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Chemical, mineralogical and physical characteristics of a material accumulated on the river margin from mud flowing from the collapse of the iron ore tailings dam in Bento Rodrigues, Minas Gerais, Brazil

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Abstract The rupture of an itabirito mining tailings dam at the headwaters of the Doce River Basin (Minas Gerais and Espírito Santo, Brazil) caused the greatest environmental catastrophe of the planet Earth related to this activity. The tailings were deposited both in the bottom and on the riverside terrace of the rivers, causing silting and deep changes in the water quality and burial of the main agricultural areas of this basin. For these areas to return to pre-disaster levels, it is imperative that the material deposited on the river terraces be thoroughly characterized. The objective of this work was to characterize the material from the rupture of the Fundão dam, deposited on the river terrace of the Carmo River, a tributary of the Doce River. The material was collected at a depth of 0 to 30 cm from a tail layer about 3 meters thick deposited on the river terrace on the right bank of the Carmo River in the urban area of Barra Longa, Minas Gerais. The physical analyses included soil, particle and porosity density, chemical analyses were pH, sorption complex, organic matter, exchangeable Fe, Mn, Cu, Zn, Pb, Cd and Ni, total oxides, and mineralogical analyses were performed by X-ray diffractometer and Mössbauer spectrometry. The reject has high levels of sand and silt and a low clay content. The densities of soil and particles are high, and the porosity is low. The pH is alkaline, the levels of organic matter, plant nutrients and CEC are very low. The exchangeable heavy metals Zn, Cd, Cu, Pb and Ni are very low, and the exchangeable Mn contents of the tailings are high. The predominant total oxides of the tailings are SiO_2 and Fe_2O_3. The most abundant minerals of the tailings are quartz and hematite. The physical, chemical, and mineralogical attributes of mine tailings restrict the restoration of native vegetation or the agricultural use of the river terraces on which it was deposited.

Keywords: Heavy metals, contamination, recovery of degraded areas, agricultural areas.

Introduction

The rupture, on 5 November, 2015, of the dam containing an aqueous suspension of mining tailings from iron ores of an itabirite rock (a metamorphic rock used for the exploitation of iron ore) deposit caused the largest documented environmental catastrophe of the planet Earth related to this activity. The disruption of the Fundão dam, located at the headwaters of the Gualaxo do Norte River, a tributary of the upper reaches of the Doce River, released more than 60 million m^3 of sandy and clayey rejects as muds. By moving downstream, the mud destroyed two districts of the municipality of Mariana, state of Minas Gerais, namely Bento Rodrigues and Paracatu de Baixo, impacted thousands of hectares of agricultural areas, and has left several cities in the states of Minas Gerais and Espírito Santo virtually desvated ever since. The rejects carried by the water flux reached the mouth of the Doce River and dramatically affected the fauna and flora, as in mangroves, of its ecosystem. More than one million people were affected and nineteen persons died. The overall losses were estimated as being about five billion dollars (Milanez and Losekann 2016).

The mineral rejects were deposited both in the bottom and on the riverside terrace of the Gualaxo do Norte, Carmo and Doce rivers, causing siltation and profound changes in the water quality and covered the main agricultural areas of the valleys, in irreversible environmental impacts. In the first 90 km, following the Gualaxo do Norte and Carmo River flows, and along with a small part of the Doce River, the tailing streams descended rapidly, and materials of varying chemical composition and granulometry were deposited both on the bottom and on the river banks. A flood was generated by mudflows and severely affected the fluvial plain with deposits of various thicknesses (Milanez and Losekann 2016), as in the municipality of Barra Longa, destroying much of its agriculture-based economy. Upon reaching the dam of the Risoleta Neves Hydroelectric Plant, the coarser material was barred; only materials of predominantly finer granulometry were and are transported downstream from that point.

Agriculture and livestock are two of the main economic activities of populations in the Doce River valley. The riverine areas are the most suitable and have been the most intensely used for farming and pasture. Any effort to gradually return to pre-disaster quality levels necessarily imply a better knowledge and require thorough investigations regarding the mineralogical nature of the deposited materials on the river terraces.

The main objective of this work was to characterize, with regard to the chemical, physical and mineralogical characteristics, the materials deposited after the rupture of the Fundão dam that were deposited on the river terrace of the Carmo River, specifically those from near the municipality of Barra Longa, Minas Gerais, Brazil.

