Geopolymer as an alternative stabilizer of waste and tailings from iron mining

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Abstract

Mining tailings are increasingly abundant and, if not properly processed or stored, can pose environmental and social risks. This work evaluates the use of metakaolin-based geopolymer as a binder in mixtures of iron mining waste and tailings, with a view to mechanically stabilizing this waste for safer disposal. The percentage of water in the tailings and binder in the mixtures, water/solid ratio of the binder, proportion of precursor and binder activator and mixing time were evaluated. The raw materials and geopolymeric products were characterized using XRF, XRD, FTIR and compressive strength - only for geopolymeric products. The mixture with the lowest percentage of water in the tailings – 15% - and the highest percentage of binder - 30% - obtained 7.72 MPa after 7 days of curing - the only value higher than the ideal – 7 MPa after 7 days of curing. This result demonstrates the importance of the greater amount of binder in the mix and the detrimental effect of water in the mix. Increasing the mixing time, combined allied to not using water in the mixture with the binder, improved the strength values to 9.13 and 14.77 MPa after 7 and 28 days of curing, respectively. This may be due to the fact that the geopolymerization reaction was more complete, generating larger, more complex and resistant structures. **Keywords:** Geopolymer; Iron tailings; Waste; Stabilization; Tailings disposal.

1 Introduction

Mining is one of the most essential activities carried out by humanity over time and its importance can be observed by analyzing economic data from countries with a strong mining tradition, such as Brazil. In 2022, the mineral balance of almost US\$ 24.9 billion represented 40% of the Brazilian trade balance. This means that in 2022 Brazil exported 344 million tons of iron ore [1]. Observing this growing production of ores and being aware of the consequent chemical impoverishment of the deposits, it is easy to imagine that the amount of tailings generated by the mineral beneficiation process has increased dramatically in recent years [2,3]. Statistics show that dams and mining pits increase ten times in volume and double in height every 30 years, which increases the probability of tailings dams failing [4].

Recently two mining dams in Brazil failed, spilling more than 50 million cubic meters of tailings, causing serious environmental, social and economic damage. These accidents involving the companies Samarco (2015) and Vale (2019) killed almost 300 people [5,6]. Some of the dam failure mechanisms include cumulative damage (overlap, slope stability, internal erosion, and multiple earthquake events), geological hazards (landslides), liquefaction and changes in weather patterns [7]. A study [8] analyzed 147 cases of tailings dam failures worldwide and concluded that the largest fraction of incidents are related to meteorological causes, representing 25% of cases. Seismic liquefaction ruptures account for 14% and inefficient management practice was responsible for 10% of incidents.

Other studies [9,10] analyzed the economic conditions of the mining companies in the periods immediately preceding the accidents and concluded that both mining companies involved in the recent Brazilian disasters were experiencing considerable production growth, despite this no investment was made in new storage structures or tailings recovery routes. They emphasize the importance of developing new alternatives for the reuse and disposal of tailings, such as the disposal of filtered tailings in layers or in piles, tailings thickening, co-disposal of tailings and waste [7]. Mining tailings can also be used as a structural and support element for underground excavations, combined with cement and water (Backfill or Cemented Paste Tailings). However, when mine tailings have potentially acid-generating properties, cement-based paste is not suitable because the resulting acid can have harmful effects on the generated matrix [11]. In this context, geopolymers appear as alternative binders.

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Geopolymers are ceramic-like inorganic polymers produced at low temperatures. They consist of chains or networks of mineral molecules linked with covalent bonds [12]. They can be produced by combining an aluminosilicate precursor and a high-alkalinity material [13-16]. Geopolymers are considered an environmentally friendly technology, mainly due to lower CO₂ emissions in their production compared to OPC [17-19]. They also have some advantages such as superior strength, lower drying shrinkage and creep, resistance to chemical attack, better thermal behavior, effectiveness in stabilizing heavy metals and durability [20,21]. They are also recognized for being able to absorb large amounts of industrial waste as sources for their production, but the use of mine tailings as a raw material in alkaline activation is a challenge due to low reactivity, being preferentially used in the function of aggregates [11,22].

Geopolymer is a type of alkali-activated material (MAA), but it is important to note that not all MAA are geopolymers. Alkaline activation involves a synthesis reaction where raw materials rich in silicon and aluminum is mixed with a highly alkaline solution. When Al and Si ions are released into the solution, they begin to form a new 3D network sharing oxygen atom. Other elements, such as Fe, Mg and Ca, also contribute to this reaction, forming hydrated calcium (sodium) aluminosilicate. When the calcium content is low, alkali-activated materials are called geopolymers [11].

