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Study of the Reuse of Iron Ore Tailing in Cementitious Compounds

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Abstract-The iron ore market is one of the main responsible for the iron demand in the steel industry and it also represents great importance in the Brazilian economy. The growing development of the sector has led to an increase in the generation of iron ore tailings (IOT). Due to the large amount produced and the form of conditioning, the IOT is related to several environmental impacts. This is especially evident when accidents occur, such as dam breaks. In this context, the iron ore tailing has been the object of studies for use with different purposes. This study aims to evaluate the prospects for reuse of the IOT as a partial substitute for Portland cement. The collected tailing, after drying and homogenization, was submitted to the comminution process, reducing its granulometry. With the powdered IOT, characterization tests were carried out in relation to its chemical and mineralogical composition, particle size distribution, specific mass and surface area. Subsequently, specimens of reference cement pastes were molded and the binder was replaced by 50% by the tailing and the water/fine factor (w/f) was varied. The physical and mechanical properties of the pastes were evaluated in the fresh and hardened state, with respect to their consistency and compressive strength. The superficial physical structure of the pastes was analyzed through digital microscope image. In the fresh state, cement pastes with 50% replacement of hydraulic binder by IOT generated higher fluidity of up to 22,19% in relation to the reference for the same water / fines (w/f) factor. In the mechanical characterization, it was observed a gain of strength in the paste made with IOT replacement when reducing the w/f factor, indicating the possibility of filler effect or pozzolanic reaction. In addition, the use of IOT reduces the consumption of non-renewable natural resources for civil construction, as well as contributes to the reduction of environmental impacts generated by the extraction of iron ore.

Keywords*-Iron Ore Tailings (IOT), Pastes with Cement Replacement, Iron Mining Waste*

I. INTRODUCTION

The iron ore market is one of the main responsible for the iron demand in the steel industry and represents great importance in the Brazilian economy, contributing strongly to the national GDP. According to data from [1], more than 400 million tons of iron ore are extracted, the main ore destined for export.

The growing development of the sector has led to an increase in the generation of iron ore tailings (IOT), inherent to its extraction and processing. These tailings are contained in the form of sludge, usually in large dams, and consist mainly of water and soil, composed of different amounts of silicon, aluminum and iron oxides. The quantities vary according to the ore beneficiation process and the lithology of the rock massif itself [2]. Due to the large amount produced and the form of conditioning, the IOT is related to several environmental impacts, this being especially evident when accidents occur, such as dam breaks. The tailing can cause damage to the ecosystem, contaminating soil, rivers and impacting wildlife and the lives of nearby residents. These impacts were observed after the break of the dam in the district of Bento Rodrigues, in Mariana, MG - Brazil [3], one of the largest environmental accidents in the world.

In this context, the IOT has been the object of studies for use with different purposes. We highlight the studies of [4], [5] and [6] on the characterization of mortars with iron ore tailing, as well as the studies of [7] on the use of ceramic materials for civil construction. Ref. [8] and [9] studied the application of the IOT for infrastructure and road pavement and Ref. [10] studied its application in concrete.

In this sense, this study aims to evaluate the reuse of the iron ore tailing as a partial substitute for Portland cement. Thus, a characterization of the material was carried out, after grinding, and tests in pastes to study the performance of mixtures containing 50% IOT.

II. MATERIALS AND METHODS

A. Materials

The materials used in this research were cement, water and iron ore tailing. It was chosen to use the Portland cement CP-V ARI, because it is a purer hydraulic binder, it contains at least 95% by mass of clinker with calcium sulphate and a maximum of 5% of carbonate material, besides having a larger specific surface, more complete burning, high C_3S (tricalcium silicate) content and lower mineral content in its composition [11]. The IOT used comes from a mud dam originating in Sarzedo, a city belonging to the Ferriferous Quadrilateral of Minas Gerais. The water used came from a local public supply network, supplied by COPASA (Companhia de Saneamento de Minas Gerais).

B. Methodology

It was collected 5kg of IOT sample, which was identified, air-dried and properly homogenized, according to guidelines of [12]. The tailing was characterized as its particle size, specific mass and mineralogical analysis.

The *in natura* IOT had a particle size higher than the desired one to perform the replacement of the IOT to the Portland Cement (Sieve # 200 - 0.074mm aperture, according to [13]). It was necessary, therefore, to reduce the granulometry through the comminution process, using a bar mill for 15 minutes, containing 42 steel bars with an average weight of 1.4kg. The mill is manufactured by PAVITEST - Contenco Indústria e Comércio LTDA.

