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Mechanical and environmental performance of polymer stabilized iron ore tailings

Andressa de Araujo Carneiro^{1,2,#} (D), Michéle Dal Toé Casagrande¹ (D)

Note

Keywords

Iron mine tailings Polymer Stabilization Strength parameters

Abstract

The huge amounts of residues generated by the mining industry have caused a number of environmental problems, not only in Brazil but worldwide. During the extraction of iron ore, the process of beneficiation produces a considerable volume of tailings, making the disposal of these materials a challenge for the environmental and geotechnical engineering. In this context, numerous researchers have been looking for alternative solutions for the disposal and use of these materials, focusing on stabilization, improvement or reinforcement in geotechnical works. Polymers are currently being increasingly used as a stabilizer for sandy soils, due to their stable chemical property and shorter curing time, compared to traditional stabilizers, such as cement, lime, fly ash and bituminous materials. This research presents the study of improvement techniques of iron ore tailings by insertion of a polymer. The mechanical and environmental properties of the composite are analyzed, aiming at the application in geotechnical structures, such as landfills and slopes. The results show that the use of this composite is interesting from the technical, economic, structural and environmental safety point of view.

1. Introduction

Huge amounts of residues generated by the mining industry have caused a number of environmental problems, not only in Brazil but worldwide. During extraction of iron ore, process of beneficiation produces a considerable volume of tailings, making the disposal of these materials a challenge for the environmental and geotechnical engineering. However, the characteristics of mine tailings limit direct use in structures, due to structural and environmental restrictions. In recent years, the implementation of alternative materials associated with tailings has guided several geotechnical works, for stabilization, improvement or reinforcement, making it a topic of great interest.

Festugato et al. (2015), Consoli et al. (2017), Sotomayor (2018) among others studied efficient methods of reinforcing ore tailings by creating a composite that meets engineering requirements. These authors found that the insertion of fibers in the tailings brings benefits to the strength parameters of the matrix.

On the other hand, the use of polymers in the formation of composites with tailings is an interesting stabilization alternative. The references show that, when correctly applied and using an adequate dosage, the polymer becomes a stabilizing agent in non-paved roads, landfills, protection and stability of slopes, erosion in paving layers, due the agglutination of grains and improving the stability of the soil matrix.

Mirzababaei et al. (2017) analyzed the effect of polymers using a non-confined compressive strength test in a sample of clay soil. Xing et al. (2018) and Liu et al. (2018, 2019) investigated the behavior of sand soil adding polymer in wet and dry conditions. Barreto et al. (2018) analyzed the improvement of sand soil with use of butadiene and modified styrene copolymer (XSBR). Okonta (2019) analyzed application of acrylic polymeric solution at different curing times, percentages and temperatures for a soil characteristic of the South African region. Lee et al. (2019) investigated the feasibility of applying biopolymer (xanthan gun), to stabilize local soil, for construction of the road in Sri Lanka, through the unconfined compression test. Silva (2020) conducted experimental studies a sand soil improved with polymeric solution as a stabilizing agent. Li et al. (2020) investigated the shear behavior of the polymer-bentonite interface.

The above and recent research results show that polymeric solutions can effectively improve the strength, de-

²Departamento de Recursos Hídricos, Geotecnia e Saneamento Ambiental, Universidade Federal do Piauí, Teresina, PI, Brazil.

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[&]quot;Corresponding author. E-mail address: andressa.deac@gmail.com.

¹Departamento de Engenharia Civil e Ambiental, Universidade de Brasília, Brasília, DF, Brazil.

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spite physical property of composite is rarely studied. As the polymer is a chemical material, it can cause changes in the matrix. That is why studying the mechanical behavior of the composite, through laboratory tests, is so important to understand and apply these new geotechnical materials in structures.

In this context, this research presents the study of improvement techniques of iron ore tailings by insertion of a polymer. The mechanical properties of the composite are analyzed, aiming at the application in geotechnical structures, such as landfills and slopes. The results show that the use of this composite is interesting from the technical, economic, structural, and environmental safety point of view.

2. Material and methods

The iron ore tailings analyzed are sourced in the raising dikes, compacted by the passage of equipment over a dam. Samples deformed, presenting moisture of 14 %, were collected in different positions, 50 m away from the central axis.

The polymer used is an acrylic copolymer of organic styrene, obtained by random polymerization reactions through an anionic aqueous emulsion presenting density of $0.98-1.04 \text{ g/cm}^3$ and pH of 8.0-9.0.

