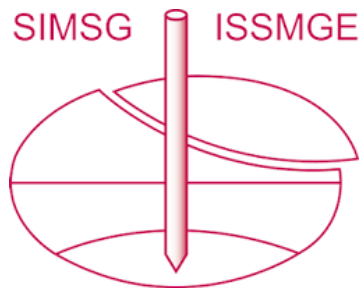


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The effect of key parameters on the mechanical response of artificially cemented iron ore tailings for dry stacking purposes

Nilo Cesar Consoli^{1,#}, Bruno Guimarães Delgado², João Paulo Sousa Silva² and Hugo Carlos Scheuermann Filho¹

¹Universidade Federal do Rio Grande do Sul, Graduate Program in Civil Engineering, Osvaldo Aranha 99, Porto Alegre-RS, Brazil

²VALE S.A, Exploration and Mineral Projects – Mineral Development Centre, Santa Luzia, MG, 33040-900, Brazil

[#]Corresponding author: consoli@ufrgs.br

ABSTRACT

Regulations prohibiting the construction of new tailings dams using the upstream method and requiring the closure of the existing ones have been recently introduced in Brazil. This scenario demands the sought for alternatives to manage both the previously disposed tailings as well as the upcoming generated mining residues. Fortunately, advances in dewatering technologies have enabled the tailings' dried disposal in stacks having hundreds of meters rather than the traditional slurry deposition. Eventually, a cement material can be incorporated into the tailings before the stacking aiming to enhance the overall material's performance, permitting the construction of higher and more inclined piles. In this regard, the present study evaluates the effect of key variables on the stiffness and strength of compacted iron ore tailings-cement blends. Specifically, the effect of the following variables was statistically evaluated through a full factorial design set: dry unit weight (17, 18, and 19 kN/m³), amount of cement (1, 3, and 5%), curing period (7, 28, and 90 days) and cement type (pozzolana Portland cement and high early strength Portland cement). The results have shown that all the main factors, and most of the second-order interactions, were significant in altering both the strength and stiffness of the studied mixtures, with a highlight to the amount of cement, curing time, and dry density. The type of cement, in turn, has exerted a marginal influence. As well, both responses could be successfully correlated to the adjusted porosity/cement index via power-type functions.

Keywords: iron ore tailings; dry stacking; artificially cemented tailings; soil stabilization.

1. Introduction

Brazil has been one of the world's top iron ore suppliers and the Iron Quadrangle Region, located in the province of Minas Gerais, corresponds to around 65% of the Brazilian iron ore production (Dauce et al. 2019, Vilaça et al. 2022). Huge amounts of iron ore tailings have, thus, been generated over the past decades, being the majority of these tailings hydraulically disposed in upstream heightened dams. Nonetheless, regulations prohibiting the construction of new tailings dams using the upstream method, and requiring the closure of the existing ones, have been recently introduced in Brazil for safety-related reasons (Schaper et al. 2020).

Lately, novel dewatering technologies have permitted the tailings' dried disposal rather than the traditional hydraulic deposition (Gomes et al. 2016). As a reason, the stacking of mounts of dry tailings becomes an alternative in the management of either the upcoming tailings or previously disposed tailings in dams that are about to be de-characterized. Eventually, a cementing material can be mixed into the tailings before the stacking intending to enhance the material's performance in terms

of strength and stiffness (e.g., Bruschi et al. 2022, Santos et al. 2022)

In this regard, the present study evaluates the effect of key variables on the mechanical response of compacted iron ore tailings-cement blends for dry stacking purposes. Specifically, it statistically addresses the influence of dry density, amount of cement, type of cement (Portland pozzolana cement and early strength Portland cement), and curing period on both the unconfined compressive strength and the initial shear modulus of the artificially cemented iron ore tailings mixtures. A full factorial design approach was used in the determination of the experimental runs. Additionally, the strength and stiffness outcomes were correlated to the porosity/cement index – η/C_{iv} .