For characterization of the iron ore tailing, the methodology presented in Table 1 was chosen.

TABLE I. EXPERIMENTAL TESTS

Material	Properties	Description	Standard
IOT	Physical Characterization	Particle Size Distribution Specific Mass	NBR NM 248/2001;
	Chemical and Mineralogical characterizations	X-Ray Fluorescence (XRF) X-Ray Diffraction (XRD)	- -
Paste	Physical	Normal Consistency Molding and Cure	NBR NM 43/2002; NBR 5738/2015
	Mechanical	Axial Compressive Strength	NBR 7215/1996

The determination of the specific mass of the grain of the IOT was done according to the prescriptions of [14], by means of helium pycnometry. The particle size analysis of the IOT sample was determined by the laser granulometry technique, the equipment used was the Sympatec Helos Vectra Laser Granulometer. The surface area was measured using the BET Method. The chemical characterization was done by quantitative analysis by X-ray fluorescence spectrometry and by the CLA70C method for the determination of iron (II) - FeO (silicate) volumetric oxide. The mineralogical characterization was performed using the X-ray diffractometer (XRD) for powder samples PHILIPS (PANALYTICAL), X'Pert-APD system, PW 3710/31 controller, PW 1830/40 generator, PW 3020/00 goniometer.

For the characterization of the cement pastes, 16 cylindrical specimens measuring 2,5x4 cm were prepared with variable water / fines (w/f) factor, such as indicated in Tab. 2, being 8 specimens reference pastes and 8 specimens with IOT replacement to Portland cement in the 1:1 proportion. The molding was performed in two layers with manual consolidation using a 4.2mm rod and 5 strokes per layer. A vibration table was also used during 15 seconds to help consolidation. The demolding was performed after 48 hours and the specimens were cured in a tank, submerged in non-current potable water, protected from contamination and solar rays inside the humid chamber.

TABLE II. PASTES PREPARATION

Pastes	Specimens	w/f Factor (%)	Cement (g)	IOT (g)	Water (g)
Reference	4	31,00	1300	-	403,00
	4	22,08	1300	-	287,00
IOT Replacement	4	31,00	350	350	217,00
	4	22,08	350	350	154,60

The physical and mechanical properties of the reference pastes and with replacement in the fresh and hardened stage were evaluated. The normal consistency test was performed with the Vicat apparatus and the Tetmajer probe (Fig. 1), according to standard recommendations [15]. The Consistency Index was measured by the Flow Table method (Fig. 2), according to [16]. And finally, the axial compression strength test was performed in the press manufacturer Instron model 5582 according to recommendations of the standard [17].

Figure 1. Normal Consistency Test – Vicat Apparatus.

Figure 2. Consistency Index - Flow Table Test.

III. RESULTS AND ANALYSIS

A. Characterization

1) Chemical and mineralogical analysis

For the analysis of the components and mineral phases of the IOT, the chemical analysis was performed by X-ray diffraction, whose results are presented in Tab. 3 and Fig. 3. The hardness of the minerals was described according to [18]. In the mineralogical analysis by X-ray Fluorescence (FRX), with results in Table 4, the composition of the tailing and Portland cement used is compared.

TABLE III. DRX: MINERAL COMPOSITION OF IOT RESULTS

DRX		
Amount	Element	
High	Fe, O	
Medium	Si	
Low	Al, P	
Trace	Mn, Ti, Cl, S, Mg, Na	
Mineral Composition		
Mineral	Hardness	Presence by region
Hematite- Fe_2O_3	5,5 - 6	X
Quartz – SiO_2	7	X
Gibbsite - $Al(OH)_3$	2,5 - 3	X
Goetite - FeO(OH)	5 - 5,5	X
Magnetite – Fe_3O_4	5,5 - 6	X
Caulinite – $(Al_2Si_2)_5(OH)_4$	2 - 2,5	-

It is observed that the tailing consists mainly of Hematite and Quartz. Ref. [19], states that quartz found in very fine particles can assume three functions when incorporated into cement mixtures: improve packing, serve as nucleation points, facilitating the cement hydration, or aid in strength as a reactivity material depending on the stage in which it is found. In this way, the IOT presents potential to improve the mechanical behavior of cementitious compounds.