Materials and methods

Sample collection point description

The material from the rupture of the Fundão dam was collected on November 20, 2015, at a depth between 0 to 30 cm from the surface of a mud mantle about 3 m thick deposited on the right margin terrace of the Carmo River, in the urban area of the municipality of Barra Longa, in the state of Minas Gerais. The sampling site is located at 385 m above sea level, at UTM coordinates (23K) 704623 South and 7756199 West, in an anthropic area previously used for the raising of domestic animals and fruit trees.The geographical location of the sampling point, along with those of the dams of mining tailings, the city of Barra Longa and the county of Bento Rodrigues, before and after the rupture of the Fundão dam are shown in Figure 1.

Physical analyses

All granulometric analyses (to assess the proportions of sand, silt and clay) were made in the triplicate: clay was determined by the pipette method, whereas the sand content was obtained by sieving. The soil density (Ds) was determined by the volumetric ring method, where as the particle density (Dp) was assessed by the volumetric flask method, according to the method described by Embrapa (2011). The total pore volume (VTP) was calculated by **Erro! Fonte de referência não encontrada.**.

$$\mathrm{VTP} \ = \ 1-\left(\frac{\mathrm{D_s}}{\mathrm{D_p}}\right) x \ 100$$

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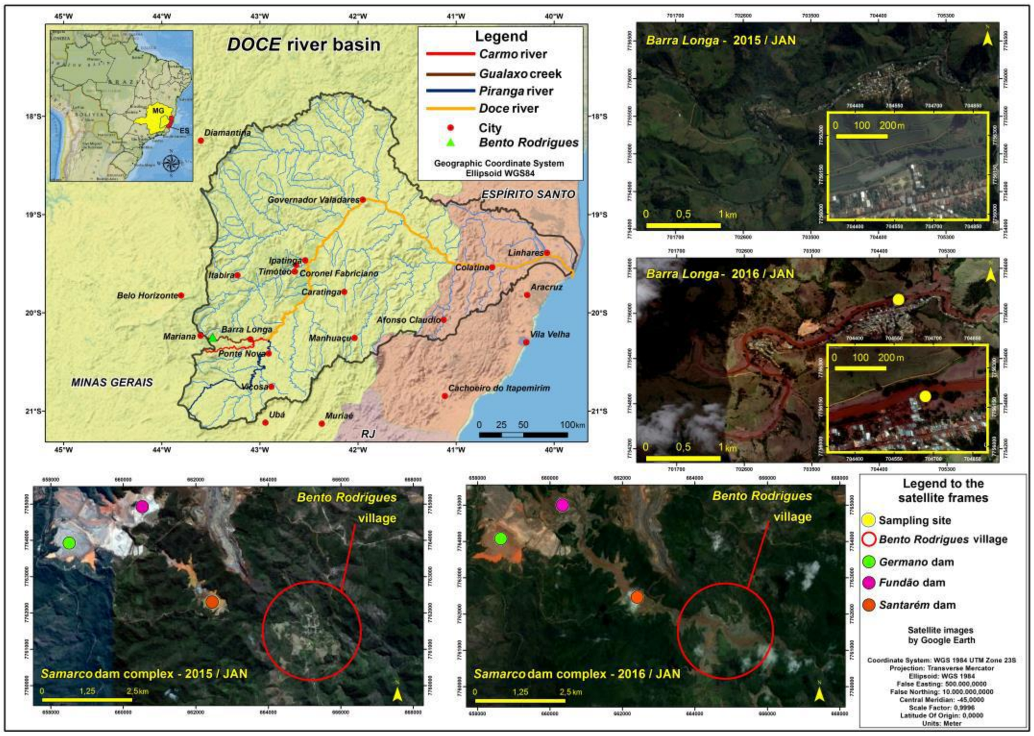

Figure 1: Upper left: the geographical context of the Doce River basin. The two pictures at the right show the sampling site in Barra Longa (just before the dam rupture, in 2015, and several months after, in 2016). The bottom pictures show the Samarco dam complex and the Bento Rodrigues county (also, in 2015 and in 2016).

Chemical analyses

Routine chemical analyses for soils and sediments (pH in water, organic matter, available P, Ca^{2+}, Mg^{2+}, K^+, Na^+, Al^{3+}, H + Al) and for the Fe, Zn, Mn, Cd, Pb and Ni contents were performed in triplicate, according to methods described in Embrapa (2011). The sum of bases (SB), the effective cation exchange capacity (t), the cation exchange capacity at pH 7 (T), the saturation by base (V) and the saturation by aluminum (m) were obtained.