Conventional geopolymers, manufactured by the two-part methodology, are formed by a reaction between the solid precursor and alkaline and concentrated aqueous solution, usually of hydroxide and silicate.

However, impracticalities related to handling large amounts of viscous, corrosive and hazardous alkaline activator solutions have put pressure on the development of one-part geopolymers. Thus, one-part geopolymers are manufactured from the mixing and homogenization of their components, precursor and activators, in the solid state with the subsequent addition of water, as in the preparation of Portland cement [23].

Therefore, this study aimed to evaluate the feasibility of using metakaolin-based geopolymer as a binder in mixtures of waste and tailings from iron ore mine, with the ultimate goal of mechanically stabilizing this waste for the safe disposal of this material.

2 Materials and methods

2.1 Materials

The following materials were used in the production of the alternative geopolymer concrete: iron ore tailings (IOT) from Iron Quadrangle region in Minas Gerais state in Brazil (constituted of 80% of tailings from the flotation stage and 20% of tailings from the desliming stage – simulating the real proportions that these tailings in the dam) and waste, as aggregates; metakaolin (MK) as precursor; sodium hydroxide, NaOH (SH), with purity >97% and sodium silicate, Na₂SiO₃, (SS), composed of 18% Na₂O and 63% SiO₂, as alkaline activators. All materials were used in solid form.

The chemical composition for MK, IOT and waste was evaluated by X-ray fluorescence (XRF) analysis with a Thermo ScientificTM spectrometer, model GEN-X 4200W, with rhodium tube. The particle size distribution of MK and IOT was obtained using CILAS 1190 laser granulometry and dry sifting was performed for the waste using Tyler series sieves with 20 cm diameter. X-ray diffraction (XRD) was performed with a PANalytical X'Pert APD diffractometer using copper radiation (CuKa) to evaluate the mineral phases. Fourier transform infrared spectroscopy (FTIR) of the samples was performed with 128 scans with a Bruker device, model Alpha, by attenuated total internal reflectance with diamond crystal and resolution of 4 cm⁻¹ to contribute to the analysis.

2.2 Design work

Some variables were fixed to simulate the real conditions and proportions of these materials found in dams and waste piles. The water content (moisture) of the waste was set at 10% and the proportion of the aggregate mixture was fixed at 65% IOT and 35% waste.

The first stage focused on evaluating the influence of the IOT water content (moisture) and the weight proportion of binder in the mixtures on the final properties of geopolymer concrete. At this stage, six geopolymer pastes were produced, 1 to 6. Table 1 shows the mix proportions, as well as the variables investigated. The water to binder ratio (w/b) ratio was fixed on 0.33 at first stage of tests. It is important to note that IOT water content is not counted in the water to binder ratio and is therefore an additional source of water in the mixing of the geopolymer with the aggregates to form the geopolymer concrete.

The second stage focused on improving the mechanical strength and decreasing the efflorescence effect of the geopolymer concretes with 20% of the weight proportion of binder and 20% of IOT water content. For this, three mixtures were produced with water to binder (w/b) ratio reduced to zero, considering that the moisture present in the waste and IOT would be sufficient for the geopolymerization process.

In sample 2-2 the mixing time was doubled and in sample 2-3, the proportion of binder reagents were changed, reducing activators by 5%. The proportions are in Table 1.

2.3 Geopolymer synthesis

The one-part geopolymer concrete was prepared in an automatic electromechanical mixer. The precursor and activators were initially homogenized in a solid state and then water was added. The geopolymer (precursor + activators + water) was mixed for 2.5 minutes. Then the waste, with the moisture adjusted to 10%, was added and mixed for another 2.5 minutes. Finally, the IOT was added, also with adjusted humidity, mixing for another 5 minutes, with the total mixing Geopolymer as an alternative stabilizer of waste and tailings from iron mining