The mineralogical analysis assists in the perception of possible reactions when mixed to the cement for the formation of paste, mortar or concrete. Reactions may be beneficial or deleterious due to the presence and concentrations of certain compounds. Tab. 4 shows that, as expected, iron oxide is the largest component in the IOT and is found to be low in cement content. SiO_2, however, is in a slightly higher amount than Portland cement. It is important to note that the IOT has low concentrations of MgO, oxide that can cause expansion in cementitious compounds when reacting with water, and NaO and K_2O, components that can react with aggregate (reactive silica) in the concrete mixture causing disintegration, known as alkali-aggregate reaction. According to [20], disaggregations can be caused by expansions due to the reaction of free lime, magnesium and calcium sulfate. Magnesium reacts in a similar way to free lime only in crystalline form, present in the clinker, where it hydrates very slowly, occupying a larger volume than the original free calcium oxide. Calcium sulphate causes expansion by the formation of calcium sulfoaluminate (ettringite) with excess calcium sulphate not used by C_3A during the cement hydration. It is also observed that the tailing has loss of ignition (LOI) close to that of cement, which indicates that this material, inserted in cementitious compounds, can present good behavior in fire situations.

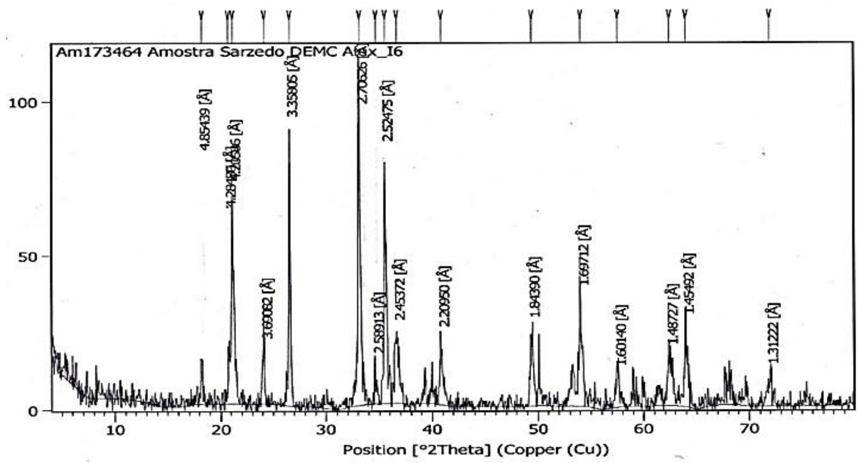

Figure 3. DRX results

TABLE IV. MINERALOGICAL COMPOSITION OF IOT AND CEMENTMINERALOGICAL ANALYSIS

Contents (%)											
Analyze	FeO	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	TiO_2	P_2O_5	Na_2'O	K_2O	MnO	LOI
IOT	0,49	25,60	3,13	59,10	3,85	0,23	0,14	0,16	<0,1	0,38	0,23	6,12
CP-V ARI	0,16	20,00	4,62	2,76	60,10	1,69	0,23	0,23	0,22	0,95	0,16	5,24

1. Physical characterization

The physical characterization was performed through laser granulometry, specific mass and surface area. The granulometric distribution, stated in Fig. 4, shows that the studied IOT has a larger amount of particles smaller than 10 µm than Portland cement. This can also be observed by the results in Table 5, where D_{50} of the residue is 38.6% lower.

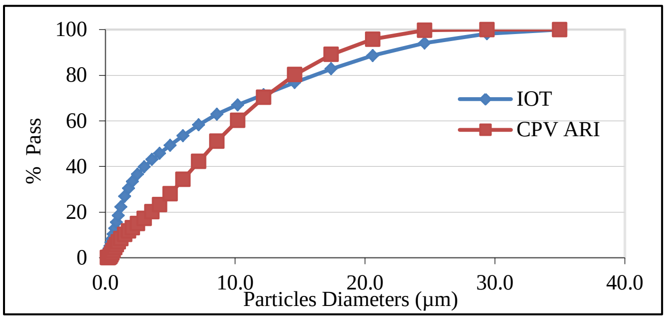

Figure 4. Distribuição granulométrica do IOT e do cimento

TABLE V. GRANULOMETRIC ANALYSIS RESULTS

	D_{10} (µm)	Δ(%)	D_{50} (µm)	Δ(%)	D_{90} (µm)	Δ(%)
CP-V ARI	1,48	-	8,44	-	17,82	-
IOT	0,61	-58,78	5,18	-38,62	21,61	21,26

According to the manufacturer, the specific mass of Portland cement CP-V ARI is 3.15 g/cm³. Through the pycnometer test, a value of 3.31 g/cm³ was obtained for the iron ore tailing, wich is close to that of the cement (difference of 5% greater). This result was similar to that verified by [21], which obtained a specific mass of 3.30 g/cm³ for mineral sample constituted basically by hematite (Fe_2O_3) and quartz (SiO_2).