2.1 Geotechnical characterization

Physical characterization tests were performed by Sotomayor (2018) to obtain index properties such as natural specific weight and grain density. The maximum dry weight and optimal moisture were obtained through the compaction test with Proctor Normal energy in samples with and without polymer to verify the influence of the additive.

In all tests, the mixing of the polymer with the tailings was done in the same way. A homogeneous solution containing 25 % of polymer and 75 % of water (using the proportion 1:3 in volume) was mixed with the tailing and molded into samples. The abbreviation T and TP was used to refer, respectively, to tailing and tailing -polymer.

After obtaining the maximum dry weight and optimal moisture, the specimens used to perform the shear test were molded in a metal mold square of transversal section, with sides of 60 mm and height of approximately 25 mm. The quantity of material was calculated according to the physical parameters and volume of the mold.

It must be noted that by the moment when occurs the mixture of tailing and polymeric solution a pasty consistency is verified. Then, the composite stiffens in contact with air and over the days of curing. The curing of the samples occurred in room temperature, approximately 26 $^{\circ}$ C, which was done to analyze the mechanical behavior of the tailings and the influence of the additive over time, considering the same conditions.

After conventional direct shearing test, a sheared plane with polished surface test was performed. Last one was carried out by a "polishing" using a thin line of resistant nylon through the sheared surface, between both boxes, in the direction of the shearing plane. After separating the boxes the polishing of the surfaces was done, always in the direction of the shearing plane. The complete test was performed again, since the stage of densification until shearing. The samples were densified during the necessary period for the occurrence of 100 % of the primary densification, being applied containment stresses of 50 kPa, 100 kPa, 200 kPa and 400 kPa for the construction of the Mohr-Coulomb shear strength envelope. The maximum horizontal displacement tested was 8 mm, in function of the equipment characteristics.

The curing periods analyzed were of 0, 7, 14 and 28 days for both tests, conventional direct shearing and sheared plane with polished surface. The analysis of the material resistance was performed using also the conventional direct shearing and sheared plane with polished surface.

2.2 Complementary tests

For a better understanding of the composite behavior, complementary tests were performed, such as hydraulic, chemical-environmental and mineralogical tests.

Test of permeability was performed using the constant and variable permeameter for the pure tailing and composite, respectively. The water absorption test, according the Brazilian Standard ABNT NBR 13555 (ABNT, 2012), was also performed to verify the reduction of voids in the tailing after the insertion of the polymer.

The total suction was determined by the use of a psychrometer (WP4C), according to ASTM D6836-16 (ASTM, 2016). The test was performed with two daily readings, at 9 a.m. and another reading at 6 p.m. The readings occurred with the composite and pure tailing, both with 10 % of moisture content. The objective of the test was not the obtainment of the material characteristic curve, but understanding, over time, the increase of suction as the curing of the composite advances.

To verify if the polymer had leaching potential, it was performed leaching test according to the Brazilian Standard ABNT NBR 10005 (ABNT, 2004). X-ray Diffraction Analyses (XRD) were performed with the Rigaku Ultima IV equipment. Energy dispersive spectroscopy (EDS) and scanning electron microscopy (SEM) were performed using the JEOL JSM 7100F Scanning Electron Microscope with acceleration voltage of 30 KV, 3 nm resolution, up to 300,000 X with EDS X-ray microanalysis system. Before being tested, the samples were submitted to the metallization process by high vacuum evaporation. This procedure consists of depositing an ultrafine coating of electrically conductive material on the samples, in which case gold was used. The tests were performed for the microstructural characterization of the tailing-polymer composite with 28 days of curing and pure tailing.

3. Results

3.1 Geotechnical characterization

The specific mass of mine tailings is generally greater than most natural soils, since in the tailings it is found a concentration of oxides whose specific mass is greater than that of the dominant minerals in natural soils, as the quartz and kaolinite. The particles of iron present density of solids of approximately 5.25 g/cm³, while quartz grains are characterized by presenting a density of solids of about 2.65 g/cm³.

Figure 1 presents the particle-size distribution of the iron ore tailings used in the research. The tailing of iron ore studied has density of grains of 2.89 g/cm³ and it is classified as silt sand according to Unified Soil Classification System (USCS).