2. Experimental program

Three parts compose the experimental program: (i) material characterization, (ii) specimens moulding and curing, and (iii) conduction of the unconfined compressive strength and initial shear modulus tests. A fully crossed design scheme (Montgomery 2019) was used in the establishment of the dosages for the last stage considering the following variables: dry unit weight ($\gamma_d =$

17, 18, and 19 kN/m³), amount of cement ($C = 1, 3,$ and 5%), cement type ($TC =$ Portland pozzolana cement and high early strength Portland cement), and curing period ($CP = 7, 28,$ and 90 days). A single moulding moisture content (w) of 11% was used regardless of the adopted dosage (e.g., Scheuermann Filho et al. 2022). Triplicates were tested within each one of the 54 mix designs, totalizing 162 tested specimens.

2.1. Materials

The iron ore tailings (IOT) were collected in a disturbed state behind a dam located in the Quadrilátero Ferrífero (Iron Quadrangle) region in the province of Minas Gerais, Brazil. Table 1 summarizes the main physical characteristics of the utilized tailings, indicating, the respective test method. X-ray fluorescence (XRF) tests have revealed that this material is mainly composed of Silicon (69.6%), iron (24.1%), and aluminum (4.8%). Mineralogically, X-ray diffraction (XRD) tests have attested the presence of prominent peaks of quartz (SiO₂), hematite (Fe₂O₃), and kaolinite (Al₂Si₂O₅(OH)₄). Two commercially available cement were used in this research: a pozzolana Portland cement (PPC) fly-ash based and a high early strength Portland cement (HES). Both cement' specific gravities lie around 3.15. Distilled water was used throughout this research.

Table 1. Physical properties of the iron ore tailings

Physical properties	Iron ore tailings	Test method
Liquid limit (%)	-	
Plastic limit (%)	-	ASTM D4318
Plastic index (%)	non-plastic	
Specific gravity	2.92	ASTM D854
Coarse sand (2.00 mm < d < 4.75 mm) (%)	0	
Medium sand (0.425 mm < d < 2.00 mm) (%)	4.0	
Fine sand (0.075 mm < diameter < 0.425 mm) (%)	49.0	ASTM D7928
Silt (0.002 < d < 0.075 mm) (%)	42.0	
Clay (d < 0.002 mm) (%)	5.0	
Effective diameter (D_{10}) (mm)	0.0085	
Maximum dry unit weight at standard effort (kN/m ³)	19.2 ($w = 11.2\%$)	ASTM D698
Maximum dry unit weight at modified effort (kN/m ³)	20.6 ($w = 9.2\%$)	ASTM D1557

2.2. Methods

2.2.1. Specimens moulding and curing

The undercompaction method (Ladd 1978) was used in the moulding of cylindrical specimens (50 mm in diameter and 100 mm in height) for the mechanical tests conducted herein. Essentially, each sample was individually moulded in three layers inside a cylindrical split mould to the target dry density via static compaction. Right after, each specimen was retrieved from the mould, measured, weighed, and forwarded to be cured in a room having a controlled environment (temperature of $23 \pm 2^\circ\text{C}$ and relative moisture of 95%). The following acceptance criteria were observed within each test sample: dry unit weight (γ_d) within $\pm 1\%$ of the target value and moisture content (w) within $\pm 0.5\%$ of the assigned value. On the penultimate curing day, each specimen was submerged in water ($23 \pm 2^\circ\text{C}$) to augment the degree of saturation, thus diminishing possible suction effects on the stiffness and strength tests (e.g., Consoli et al. 2007, Scheuermann Filho et al. 2022).

Concerning the dosage, the amount of cement (C) was based upon the mass of dry tailings and the chosen quantities are by the current practice of artificially cemented tailings (e.g., Festugato et al. 2013, Consoli et al. 2017). The moulding moisture content (w) was calculated over the mass of dry solids (tailings + cement) and the results of the compaction tests (Table 1) supported the definition of the utilized dry unit weight values. Following Consoli et al. (2007), the porosity (η) and the amount of cement can be gathered into a unique parameter, the porosity/cement content index (η/C_{iv}^a), as expressed in Eq. (1).

$$\frac{\eta}{(C_{iv})^a} = \frac{100 \cdot \left[1 - \frac{\gamma_d}{\gamma_s \cdot (1 + C/100)} + \frac{\gamma_d \cdot (C/100)}{(1 + C/100) \cdot \gamma_c} \right]}{\left[100 \frac{(C/100) \cdot \gamma_d}{(1 + C/100)} \right]^a} \quad (1)$$

where γ_d is the dry unit weight of the specimen, γ_s is the unit weight of the solids of the iron ore tailings, γ_c is the unit weight of the solids of the cement, C is the amount of cement expressed in percentage, and a is the adjustment exponent.

