis

Parametric analysis were performed for the results of BR, MBC, qCO₂, SOC and fertility results using the Bartlett tests (Sileshi, 2012). The values were submitted to analysis of variance (One-way ANOVA) and the means compared by the Tukey HSD test. All analyzes were performed by Statistica 7.0 software.

3. Results and discussion

The granulometric analysis showed that the fine particles were predominant in site I and II and coarse particles (Table 1) in site III. Site I presented higher percentage (68.5%) of particles size between 0.075 and 0.6 mm. Particles with diameter smaller than 0.075 mm were predominant in the site II (61.7%). Coarse particles (0.6–2.38 mm) were found in a higher amount in site III (47%) (Table 1).

The results of the mineralogical characterization obtained by X-ray diffraction are shown in Fig. 2. Site I presented quartz as the main mineral. Site II presented both quartz (SiO₂) and hematite (Fe₂O₃), which are typical minerals of tailing dam from iron mining. Site III, on the other hand, presented greater diversity in the mineralogical composition consisting of the following mineral phases: quartz (SiO₂), hematite (Fe₂O₃), goethite (FeOOH), kaolinite (Al₂Si₂O₅ (OH)₄), muscovite (KAl₂(AlSi₃O₁₀)(OH)₂) and calcite (CaCO₃).

The organic carbon was significantly lower in the site I ($P < 0.0001$) and higher in the site where the iron ore tailing was not deposited (site III) (Table 1). The fertility values were compared to reference values for the Cerrado soil according to the classification proposed by Freire et al. (2008). The site III was considered of medium acidity (pH = 5.1–6), presenting higher acidity results than the other samples. All sites presented low results (<4.3%) of cation exchange capacity (CEC); site III presented the highest result. Site II presented a high base saturation result (60.1–80%), while site I and III, average (40.1–60%) (Table 1). Site III presented the sum of the bases higher.

Studies have shown that the increased presence of sandy sediment is a disadvantage for organic carbon accumulation, while greater presence of fine particles such as silt and clay, provides better organic carbon packaging and micronutrients due to the protection afforded by the fine particles (Gao et al., 2014). The same correlation is established for the CEC; clay soils have a higher ability to retain cations by electrostatic forces than sandy soils (Liang et al., 2006). This justifies the higher values of organic carbon and CEC in site III (composed by pasture soil),

Table 1

Samples physical and chemical properties (layer: 0–5 cm) of an experimental area in the site I, II and III on the Carmo riverbank, Barra Longa (MG), Brazil.

Properties*	Sites		
	I	II	III
Particle size % (0.075–2,38 mm)	69.6	44	74.1
Particle size % (<0.075 mm)	30.4	56	25.9
Organic Carbon (g C kg ⁻¹)	15.07 ± 0.15 _a	19.02 ± 0.44 _b	41.54 ± 0.09 _c
pH (H ₂ O)	6.4 ± 0.2 ^a	7.5 ± 0.2 ^b	5.7 ± 0.1 ^c
Ca ²⁺ (cmol _c kg ⁻¹)	0.44 ± 0.04 ^a	1.31 ± 0.05 ^b	1.04 ± 0.02 ^c
Mg ²⁺ (cmol _c kg ⁻¹)	0.07 ± 0.01 ^a	0.04 ± 0.00 ^b	0.97 ± 0.02 ^c
Cation-exchange capacity (%)	1.18 ± 0.06 ^a	2.05 ± 0.05 ^b	4.25 ± 0.03 ^c
Base saturation (%)	44.2 ± 1.6 ^a	69.3 ± 1.0 ^b	52.1 ± 0.4 ^c
Sum of the bases (cmol _c kg ⁻¹)	0.53 ± 0.04 ^a	1.42 ± 0.05 ^b	2.18 ± 0.07 ^c
P (mg kg ⁻¹)	3.93 ± 0.4 ^a	11.70 ± 0.3 ^b	1.43 ± 0.42 ^c
Fe (mg kg ⁻¹)	92.2 ± 12.3 ^a	216.1 ± 21.9 ^b	81.6 ± 31.4 ^c
Cu (mg kg ⁻¹)	0.37 ± 0.06 ^a	1.13 ± 0.06 ^b	1.20 ± 0.10 ^b
Zn (mg kg ⁻¹)	0.40 ± 0.00 ^a	0.80 ± 0.10 ^b	1.13 ± 0.12 ^c
Mn (mg kg ⁻¹)	53.0 ± 5.9 ^a	248.7 ± 8.8 ^b	71.7 ± 13.5 ^a

* The values represent the mean ± S.E. (n = 3). Mean values followed by the same letters in the same column at each soil depth indicate no significant difference among treatment by the Tukey test ($P < 0.05$).