The determination of the C, H and N contents in the sample was performed in triplicate, with a LECO TruSpec Micro elemental analyzer equipped with an infrared detector. The samples were burned at 1075 °C in a quartz tube to quantify the C, H, and N contents.

The X-ray fluorescence analysis of the sample was performed with a Shimadzu EDX-720 energy dispersive X-ray fluorescence spectrometer, with a rhodium tube and silicon-lithium detector. Data were collected under vacuum of 40 Pa with a 10-mm collimator.

Mineralogical analyses

The powder X-ray diffraction (XRD) pattern for the sample was collected in a Rigaku model D/Max Ultima Plus diffractometer set to a current of 30 mA and a voltage of 40 kV, with CuKα ($\lambda = 1.541838$ Å) radiation, at a scan rate of $1° \ 2\theta \ min^{-1}$, from 4º to 100º 2θ. Silicon was used as the external standard, according to the method proposed by Theisen and Harward (1962).

Mössbauer spectroscopy

The Mössbauer spectra were collected at room temperature (~298 K), 80 K and 25 K in a conventional transmission spectrometer at a constant acceleration configuration with a ^{57}Co/Rh gamma-ray source and nominal activity of about ~25 mCi. Doppler velocities were approximately ±11.6 mm s^{-1}. Mössbauer isomer shifts are quoted relative to an α-Fe foil at room temperature. The experimental data were fitted with Lorentzian functions by least-square fitting with a NORMOS™-90 computer program. Magnetization measurements were performed with a vibrating sample magnetometer (Lake Shore 7404; with a noise base of 5×10^{-5} emu, a time constant of 100 ms at room temperature, and a maximum magnetic field of 2 T).

Results and discussion

The mine reject was found to have a sandy loam size distribution (Embrapa 2013) and low clay content. Shaefer *et al.* (2016) found similar granulometric compositions in samples collected at several points close to the sampling of this study. The bulk density (Ds) of the tailings was relatively high, as was the particle density (Dp). These characteristics explain their very low porosity (Table 1). Shaefer *et al.* (2016) obtained Ds values between 0.94 and 2.38 g cm^{-3} and mean Dp values between 2.75 and 2.80 g cm^{-3}. These reported data corroborate the results of this study. All these attributes make it difficult to re-establish the vegetation covering on the terraces, which are the areas with the best agricultural capacity, being most intensively used for agricultural crops and raising of livestock.

Shaefer *et al.* (2016) also pointed out that the settlement of the tailings progressively led to the hardening of the surface, making it difficult for the seed to germinate and for the roots to penetrate into the mineral stratus. In addition, the slow reestablishment of the vegetation covering in these terraces tends to favor the transport of the deposited waste by the waterways during the annual flood periods, making it a cyclical event.

Sand	Silt	Clay	Ds	Dp	VTP
	_______________%_______________		__________t m^{-3}_________		%
58(2)	36(2)	6(1)	2.12(1)	2.85(8)	25.6

Table 1: Grain size composition, soil density (Ds), particle density (Dp) and total pore volume (VTP) for the sample of mine tailings.The number in parentheses represents the uncertainty with respect to the last significant digit, as obtained from standard deviations of the mean, estimated from the measuments in triplicate.

The mining reject is alkaline and very poor in nutrients, presenting only moderate Ca contents (CFSEMG, 1999). According to Shaefer *et al.* (2016), the high pH value can be due to the use of NaOH in the beneficiation of the ore. The CEC value is very low because of the low clay and organic matter contents (Tables 1 and 2) and the highly oxidized nature of the material (Schaefer et al., 2016). Soils and petroplintites from the region of this study (Quadrilátero Ferrífero) also present a low CEC (Sahefer *et al.* 2015). CHN analysis yielded approximately 0.50 mass% of C, 0.06 mass% of N and 0.07 mass% of H, corroborating the small content of organic matter in the sample (Table 2).

pH (water)	**P**	**K**	**Ca**	**Mg**	**Al**	**H+Al**	**BS**	**CEC**	**m**	**V**	**OM**	**Na**	**Fe**	**Zn**	**Cu**	**Mn**	**Cd**	**Pb**	**Ni**
1:1	**mg dm^{-3}**	**------------------cmol$_c$ dm^{-3}------------**							**------%-----**		**dag kg^{-1}**	**---------------------------mg dm^{-3}-----------------------------**							
8.24	3.58	1.44	1.76	0.02	nd	0.95	1.79	2.74	nd	65.20	0.71	1.67	409.2	1.5	2.4	441.4	nd	1.9	0.9

Table 2: Chemical attributes and contents of metallic elements of the sample of mine tailings. OM: organic matter; BS: sum of bases; CEC: cation exchange capacity at pH 7; V: saturation by bases; m: aluminum saturation; nd: not detected.