2.2.2. Ultrasonic pulse velocity tests

The initial shear modulus (G_0) of an isotropic and elastic medium can be determined through the product between the square of the shear wave velocity (V_s) and the apparent density (ρ) of this medium (ASTM 2019). Herein, an ultrasonic pulse velocity (UPV) device was used in the G_0 assessment of the compacted iron ore tailings-cement specimens. The UPV device emits shear waves at a constant frequency of 250 kHz via transducers coupled to the top and bottom of the testing samples with the aid of a special coupler gel. The shear wave velocity was, thus, calculated by measuring the travel time of the shear waves (t_s) that cross the specimens. This is a non-destructive test, so it was conducted on the same specimens which were about to be submitted to the unconfined compression tests.

2.2.3. Unconfined compressive strength tests

An automatic loading press, coupled to a 10 kN load cell, was used for the unconfined compressive strength tests. The tests were strain controlled considering a displacement rate of 1.14 mm per minute. The top load was registered within each test.

3. Results and discussion

3.1. General aspects

Following Diambra et al. (2017, 2019), both the initial shear modulus (G_0) and the unconfined compressive strength (q_u) of artificially cemented geomaterials can be expressed as a function of the porosity/cement index via power-type relationships as expressed by Eq. (2).

$$G_0, q_u = B \cdot 10^4 \left[\frac{\eta}{(C_{iv})^c} \right]^a \quad (2)$$

In this regard, the stiffness and the strength outcomes, for both the pozzolana Portland cement (PPC) and the high early strength Portland cement (HES), were correlated to the η/C_{iv}^c index. Table 2 summarizes the Eq. (2) parameters considering each cement type and each curing period. A c exponent equal to 1.0 has best modeled the response of the studied mixtures.

Table 2. Parameters for Eq. (2)

Curing period	Cement	Curing period	c	G_0		q_u	
				B	R^2 (%)	B	R^2 (%)
7	PPC	7	-	5.9	91	4.2	93
28		28	-	10.1	99	7.4	97
90		90	-	14.2	99	10.9	99
7	HES	7	-	4.6	98	3.4	94
28		28	-	5.2	97	4.6	94
90		90	-	6.8	94	5.6	96

Graphically, Fig. 1 presents the stiffness results as a function of the η/C_{iv} parameter for the specimens moulded using the pozzolana Portland cement whereas Fig. 2 presents the same considering the high early strength Portland cement. The strength outcomes are presented following the same logic in Fig. 3 (PPC) and Fig. 4 (HES). Considering the dry unit weight, the 17 kN/m³ specimens are represented by squares, the 18 kN/m³ by circles, and the 19 kN/m³ samples by triangles. The symbols relative to 7 days of curing are black, 28 days grey, and 90 days white. Within each figure, three well-defined groups in the η/C_{iv} axis are related to the amount of cement.

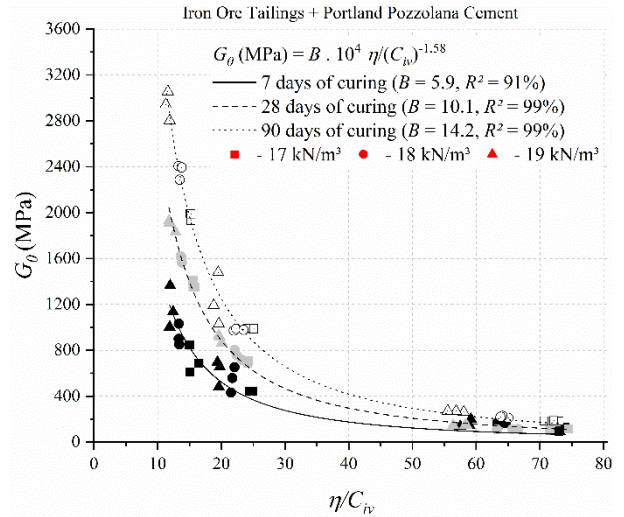


Figure 1. G_0 versus η/C_{iv} for the pozzolana Portland cement.