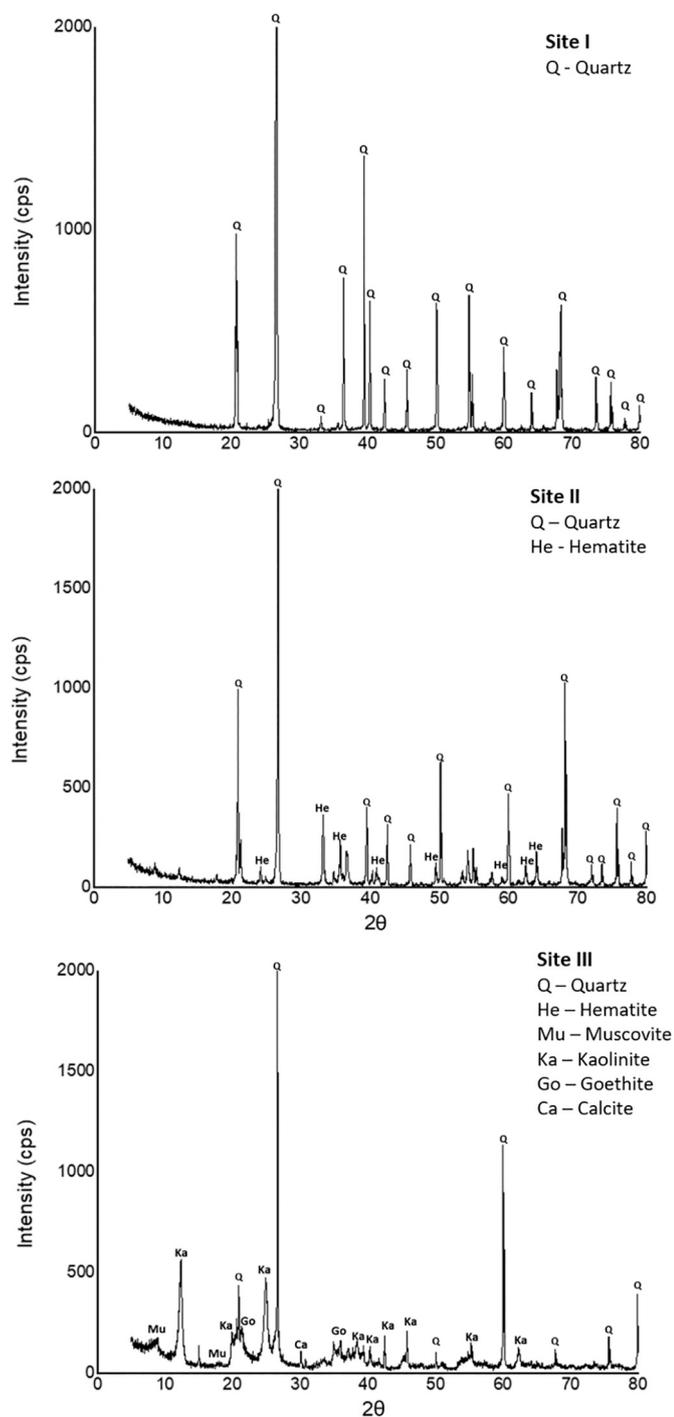


Fig. 2. X-ray diffractograms showing the mineralogical composition of the samples from experimental area (layer: 0–5 cm) in the site I, II and III on the Carmo riverbank, Barra Longa (MG), Brazil.

which presents clay minerals, such as kaolinite and muscovite (Fig. 2), that are capable of retaining carbon and micronutrients. On the contrary, site I, which presented the lowest values of organic carbon and CEC (Table 1) is mainly composed of quartz (Fig. 2). The fine particles presented in site II are mostly due to the processing to obtain the iron ore (quartz and hematite), and, for this reason, they may not have the capacity to retain organic carbon and micronutrients as the soil in site III, as observed in Table 1.