The percentages of heavy metals encountered — Cu, Cd, Pb and Ni in the form available for the plant use — are small or not detected, except for Fe and Mn, which are high (Table 2). Shaefer et al. (2016) found mean Fe and Pb levels of 499.2 and 0.41 mg dm^{-3}, respectively, in samples of tailings from the same region, similar to those found in this study (Table 2).

Although Mn and Fe are plant micronutrients, high levels such as those found in the tailings (Table 2) can be toxic to plants (CFSEMG, 1999). The Mn and Fe contents in the waters of the Carmo River and the Doce River, respectively, were greater than the levels allowed for class 2 water (waters that can be destined for human consumption, after conventional treatment to protect aquatic communities and recreation), according to CONAMA Resolution 357 (Milanez and Losekann, 2016). The chemical attributes in the set do not favor the establishment of a vegetal cover.

A predominance of Si and Fe was observed by the X-ray fluorescence analysis (Table 3). Brant Meio Ambiente (2005) found similar Si and Fe values in the material deposited at the Fundão dam, 62.39 and 40.43 mass% (weighted average between clayey tailings and sandy tailings), respectively. Si predominates in both the sand fraction and the silt fraction (Table 1), which are the main wastes from the itabirite mine. The high Fe contents, both in oxide form and in the exchangeable form (Tables 3 and 2), indicate that the ore beneficiation process was not very efficient.

	Oxides content/mass%
SiO_2	53.2(1)
Fe_2O_3	37.33(3)
Al_2O_3	8.45(5)
SO_3	0.47(1)
K_2O	0.250(1)
MnO	0.127(1)
CaO	0.083(1)
P_2O_5	0.07(2)
CuO	0.017(1)
ZnO	0.002(1)
NiO	0.001(2)

Table 3: Chemical composition of the sample as determined by X-ray fluorescence spectroscopy.The numbers in parentheses are uncertainties with respect to the last significant digit, as provided by the spectrometer.

Al was not detected in the exchangeable form, but its contents were significant in the oxide form (Tables 2 and 3). Brant Meio Ambiente (2005) also found significant levels of total Al. The Mn concentrations are low in the oxide form (Table 3) and similar to those found by Brant Meio Ambiente (2005), but they are high in the exchangeable form (Table 2). The Cu, Zn, Pb and Ni concentrations are low both in the oxide form and in the exchangeable form (Tables 3 and 2). The levels of these heavy metals in the waters of the Carmo River and the Doce River were lower than the levels allowed for class 2 water, according to resolution CONAMA 357 (Milanez and Losekann 2016).

The results of the mineralogical analysis (Figure 2) are in line with the results from the physical and chemical analyses. Characteristic reflections of the crystallographic phases for quartz SiO_2 (JCPDS card # 46-1045), hematite, αFe_2O_3 (JCPDS card # 33-664), goethite, αFeOOH (JCPDS card # 29-713), muscovite, $KAl_2Si_3AlO_{10}(OH)_2$ (JCPDS card # 7-25) and kaolinite, $Al_2Si_2O_5(OH)_4$ (JCPDS card # 58-2001) were observed in the X-ray diffraction pattern. Schaefer et al. (2016) found quartz, goethite, hematite, and kaolinite in the tailings of the Germano and Santarém dams near the Fundão dam.

Si predominates in the sand and silt fractions, which together correspond to 94 mass% of all the material (Table 1). The kaolinite and Muscovite (Figure 2) correspond to minerals present in the silt and clay fractions. All the minerals identified have low CEC, corroborating the results of the chemical analyses (Table 2).