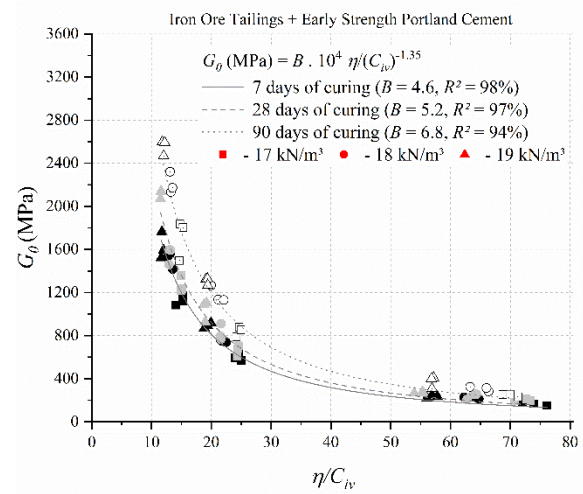


Figure 2. G_0 versus η/C_{iv} for the high early strength Portland cement.

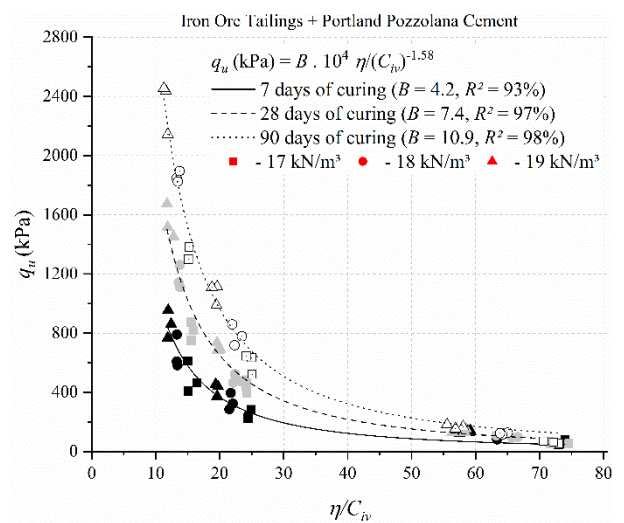


Figure 3. q_u versus η/C_{iv} for the pozzolana Portland cement.

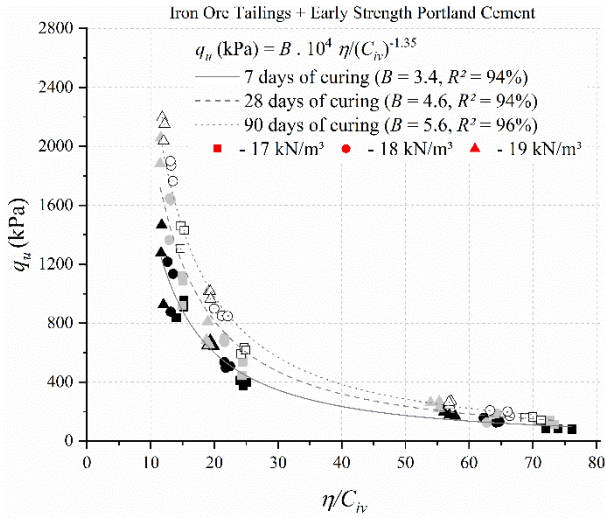


Figure 4. q_u versus η/C_{iv} for the high early strength Portland cement.

Regardless of the cement type, inverse trends have been obtained between either G_0 or q_u and the η/C_{iv} index. Namely, the densest specimens containing higher amounts of cement (i.e., designated by lower η/C_{iv} values) have presented a greater performance. Within each curing period, the great coefficient of determination (R^2) values previously shown in Table 2 are corroborated by the reduced amount of scatter around the fitted power-type equations. As well, the greater the curing period, the higher the registered G_0 and q_u values owing to the cement hydration products formed during time. Still, differences in both G_0 and q_u gains over the curing period are observed between the two types of cement.