Nutrient phosphorus was present in a high content ($> 10 \text{ mg kg}^{-1}$) in site II, while potassium content was higher in site III (Table 1). The

results suggest that the high presence of soluble phosphorus adsorbed into the samples from site II might be because of the high affinity of iron oxides for phosphate (Fink et al., 2016), since it presents high amount of iron oxides as hematite (Fig. 2). This is due to a process undertaken by functional groups in the iron oxide, which resulted in the formation of surface complexes with no intercalated water molecules that are specifically adsorbed (Fink et al., 2016). It is believed that the source of the phosphorus adsorbed in site II is the Carmo River, where the mud followed its flow from the rupture of the dam to the collection area. This is justified by the presence of areas of agricultural activity where the river goes through and is subject to phosphorus contamination by the leaching of the soil fertilized with phosphate compounds, as well as other variables, such as the river pollution by sewage and the death of thousands of fish caused by the dam rupture.

Regarding iron and manganese micronutrients, site II presented significantly higher results ($P < 0.001$). However, all sites had high levels of iron and manganese ($> 45 \text{ mg kg}^{-1}$ and $> 12 \text{ mg kg}^{-1}$, respectively). Both copper and zinc content were higher for site III, which is considered to be of medium availability for zinc ($1\text{--}1.5 \text{ mg kg}^{-1}$) and for copper ($0.8\text{--}1.2 \text{ mg kg}^{-1}$) (Table 1). Site II showed low availability of zinc ($0.5\text{--}0.9 \text{ mg kg}^{-1}$) and average availability for copper. Site I presented the lowest results for all micronutrients analyzed (Table 1). The manganese was significantly higher in site II ($P < 0.001$), which can be justified by the pH of the iron ore tailing disposed in this site (Table 1). One of the factors that determines the availability of manganese in the soil is the pH, since values lower than 6.5 promote the reduction of Mn^{4+} to Mn^{2+} . However, the high pH in region II contributes to the formation of insoluble manganese compounds such as MnO_2 , Mn_2O_3 and Mn_3O_4 , which are not usable by microorganisms (Stevenson and Cole, 1999).

The SOC values were lower in the site I and II compared to the ones found in site III, and the same relation was observed to microbial biomass (Table 2). Site I showed significantly lower values of BR, MBC and SOC and a higher value of $q\text{CO}_2$, unlike site III, which presented lower values of $q\text{CO}_2$ and the highest values of BR, MBC and SOC (Tables 1 and 2).

The development of microbial biomass is related to the amount of organic carbon present in the soil and to the synthesis of this carbon by the microbial community (Sparling, 1992; Frouz and Nováková, 2005; Nogueira et al., 2006; Anderson and Domsch, 2010; Niemeier et al., 2012; Quadros et al., 2016). In this context, the areas that presented high MBC and SOC, according to Kaschuk et al. (2011), can provide greater nutrient cycling if organic carbon is used efficiently by the microorganisms' diversity.

The increase of the microbial diversity in an ecosystem coincides with the increase energy use efficiency during the growth process of the microbial community (Anderson and Domsch, 2010). When the

Table 2

Soil basal respiration (BR), microbial biomass carbon (MBC), metabolic quotient ($q\text{CO}_2$) and soil organic carbon (SOC) at 0–5 depths of experimental area in the site I, II and III on the Carmo riverbank, Barra Longa (MG), Brazil.