Mix ID		Mix	proportion	s (%)						
	Geopolymer Binder			Aggregates		- investigated variables				
	MK	SS	SH	ЮТ	Waste	IOT water content (%)	The weight proportion of geopolymer in the mix (%)	Water to binder ratio (w/b)		
1	7.0	2.4	0.6	58.5	31.5	20	10	0.33		
2	14.0	4.8	1.2	52.0	28.0	20	20	0.33		
3	21.0	7.2	1.8	45.5	24.5	20	30	0.33		
4	7.0	2.4	0.6	58.5	31.5	15	10	0.33		
5	14.0	4.8	1.2	52.0	28.0	15	20	0.33		
6	21.0	7.2	1.8	45.5	24.5	15	30	0.33		
2-1	14.0	4.8	1.2	52.0	28.0	20	20	0.00		
2-2	14.0	4.8	1.2	52.0	28.0	20	20	0.00		
2-3	15.0	4.4	0.6	52.0	28.0	20	20	0.00		

Table 1. Designed mix proportions

Table 2. Chemical composition of raw materials

Materials	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	MgO	TiO ₂	MnO
Metakaolin	49.53	43.09	3.02	0.09	-	0.87	0.17	1.32	-
Waste	22.76	0.64	70.63	0.03	1.22	0.09	0.01	0.24	0.10
Flotation tailing	82.03	0.32	16.06	0.20	-	0.01	-	-	-
Desliming tailing	17.04	3.36	74.65	0.19	-	0.07	0.02	0.10	-

time equal to 10 minutes. For the geopolymers produced in the second stage, the precursor and activators were initially homogenized and shortly thereafter the IOT and the waste, with adjusted moistures, were added and mixed for 10 minutes or 20 minutes in the case of the 2-2 sample. All geopolymer mixtures were placed in cubic molds with a 40mm edge.

The samples were cured at room temperature. The geopolymer samples were demolded after 24 h of curing. Compressive strength tests were performed after 1, 3, 7, 14 and 28 days in an electric press at a loading rate of 11.8 kN/min. Compressive strength values correspond to the mean from three testing results.

3 Results and discussions

3.1 Geopolymer synthesis

Table 2 shows the chemical composition of the raw materials used to produce geopolymeric pastes. According to the manufacturer data, the SH has a purity >97% and the SS is composed of 18% Na₂O and 63% SiO₂.

The particle size distribution is shown in Figure 1. MK has a d_{50} of approximately 23 µm and 90% of the material is smaller than 38 µm. The IOT size distribution (consisting of 80% of flotation tailing and 20% of desliming tailing) indicates a d_{50} of approximately 56 µm.

It is also worth mentioning that 80% of the material is smaller than 100 μm and 90% is below 125 $\mu m.$ For waste,



Figure 1. Particle size distribution.

it is observed that it is a coarser material, its top size is around 25.4 mm. The curve also shows that approximately 80% of the particles are smaller than 12.5 mm. The d_{50} is approximately equal to 1,7 mm.

3.1.1 X-Ray Diffraction (XRD)

A characteristic peaks of quartz and iron minerals, hematite and goethite, can be observed in the waste, flotation tailing and desliming tailing. The desliming tailing also has a characteristic peak of kaolinite. The metakaolin is mainly amorphous, but it is possible to observe small amounts of crystalline species through the characteristic peaks of quartz, hematite and muscovite, which are possibly gangue minerals from the original kaolin. In addition, the presence of less intense kaolinite peaks is noted, indicating that the calcination was not complete (Figure 2 A).

The XRD spectra of the analyzed geopolymer mixtures showed peaks related to crystalline phases, corresponding mainly to quartz, hematite, and goethite (Figure 2B). This indicates that, as pointed out in the literature, the tailings and the waste had an aggregate function in the geopolymerization process, as their crystalline phases were repeated in the geopolymeric pastes produced. This reaffirms the low reactivity of mining tailings, demonstrating that use as an aggregate is perhaps still the most appropriate [22]. According to [24], the minerals present in geological rocks, such as quartz, feldspar, and muscovite, are considered inert loads, do not enter the geopolymer reaction and appear in the XRD spectra after the production of geopolymer concretes and cements.