Through the BET, a surface area of 7.790 m²/g was obtained for the IOT. As verified by [19] Portland cement CPV-ARI has 1.70 m²/g of surface area, obtained by the same method. Therefore, the residue is more than 350% thinner than the binder. This result was already expected since a higher percentage of fines in the residue were verified in the particle size analysis. These ultra-fine IOT particles serve to raise the compactness of the cementitious matrix and facilitate cement hydration by increasing water retention [20]. These effects may be beneficial for both fresh and hardened properties, improving workability and maintaining the compressive strength properties of the cementitious compound with mineral tailing content.

B. *Characterization of the paste*

1) Physical characterization

The physical characterization of the cement paste was performed through the consistency index test. The purpose of physical characterization of the cement paste with two w/f factors was to evaluate the minimum cement consumption and workability. The average measurements of the spreading of the paste cone trunk base after the abatement are described in Table 6. The results of the Flow Table test are presented in Fig.5.

TABLE VI. AVERAGE CONSISTENCY SCATTERING.

w/f Factor	Sample	Reference
	IOT	CP V ARI
31,00%	26,00 cm	20,23 cm
22,08%	17,83 cm	-
% of Reduction	31,42	-

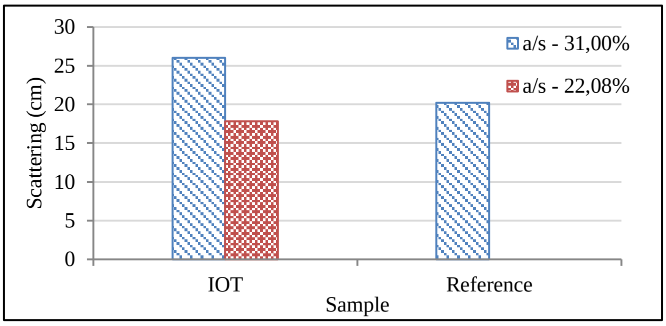

Figure 5. Results from the Flow Table Test – Average Scattering.

In the analysis of the data presented in Tab.6 and Fig.5, it can be seen that the pastes with replacement of the binder by IOT with the w/f factor = 31% are more fluid than the reference paste, reaching 22.19% more scattering. Due to the dry consistency of the reference paste with w/f factor = 22.08%, it was not possible to measure its scattering, however the pastes with IOT replacement with w/f factor = 22.08% reached a value of average scattering close to the reference. There was a reduction of 31,42% of scattering when comparing the w/f factor for the sample with IOT replacement. Correlating the fluidity results with those of the granulometric analysis, it was expected that there would be an increase in water demand for the sample with IOT replacement, since it has finer granulometry than the cement, nevertheless this was not observed. This result is possibly related to the difference in porosity of the material and the cement, because the tailing was much thinner, it increases the water demand.

2) Axial compressive strength

The results of the compressive strength test, presented in Fig. 6, allow concluding that there was no deterioration of the mechanical properties with the introduction of the tailing in the mixture. In the paste with a w/f factor of 22.08% it was observed a 22% improvement in strength.

According to [21], with an increase in the water addition, a reduction in the compressive strength is expected. However, for the reference paste with lower w/f factor, the mixture became very dry, which increased the porosity, reducing the mechanical strength by 26% in relation to the sample with a factor of 31%.

Since the IOT has a fine granulometry, the increase in the compressive strength of the IOT samples in relation to the reference can indicate an improvement of the particle packaging, such as in a filler material. Another possibility pointed out by this result is a chemical reaction between the IOT and the cement.