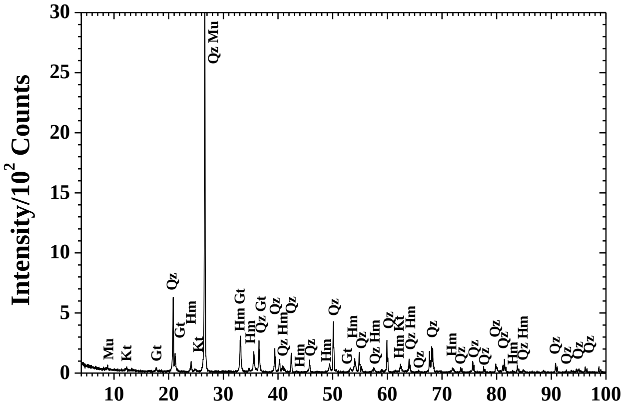

Figure 2: Powder X-ray diffraction pattern of the sample. Qz = quartz, Hm = hematite, Gt = goethite, Mu = muscovite, Kt = kaolinite.

From the Mössbauer measurements (spectra in Figure 3 and corresponding hyperfine data in Table 4), hematite (αFe_2O_3), in greater proportion, and goethite (αFeOOH) were identified, corroborating the results of the mineralogical analysis by X-ray diffraction (Figure 2). The predominant portion of this hematite was found to undergo the Morin transition (characteristic temperature, T_M~ 260 K), with a quadrupole shift from 0.485(2) mm s^{-1} (at 80 K). This fraction is likely to be composed of larger particles of less isomorphically substituted and better crystallized hematite than that not undergoing the Morin transition. In addition to these Fe minerals, a small proportion of maghemite (γFe_2O_3), a magnetic mineral usually associated with hematite and found in highly weathered materials of tropical and subtropical regions, was also identified (Breemen and Buurman, 2002).

Temperature/K	Fe site	δ/mm s^{-1}	2ε/mm s^{-1}	Γ/mm s^{-1}	B_{hf}/T	*AR*/%
80	αFe_2O_3	0.485(2)	0.385(5)	$0.354(9)^{**}$	53.75(2)	51(1)
	αFe_2O_3	0.48(1)	-0.18^{*}	$0.354(9)^{**}$	53.2(1)	9(1)
	γFe_2O_3	0.59(3)	0^{*}	$0.354(9)^{**}$	48.9(2)	4(3)
	$\alpha FeOOH$	0.435(7)	-0.22(4)	0.49(3)	49.4(5)	36(5)

Table 4:Mössbauer parameters corresponding to spectrum recorded at 80 K. δ = isomer shift relative to αFe; 2ε = quadrupole shift; Γ = line width; Bhf = magnetic hyperfine field; RA = relative subspectral area. (*) Fixed parameter during the fitting procedure. (**) Constrained parameter during least-squares fitting convergence. The number in parentheses are uncertainties over the last significant digit, as was estimated from the least squares fitting algorithm.

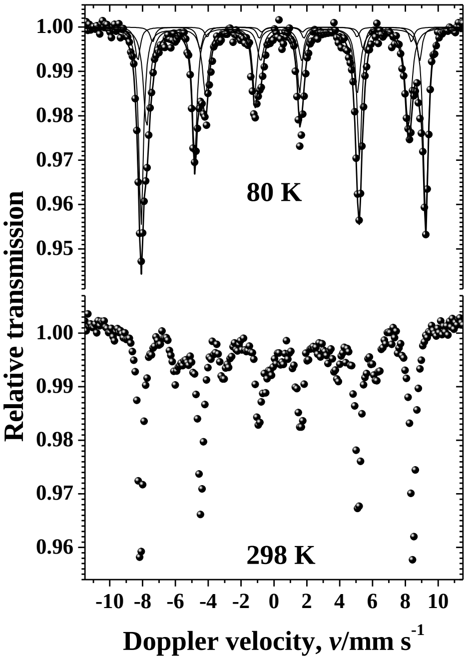

Figure 3:^{57}Fe Mössbauer spectra for the sample registered at 298 K and 80 K.

Figure 4 shows the magnetization hysteresis curve for the mine tailings sample. The saturation magnetization, Ms, is of 0.66(3) emu g-1; the remnant magnetization, Mr, is of 0.11(3) emu g-1 and the coercivity, Hc, is of 238.38(4) Oe.

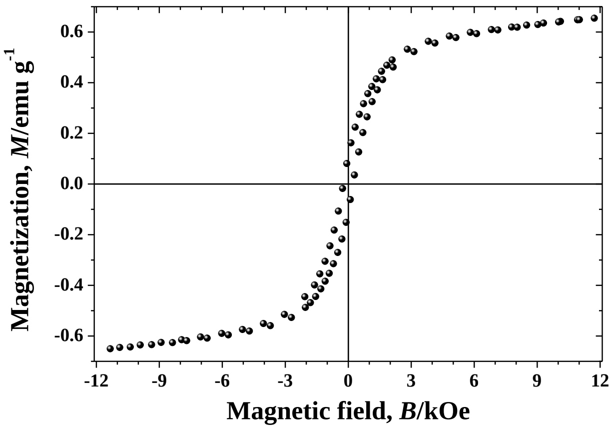

Figure 4: Magnetization curve for the sample at room temperature.