Microbial attributes*	Sites		
	I	II	III
BR ($\text{mg C-CO}_2 \text{ kg}^{-1} \text{ h}^{-1}$)	0.442 ± 0.143^a	0.682 ± 0.124^b	0.667 ± 0.069^b
MBC (mg C kg^{-1})	196.5 ± 67.9^a	194.8 ± 52.2^a	542.5 ± 113.1^b
$q\text{CO}_2$ ($\text{mg CO}_2\text{-C mg}^{-1} \text{ C}_{\text{mic}}$ h^{-1})	$0.0041 \pm$ 0.002^a	$0.0037 \pm$ 0.001^a	$0.0013 \pm$ 0.0002^b
SOC (mg C kg^{-1})	$15,076.5 \pm 0.15$ _a	$19,017.5 \pm 0.44$ _b	$41,541.1 \pm 0.09$ _c
$\text{C}_{\text{mic}}/\text{C}_{\text{org}}$ ratio (%)	0.98 ± 0.29^a	0.99 ± 0.23^a	1.48 ± 0.06^b

* The values represent the mean \pm S.E. Mean values followed by the same letters in the same column at each soil depth indicate no significant difference among treatment by the Tukey test ($P < 0.05$). C_{mic} , microbial carbon; C_{org} , organic carbon.

equilibrium of the microorganisms in the soil is reached, any increase of the organic matter concentration results in a greater respiration, neither accumulating nor reducing the amount of organic matter presented in the soil. A low qCO_2 and a high C_{mic}/C_{org} ratio were observed in the samples collected in site III (Table 2). This represents the maturity of the microbial community in this area. The same cannot be applied to the samples collected in site I and II. The high qCO_2 and low C_{mic}/C_{org} ratio observed in these samples (Table 2) suggest an unstable microbial community.

The samples collected from each study site presented biological crust formation with 63 days in the incubation chamber. In site I, it could be noticed the growth of bryophyte (Fig. 3A), cyanobacteria of the *Anabaena* genus (Fig. 3B) and alga of the *Trentepohlia* genus (Fig. 3C). The filamentous cyanobacteria *Anabaena* sp. presents heterocyst cells, which make them capable of transforming elemental nitrogen into organic nitrogen (Popa et al., 2007). Thus, these species are important in the process of nitrogen cycling for the area with ore tailings mud (site II), as well as the species of *Scytonema* sp. found in site III (Fig. 3F, G). Although the *Scytonema* genus is considered cosmopolitan, it has been substantially found in tropical forests (Myers et al., 2000). *Anabaena*

species may be associated with different species of bryophytes (Arróniz-Crespo et al., 2014).

The presence of the *Pseudanabaena* sp. was observed in site II (Fig. 3D), a cyanobacteria species unable to fix nitrogen. However, according to Acinas et al. (2009), smaller cyanobacteria such as *Pseudanabaena* sp., often exceed the larger species of cyanobacteria in terms of biomass, playing an important role in the composition of the biological crust. In site III, it could be observed the presence of pteridophyte gametophyte (prothallus) (Fig. 3E), bryophyte and cyanobacteria *Scytonema* sp. of the Nostocales order (Fig. 3F and G). According to Bu et al. (2015), cyanobacteria, lichens and bryophytes are the predominant organisms in soil biological encrustation, but the cyanobacteria are considered the most important, since they are pioneers in the process of colonization and photosynthesizes. Studies using artificial biological crusts demonstrate that the presence of cyanobacteria increased the activity of soil biological communities and accelerated the process of restoration of areas located in arid zones considered difficult to restore (Rao et al., 2009; Wang et al., 2009; Bowker et al., 2017).

We found that pasture soil (not contaminated) presented higher C_{mic}/C_{org} ratio and reduced qCO_2 , indicating that most of the