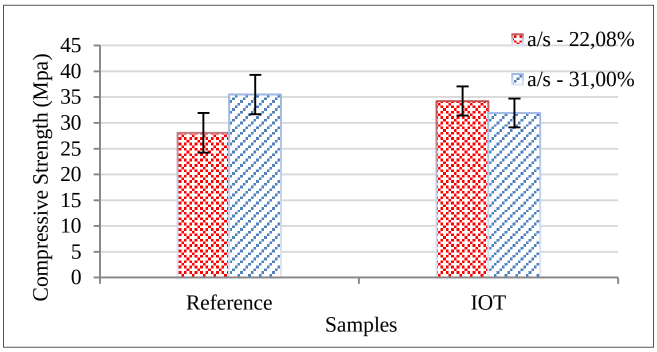

Figure 6. Results of the axial compressive strength test

3) Surface Structure

Through the images presented in Fig. 7 a Fig. 10, the reduction of porosity as a function of the w/f factor is observed. For the reference sample with a lower w/f factor, a higher porosity is observed, due to the difficulty in molding the specimen, once the paste mixture has become very dry. This was reflected in the lower results for compressive strength of this sample. In the sample with IOT and higher w/f factor, there was a pore closure. However, its mechanical strength was lower due to excess water.

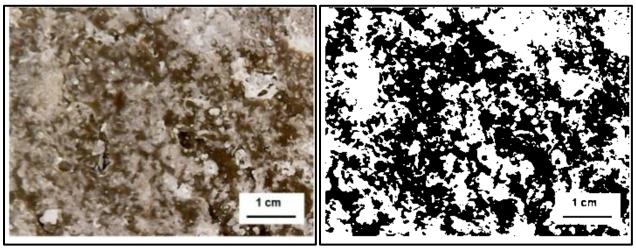

Figure 7. Reference paste – w/f factor =22,08%

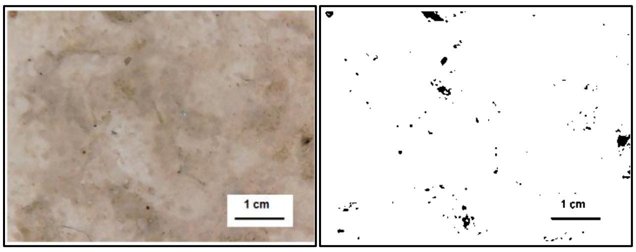

Figure 8. Reference paste – w/f factor =31%

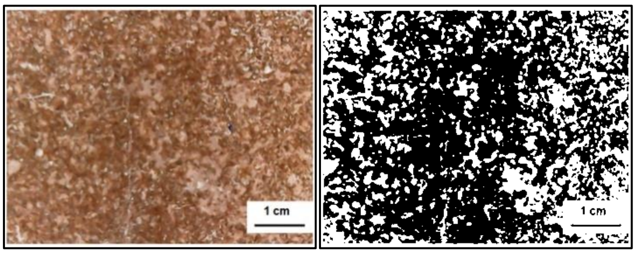

Figure 9. Paste with IOT replacement– w/f factor = 22,08%

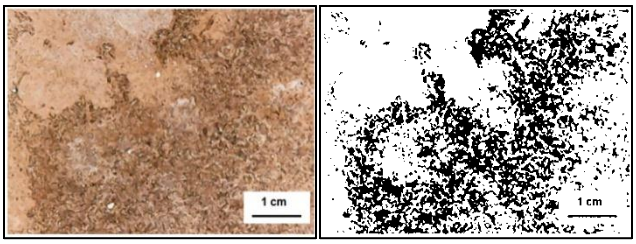

Figure 10. Paste with IOT replacement – w/f/ factor = 31%

IV. CONCLUSION

In the physical characterization of the IOT it was observed that the effectiveness of the milling process of the tailing depends on the hardness of the constituent minerals. In the mineralogical characterization, it was observed heterogeneity and also predominant presence in the IOT sample of Quartz-SiO_2, Hematite-Fe_2O_3, Goethite-FeO (OH) and Gibbsite-Al (OH).

In the fresh state, cement pastes with 50% replacement of hydraulic binder by IOT generated higher fluidity of up to 22,19% in relation to the reference for the same water / fines (w/f) factor. In the mechanical characterization, a gain of strength in the paste with IOT replacement was observed when reducing the w/f factor, indicating the possibility of filer effect or pozzolanic reaction. In the analyses made for the consistency index, it was observed that the tailings porosity influences on the properties of the cement paste, which requires further investigations to be made.

According to the experimental results and verifications of this study, it was observed a good compatibility of cementitious products with the use of the iron ore tailing. In addition, the use of IOT reduces the consumption of non-renewable natural resources for civil construction, as well as contributes to the reduction of environmental impacts generated by the extraction of iron ore.

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