Figure 2 presents the compaction curves of the studied materials. After polymer addition, the unit weight increases and the moisture content decreases. Therefore, the polymer works as a particle binder, reducing the space of voids.

Figure 3 presents the curves of shear stress by horizontal displacement of pure tailing and composites studied. The applied stresses were of 50 kPa, 100 kPa, 200 kPa and 400 kPa to tailing, T, and tailing -polymer, TP, in respectively separated with curing periods of 0, 7, 14 and 28 days.

After the polymer insertion, a gain of shearing strength of the iron ore tailing was observed for all mixtures and stresses tested. For the samples with 0 days of curing, there was no significant decrease in the post-peak, tending to a curve stabilization indicating the strength after shear. Considering the samples submitted to different curing periods, it was verified an increase of resistance to shearing for all composites, observing a more significant peak for the samples with longer period of curing and for high stresses.



Figure 2. Compaction curves.

Tests performed on shearing plan with polished surface were performed after the conventional test. The results for each period of curing of 0, 7, 14 and 28 days are presented in Figure 4.

Considering the tests with shearing plan polished surface, there was not a peak on the curves, since the samples were already sheared. A greater resistance for the composites submitted to greater normal stress was noticed; however, there was not a significant change in strength over the curing time. In other words, after the shearing, there is no strength reduction and the change in the composite only occurred during the initial rupture, indicating the inexistence of a greater instability in the samples tested with polished surface.

Figure 5 shows the curves of vertical displacement vs. horizontal displacement for composites tailing-polymer (TP) with 0 and 28 days of curing at the conventional shear test and at shearing plan polished surface test.



Figure 1. Granulometric distribution of the iron ore tailings.



Figure 3. Shear stress-horizontal displacement responses: (a) 0 days; (b) 7 days; (c) 14 days and (d) 28 days.

Figure 5a indicates that the behavior of the composite with no curing showed an expansion, mainly, for higher normal stress. When cured, the composites tended to be compressed, but the tendency is suppressed by 4 mm. Afterwards, there is a stabilization until the end of the test for all normal vertical stresses. The curves in Figure 5b show a similar trend for all normal vertical stresses showing some homogeneity in the behavior of the composite with a polished surface.

Figure 6 presents the shear strength envelopes obtained at peak and residual stresses during the direct shearing test of the tailing -polymer, TP. The residual stresses refer to the horizontal displacement of 7 mm. An increase in the shear strength parameters for the composites was observed according to the curing period. Considering the composite without period of curing, no significant change was not found. This was expected given that by adding the polymer the sample had a higher moisture content, therefore reducing its resistance.

For the peak strength, it is observed an increase on the values of the effective angle of friction and apparent cohesion, mainly for the greatest curing periods. The apparent cohesion increased considerably over the curing period, which was expected when the polymer was added.

The friction angle and cohesive intercept obtained by Silva (2020) using soil-polymer composite with the same



Figure 4. Shear stress-horizontal displacement responses for shearing plan with polished surface: (a) 0 days; (b) 7 days; (c) 14 days e (d) 28 days.



Figure 5. Curves vertical displacement vs. horizontal displacement: (a) conventional test and (b) polished surface.



Figure 6. The Mohr failure line (a) peak strength and (b) residual strength.

proportion of the present research increased, respectively, from 31.9° to 32.9° and from 4.05 kPa up to 169.67 kPa, with 30 days of curing. The values are similar to those found in the current study, which indicates similar performance of the polymer when added to the soil or iron ore tailings.

Considering the residual strength, there was an increase on the friction angle and apparent cohesion, showing that the polymer insertion was positive for the samples submitted to the conventional shearing test.

The shear strength envelopes obtained with the direct shearing tests on tailing -polymer samples, TP, with polished surface are presented in Figure 7. As reported previously, the samples were already sheared, thus there was not discrepancy in the values of the parameters of resistance for the peak and residual strength.

Regarding the samples with polished surface, there was a reduction on the effective angle of friction, since the samples were already sheared, however, the apparent cohesion increases. Thus, even with previous shearing, the grains remained with a certain adherence, hence it is observed an improvement of one shear strength parameter.

3.2 Complementary tests

The permeability coefficient is a necessary parameter to be analyzed, mainly regarding the improvement and stabilization of tailing. The values of the permeability coefficient of the tailing with 0 days and of the tailing-polymer composite with 28 days of curing were 2.96×10^{-5} and 6.08×10^{-7} m/s, respectively. The polymer significantly reduced the iron ore tailing permeability. Therefore, the action of the polymer application in the tailing permeability.