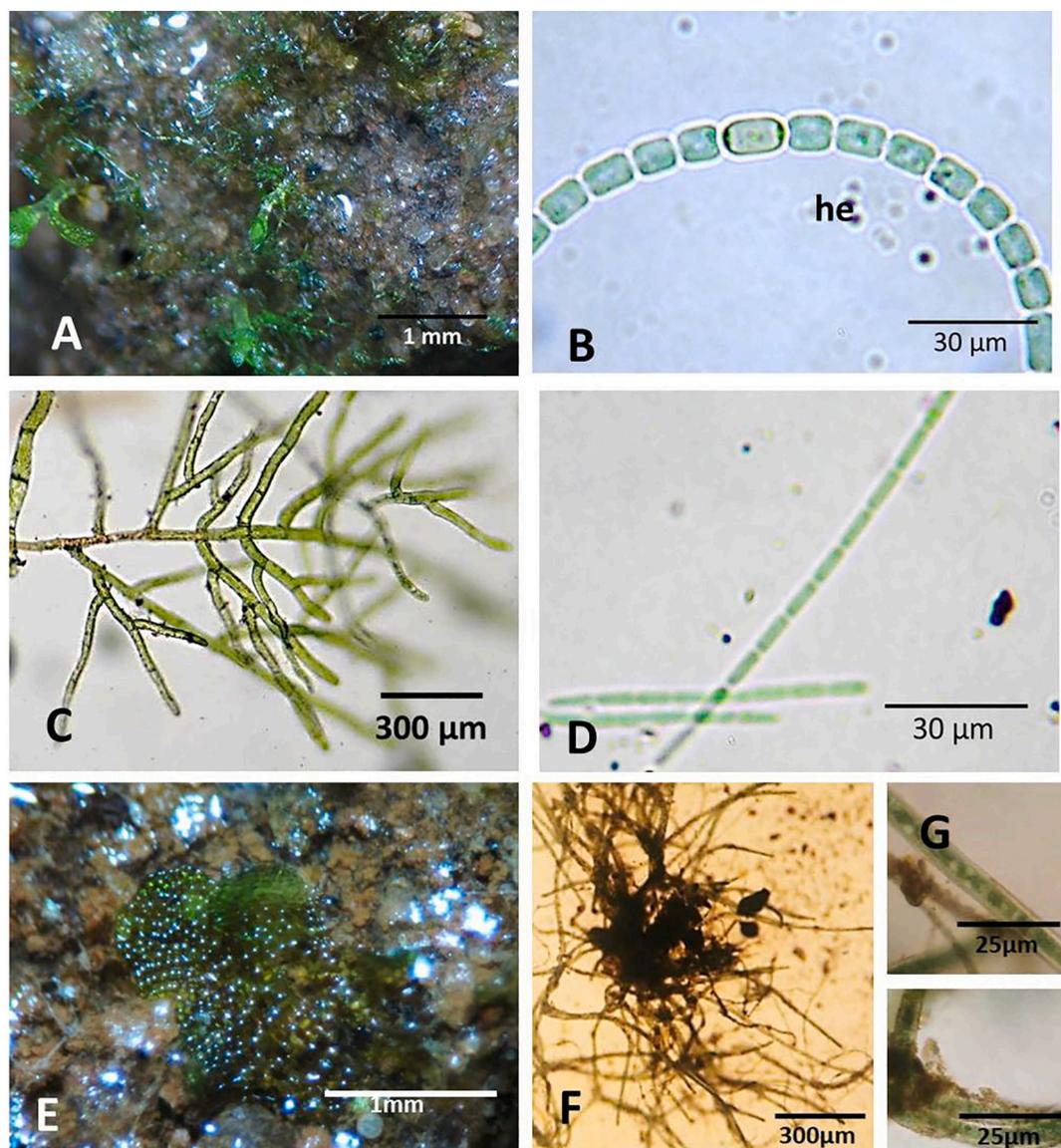


Fig. 3. Photobiotic organisms found in the samples collected on the Carmo riverbank, Barra Longa (MG, Brazil) in site I: bryophyte (A); *Anabaena* sp. (B) and its heterocyst (he); *Trentepohlia* sp. (C); in site II: *Pseudanabaena* sp. (D); and in site III: pteridophyte (E), *Scytonema* sp. (F) and details of the cyanobacteria (G).

immobilized carbon from the soil was being used to form new microbial biomass. Furthermore, the microbial metabolic activity was highly efficient, and the soil microorganisms were released from environmental stress. The iron ore tailing mud caused changes in microbial metabolic activity indicators that may be associated with the organic carbon availability for soil microorganisms. However, natural succession will permit the rapid recolonization of microbial populations when increases in soluble C and nutritional conditions start getting favourable. The presence of photobiotic organisms in the collected samples may be an indicative that the region has the potential to recover. The authors recommend continuous monitoring of the area in order to follow the process of ecological succession of the microbial community. This will aid in a better understanding of the microorganisms' performance in areas where the iron ore tailing mud was brought by the river after the dam failure.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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