Both the physical, chemical and mineralogical attributes of mine tailings deposited on river terraces of the Gualaxo do Norte River restrict the restoration of native vegetation cover or agricultural activities. The main limiting factors are the high density and low porosity and the high exchangeable Mn contents. The exposed soil favors erosion on these terraces, making it even more difficult to restore vegetation and causing the silting of the river bed and contamination of its waters with Mn. The erosion-assortion-contamination cycle will be repeated annually, especially during rainy periods, to further degrade natural resources.

To minimize this degradation, some measures were experimentally tested by a team from the Federal University of Viçosa(Shaefer *et al.* 2016). The characterization of the material with a view to its use as a substrate for plants, including those to be used in the restoration of riparian forests, or its use as a raw material for civil construction are among the contributions of soil science proposed by Viana and Costa (2016) for the recovery of areas affected by the disaster. This is the main contribution of this work.

Conclusions

- The reject has high levels of sand and silt and a small clay content. Its densities of soil and particles are high, and the porosity is low.
- The pH is alkaline ante the contents of organic matter; plant nutrients and CEC are very low.
- The concentrations of exchangeable heavy metals Zn, Cd, Cu, Pb and Ni are very low and the exchangeable Mn contents of the tailings are large.
- The predominant total oxides of the tailings are SiO_2 and Fe_2O_3.
- The most abundant minerals of the tailings are quartz and hematite.
- The physical, chemical and mineralogical attributes of mine tailings restrict the restoration of native vegetation or the agricultural use of the river terraces on which it was deposited.

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Características químicas, mineralógicas e físicas do material acumulado em terraços fluviais, originado do fluxo de lama proveniente do rompimento de barragem de rejeitos de mineração de ferro em Bento Rodrigues, Minas Gerais, Brasil

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Resumo O rompimento de uma barragem de rejeitos de mineração de itabirito na cabeceira da Bacia do Rio Doce (Minas Gerais e Espírito Santo, Brasil) provocou a maior catástrofe ambiental do Planeta Terra, relacionada a esta atividade. Os rejeitos foram depositados tanto no fundo como no terraço fluvial dos rios provocando o assoreamento e mudanças profundas na qualidade das águas e soterramento das principais áreas agrícolas desta bacia. Para que elas retornem aos níveis anteriores ao desastre é imprescindível que o material depositado nos terraços fluviais seja minuciosamente caracterizado. O objetivo deste trabalho foi caracterizar química, física e mineralogicamente o material proveniente do rompimento da barragem do Fundão, depositado no terraço fluvial do Rio do Carmo, afluente do Rio Doce. O material foi coletado na profundidade de 0 a 30 cm de uma camada de rejeito de cerca de 3 metros de espessura, depositado no terraço fluvial da margem direita do Rio do Carmo, na área urbana de Barra Longa, em Minas Gerais. Foram realizadas análises físicas (granulometria, densidade do solo e de partículas e porosidade), químicas (pH; complexo sortivo; matéria orgânica; Fe, Mn, Cu, Zn, Pb, Cd e Ni trocáveis; óxidos totais) e mineralógicas (difratometria de raios X e espectrometria Mössbauer). O rejeito apresenta elevados teores de areia e de silte e baixo teor de argila. Suas densidades do solo e de partículas são elevadas e a porosidade é baixa. O pH é alcalino, os teores de matéria orgânica, de nutrientes de plantas e a CTC são muito baixos. Os teores dos metais pesados Zn, Cd, Cu, Pb e Ni trocáveis são muito baixos e os teores de Mn trocável do rejeito é elevado. Os óxidos totais predominantes do rejeito são o SiO_2 e o Fe_2O_3. Os minerais mais abundantes do rejeito são o quartzo e a hematita. Os atributos físicos, químicos e mineralógicos do rejeito da mineração restringem o restabelecimento da vegetação nativa ou o uso agrícola dos terraços fluviais nos quais foi depositado.

Palavras-Chave:metais pesados, contaminação, recuperação de áreas degradadas, áreas agrícolas.

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