Due the significant reduction of permeability, it was verified the absorption rate of water of the composite with 28 days of curing. The temperature of the test was main-



Figure 7. The Mohr failure line in polished.

tained at 25 °C and an absorption rate of 18.37 % in the composite was observed, which occurs because the polymer stiffen the material, reducing the water percolation.

The psychrometer test (WP4C) was conducted to check the hypothesis that the gain of shear strength is only associated to the increase of suction over the curing period. In day 0, with no curing of the tailing-polymer composite, the suction was of 95.37 MPa. The day 0 for the tailing added with water resulted in a value of 67.84 MPa. There was observed a stabilization of the values of total suction, what is seen on day 28 when the tailing-polymer composite resulted in a value of 98.53 MPa. The final measurement for the pure tailing occurred on day 4 of curing, until near total evaporation of water and the last possible reading obtained a value of 92.5 MPa. The composite with short pe-

riod of curing already presented stabilization of the values of suction, while the tailing with water had greater humidity and thus lower value of suction.

It is possible to affirm that the curing outdoor would cause the shear strength to increase, since the moisture content is decreases and, thus, the suction increases. However, it was proved that the addition of polymer acted more intensely on the increase of shear strength, given that there was no significant change of the composite tailing-polymer suction with increase of the curing period.

Any contamination that may occurs will be through process of infiltration. The water when initially touches the composite infiltrates it and may cause contamination. Such process may be called leaching. The water of the tailing-polymer composite with 28 days of curing was collected and chemically analyzed. Table1 lists the chemical elements in the samples analyzed.

Evaluating the data of leachate, through the resolution CONAMA 420 (CONAMA, 2009), which regulates the maximum values allowed and advisors for soil and groundwater, the maximum values allowed (MVA) for dissolved copper in groundwater is of 2 mg/L and for tailing-polymer composite is less than 0.1 mg/L.

Another element described in the norm is aluminum, its MVA being 3.5 mg/L, the value of the composite is inferior than 1 (0.0018 mg/L), according to the resolution.

The last element cited in the resolution is manganese, whose MVA is 0.4 mg/L, thus there is no coherence between that and the value found for the composite, since it was found in larger amounts. However, the manganese MVA may pose risk to human health when consumed, not to direct contamination of natural elements.

Considering the leachate as an effluent of a polluting source, the Section II Article 16 of the resolution determines the conditions and procedures for the directly discharge of effluents from any polluting source at the receptor body. Thus, according to the resolution, all elements from both leachates are possible to be directly discharged to the hydric body. The MVA of all analyzed elements were within the values specified by the regulation.

Considering the application of polymer for the tailing stabilization, even when the polymer is applied to an area of environmental preservation, there would not be contamination of water bodies.

Table 1. Values of leaching of the composite tailing-polymer.

Element	Values
pH	8.94
Total nitrogen (mg/L)	1.98
Iron (mg/L)	0.1554
Copper (mg/L)	< 0.1000
Manganese (mg/L)	0.7505
Aluminum (mg/L)	< 0.0018

X-ray Diffraction (XRD) analysis was conducted to evaluate the possibility any geological alteration of the soil caused by the polymer addition. Figure 8 presents the diffractogram referring to the XRD test of the pure tailing and tailing-polymer composite with 28 days of curing. Figure 9 presents the microscopic images of pure tailing and tailing-polymer composite.

With the use of phase analysis via XRD one can note that the iron ore tailing studied is basically composed by minerals of quartz (SiO₂), goethite (FeO(OH)), hematite (Fe₂O₃), kaolinite (Al₂Si₂O₅(OH)₄)), and other minerals in smaller quantities, independently of the addition of polymer (Figure 10).

An increase of the hematite proportion is observed in the diffractogram of the composite. It is worth mentioning that hematite is known by its thin texture and as a cementing agent for the formation of aggregate. It is considered that the increase of hematite occurs due the presence of the polymer in the matrix, in nodular format and also as a cementing agent for the grains, which presents properties similar to hematite.

Using the optical microscopy, it is verified that the grains of iron ore tailing are looser and it is identified the light and dark grains, probably minerals of quartz and hematite, as indicated in the test XRD. In Figure 9 is possible



Figure 8. Test of XRD (a) pure tailing and (b) tailing-polymer composite.