3.1.2 Fourier transform infra-red spectroscopy (FTIR)

The infrared (FTIR) spectra for the raw materials are shown in Figure 3A. It's observed that both waste and flotation and desliming tailings showed band peaks between

520-530 cm⁻¹, attributed to the stretching vibration of the Fe-O bond in iron phases (hematite and goethite) [14,25]. The bands 692 cm⁻¹, which appears in the flotation tailing, and 796 cm⁻¹, in common both in the waste and in the two tailings, are attributed to the symmetric vibration Si-O-T (T: tetrahedral Si, Al or Fe) in quartz [14,26]. The peak at 455 cm⁻¹ that appears in the flotation tailing is attributed to the Si-O-T stretching vibration [14]. The desliming and flotation tailings also showed peaks at 1090 cm⁻¹, attributed to the Si-O-Si stretching vibration [26]. The bands between 3600 and 3700 cm⁻¹ are attributed to the O-H stretching linked to the residual modes of kaolinite [14,25,27], therefore, it is understood that for the desliming tailing, the two observed peaks at 3691 and 3618 cm⁻¹ are assigned to this band. For metakaolin the main band observed is at 1039 cm⁻¹. The main band characteristic of the amorphous 2SiO₂*Al₂O₂ appears at approximately 1016-1037 cm⁻¹, when standard metakaolin is under study, and is attributed to the Si-O-T asymmetric stretching band [28], so it is understood that this is the band represented at the peak of 1039 cm⁻¹. Peaks at 530 cm⁻¹ are also observed in metakaolin, attributed to



Figure 2. X-Ray Diffractometry of raw materials (A), and of geopolymer products (B).



Figure 3. FTIR of raw materials (A), and of geopolymer products (B).

the stretching vibration of the Fe-O bond, and at 772 cm⁻¹, attributed to the symmetrical vibration Si-O-T in quartz.

For the geopolymeric pastes produced and selected for the characterization tests, the spectrum basically follows the same pattern, with the most important band centered around 1000 cm⁻¹ (main geopolymeric pattern), as shown in Figure 3B. During dissolution, the bands change slightly to low wavelength values and when the geopolymerization process is completed the wavelength is stabilized around 987-1010 cm⁻¹, which justifies the bands around 1000 cm⁻¹ in the spectra of all samples. Thus, it is understood that the band centered at 1090 cm⁻¹, observed in the flotation and desliming tailings, was displaced due to the structural change attributed to the substitution of Si by Al and Fe in the silicate chain, inducing a decrease in the wavenumber after geopolymerization [25,26]. This band is, therefore, attributed to the asymmetric stretching vibration of Si-O-T (T: tetrahedral Si, Al or Fe) being the characteristic peak of the geopolymer structure [14].

The bands 692 cm⁻¹, which appears in 6, 3 and 2-1, and 796 cm⁻¹, observed in 2, 2-1, 2-2 e 2-3, are attributed to the symmetric vibration Si-O-T (T: tetrahedral Si, Al or Fe) [14,26]. These bands were also observed in the waste and tailings. The bands centered around 3400 cm⁻¹ and 1630 cm⁻¹ are attributed, respectively, to the symmetrical vibration of O-H elongation and symmetrical vibration of O-H flexion, in water [14,25,27]. These two peaks appear in the 6 and 3 geopolymers. For all others, only the peak centered at 1630 cm⁻¹ is observed. This indicates that in all samples there is presence of residual water.

Another important band that appears in the spectra of geopolymer mixtures is the band centered between 1370-1470 cm⁻¹. This band corresponds to the vibration of asymmetric stretching of the O-C-O bonds of the carbonate groups (CO₃²⁻) that arise from the reaction of unreacted sodium with atmospheric CO₂, characterizing excess sodium in the mixtures and the phenomenon of efflorescence [14,25,27,28].

The phenomenon of efflorescence was observed (although in different intensities) in all mixtures produced in this research.

The stretching vibration band, centered at 455 cm⁻¹, attributed to the Si-O-T bonds, observed in the flotation tailings, shifted to a low wavenumber value, around 420 cm⁻¹, in all analyzed geopolymeric pastes, induced the substitution of Si atoms by Fe [14,25-27]. These variations indicate that part of the iron, originating from the tailings, possibly reacted during geopolymerization, however, further studies are needed to confirm this hypothesis.

3.2 Compressive strength of geopolymers

The results of the compressive strength test of the prepared geopolymers are shown below, in Figure 4. So far, there are no standards that establish a minimum uniaxial compressive strength value for this type of material and for the purpose desired in this study (alternative geopolymer concrete for mining waste/tailings disposal). Therefore, based on the norm of the National Department of Transport Infrastructure (DNIT 143/2010 - ES - Brazil), which determines that the soil-cement mixture used in the base layer of pavements must have a minimum value of 2.1 MPa for the compressive strength after 7 days, the present work assumed a minimum value of 7.0 MPa for the same period (7 days of curing) as a satisfactory stability parameter, for the disposal purpose for which the alternative geopolymer is intended.