Figure 9. Images obtained by the microscopy increase 40 x: (a) pure tailing and (b) tailing-polymer composite.

to respectively verify the pure tailing and tailing-polymer composite with 28 days of curing.

Considering the images obtained by the microscopy, it is possible to note the bond of the tailing grains when the polymer is added, a brighter appearance may also be noted on the grains, similar to a glue or pellicle that envelopes the grains.

Figure 10 shows the main chemical elements found in the pure tailing and in the tailing-polymer composite with 28 days of curing, using the EDS test.

The tests of EDS were performed to confirm what was found by the XRD test. The considerable difference is that the XRD performed show chemical elements as minerals, to geologically classify them, while the EDS scans constitutive



Figure 10. EDS (a) pure tailing and (b) tailing-polymer composite.

chemical elements. The tests of EDS detected chemical elements of silicon (Si), Iron (Fe), Carbon (C) and Oxygen (O). With the addition of polymer, an even greater increase in these chemical elements and a reduction in silicon was observed. The letter K is not related to the chemical element, but with the manner that these chemical elements were identified by the method. Thus, although the data interpretation, both samples exhibit similar chemical elements.

Obtained with the use of using scanning electron microscopy (SEM) for the microscopical characterization, Figure 11 exhibits the distribution of voids in the iron ore tailing studied in its pure form.

It is observed in Figure 11a that the grains are loosen and with a number of sizes. It is not observed an element that bonds the grains, the smaller grains are close to the bigger ones, as if they occupy the void spaces (Figure 11a). The existing voids and the grain surface are well seen in Figure 11b.

In Figure 12, using the analysis from the scanning electron microscopy for the microscopical characteriza-

tion, it is possible to observe in the iron ore tailing studied the voids distribution after the insertion of the polymer solution and 28 days of curing.

Based on Figure 12, it is possible to verify that, after the addition of polymeric solution insertion, a greater bond occurred between the grains. Figure 12b shows that, in certain grains, their boundaries cannot be seen due to the bond between the particles. It is considered that these bonds and meniscus arise from the increment of polymer, and over the curing period it externs the grains bonding them and causing the cementation of the particles.

4. Conclusions

This paper emphasized the main results obtained through tests of direct shearing using polymer stabilized iron ore tailings for application in geotechnical works such as landfills and slopes. For the mechanic tests, a mix of the tailing a solution containing 25 % of polymer and 75 % of water was evaluated. Chemical and mineralogical tests



Figure 11. SEM (a) pure tailing grains increase 110 x e (b) increased grain surface 370 x.



Figure 12. SEM (a) tailing-polymer composite grains increase e 80 x (b) tailing-polymer composite grains increase 550 x.

were performed to better understand the behavior of the studied material.

The analysis allowed a better understanding of the mechanical behavior of the tailings and the influence of the additive over time. However, more research is necessary for a further comprehension of the behavior of this composite.

According to the research performed, regarding the mechanical and environmental behavior of the iron ore tailing composite with polymer insertion, it can be concluded that:

- The addition of polymer to the iron ore tailing is viable given the improvement of the shear strength as the curing period of the composites increase. The composite microstructure shows that there was cementation between the grains, by polymer addition, forming a film on the grains and bonding them;
- A reduction of permeability was verified for the tailing-polymer composite when compared to the pure tailing due the fact that the polymeric solution fills the voids between the grains;
- The resistance gain of the composite due only to the suction increase is discarded for longer curing days, since suction was maintained nearly constant during 30 days, and the values of suction were close to the tailing without additive;
- The analysis of the leachate material from the tailing-polymer composite did not present exceeding values of chemical elements that could cause contamination to the environment;
- There is technical feasibility for the application of the tailing-polymer composite in geotechnical works, as landfills and slopes, thus presenting considerable technical, economical, security, maintenance reduction and environmental viability, which grants a more proper end to this material.

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List of symbols

T: tailing *TP*: tailing -polymer

- L: linear adjustment 7d, 14d, 28d: 7,14 and 28 days respectively DRX: X-ray diffractometry EDS: energy dispersive spectroscopy SEM: scanning electron microscopy d: diffractometric pattern (calculated by the focal point of the diffractometer optics) Si: silicon Fe: Iron C: Carbon O: Oxygen Al: Aluminum Ni: Nickel Kev: Critical Ionization Energy K, L: layer where ionization occurred
- *a*, *esc*, *b*, *l*: layer from which the electron came out