Analyzing the results obtained in the first stage of the research, in general, and as expected, we can observe that the increase in the water content in the tailings had a negative impact on the compressive strength results. On the other hand, the increase in the binder content, between mixtures with the same water content in the tailings, proved to be beneficial, ensuring greater resistance to the specimens. Proof of this is the mixture 6 (higher % of binder and lower % of water), which achieved the best performance, reaching a value of 7.72 MPa after 7 days of curing, reaching 10.97 MPa with 28



Figure 4. Compressive strength of geopolymers produced in the first (A) and second (B) stages of the research.

days of curing, compatible with the target set. Studies in the literature [29,30] have already shown the negative influence of excess water on the compressive strength of geopolymers and concluded that the increase in the space occupied by water within the geopolymer matrix results in an increase in porosity, which in turn leads to a decrease in resistance to compression.

In the second stage of the research, mixtures were produced with 20% of water content in the tailings and 20% of binder in their compositions, but the water to binder (w/b) ratio was reduced to zero. It is noted, once again, that the mixtures with the lowest total water/solid ratio achieved better compressive strength results, confirming the conclusions of the first stage. As for the other variables investigated, we noticed that the increase in the total mixing time had the best effect on the strength results, possibly because the geopolymerization reaction was able to be more complete, which may favor the construction of three-dimensional networks between the silicon tetrahedrons and aluminum, generating larger, more complex and more resistant structures. In addition, possibly less unreacted alkaline ions were left and consequently decreasing the effect of efflorescence was attenuated. Sample 2-2 achieved the best results, reaching 9.13 MPa after 7 days and 14.72 MPa after 28 days of curing.

4 Conclusions

To carry out this research, a kind of alternative geopolymer concrete was developed, in which the geopolymer, based on metakaolin, replaced Portland Cement, and the waste and the tailings performed the function of coarse and fine aggregates, respectively. This geopolymer concrete has been studied primarily based on its mechanical performance and chemical analysis.

XRD analyzes showed that the tailings and waste were basically crystalline, which is intrinsically related to their low reactivity, being constituted mostly of hematite, goethite and quartz.

As for metakaolin, the XRD analysis showed that the material did not have very high purity, since the presence, in the crystalline form, of residual kaolinite and some of the gangue minerals from the original kaolin were detected.

Infrared spectra for metakaolin, tailings, waste and produced geopolymeric pastes demonstrated, through specific band patterns, that the main reactions of geopolymer materials occurred. The bands related to the tetrahedral Si-O-T bonds present in the tailings, centered at 1090 cm-1, were shifted to lower wave numbers when the Si atoms were replaced by Al atoms in the structures of the produced pastes, reaching bands centered on 990-1000 cm⁻¹, value at which the stabilization of the geopolymers occurs. There are also some suggestions that part of the iron in the tailings reacted during geopolymerization, occupying part of the Si-O-T tetrahedral sites. Bands centered between 1370-1470 cm⁻¹, attributed to the vibration of O-C-O bonds of carbonate groups (CO₃²⁻), which arise from the reaction of unreacted sodium with atmospheric CO₂, characterized excess sodium in geopolymer mixtures, confirming the efflorescence effect visually observed in the specimens. Bands attributed to the O-H vibrations of the water indicated the presence of residual water in the geopolymer samples.

The uniaxial compression tests clearly demonstrated the negative influence of excess water on the mechanical strength of geopolymer mixtures. The increase in the binder content, among mixtures with the same water content in the tailings, proved to be beneficial, ensuring greater resistance to the specimens. In the first stage of the research, only the mixture with 15% of water in the tailings (lowest percentage tested) and 30% of binder (highest percentage tested) reached the strength value established as ideal for the purpose of geopolymer concrete in this study. For the mixtures with 20% of water in the tailings and 20% of binder, the increase in the total mixing time guaranteed better values of compressive strength, around 9 MPa with 7 days of curing and 14 MPa with 28 days, values consistent with the objective established in the research.

The alternative geopolymer developed showed, on a laboratory scale, that, under ideal conditions of mixing time and total water/solid ratio, it can reach resistance values compatible with the soil-cement mixture used in the base layer of pavements, thus suggesting, mechanics stability adequate for alternative disposal of tailings. More investigations are needed to better understand the interactions between geopolymer matrices and aggregates, the economic viability and the most appropriate ways, on a pilot and industrial scale, to dispose of this material.

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