The hydration of the pozzolana Portland cement is notably slower than the high early strength Portland cement, reflecting the higher rates of G_0 and q_u gains along the studied curing periods for the first. Mathematically, this is expressed by the values of B reported in Table 2, which, for example, range from 4.2 to 10.9 considering the strength of the PPC specimens whereas vary from 3.4 to 5.6 for the HES cement. The latter have also presented slightly higher G_0 and q_u values for the two lowest curing periods in comparison to the pozzolana cement. Nonetheless, the PPC samples have exhibited a greater performance when the curing period of 90 days is examined. Probably, this is related to secondary cementing products formed owing to the hydration of the existing pozzolan in the cement composition (Vedalakshmi et al. 2003, Soriano et al. 2013).

3.2. Statistical analysis

An analysis of variance (ANOVA) was carried out for the stiffness and strength results considering interactions up to the second order. The typical level of significance (α) equal to 5% was adopted. In this regard, Table 3 presents these results for the initial shear modulus data, whereas Table 4 summarizes the same for the unconfined compressive strength results. Concerning the notation, γ_d designates the dry unit weight, C is the amount of cement, CP is the curing period, and TC is the type of cement.

Table 3. ANOVA table for the G_0 data

Source	Degrees of freedom	Sum of Squares	Mean squares	p -value
Linear	7	71,724,049	10,246,293	
γ_d	2	3,167,394	1,583,697	0.000
C	2	59,877,555	2,993,8778	0.000
CP	2	8,383,146	4,191,573	0.000
TC	1	295,954	29,5954	0.000
Interactions	18	7,412,233	411,791	
$\gamma_d * C$	4	1,593,576	39,8394	0.000
$\gamma_d * CP$	4	205,974	51,493	0.004
$\gamma_d * TC$	2	73,414	36,707	0.059
$C * CP$	4	4,888,404	1,222,101	0.000
$C * TC$	2	10,799	5,400	0.654
$CP * TC$	2	640,066	320,033	0.000
Error	136	1,722,416	12,665	
lack of fit	28	1,029,621	36,772	
pure error	108	692,795	6,415	
Total	161	80,858,698		

Table 4. ANOVA table for the q_u data

Source	Degrees of freedom	Sum of Squares	Mean squares	p -value
Linear	7	49,254,881	7,036,412	
γ_d	2	3,305,513	1,652,756	0.000
C	2	39,512,509	19,756,255	0.000
CP	2	5,805,808	2,902,904	0.000
TC	1	631,051	631,051	0.000
Interactions	18	5,981,144	332,286	
$\gamma_d * C$	4	1,459,250	36,4812	0.000
$\gamma_d * CP$	4	281,485	70,371	0.000
$\gamma_d * TC$	2	25,631	12,816	0.289
$C * CP$	4	3,778,767	944,692	0.000
$C * TC$	2	155,99	77,995	0.001
$CP * TC$	2	280,02	140,01	0.000
Error	136	1,389,975	10,22	
lack of fit	28	680,8	24,314	
pure error	108	709,175	6,566	
Total	161	56,626,000		

For both the G_0 and q_u data, the influence of the single factors was, generally, considerably greater than the second-order interactions as can be perceived by analyzing the values of the sum of squares. All the factors were statistically significant in altering the G_0 and q_u responses of the compacted iron ore-cement blends (i.e., p -value $< \alpha$). As well, most of the second-order interactions were statistically significant at that adopted level of significance, with a highlight to the $C * CP$

interaction responsible for a substantial sum of square value in both the stiffness and the strength ANOVA analysis. Physically, this means that the cement hydrates over time, producing binding compounds that positively affect the material's behavior.

The main effects plot for the G_0 and q_u data are presented, respectively, in Fig. 5 and Fig. 6. Both the stiffness and the strength of the artificially cemented iron ore tailings are qualitatively affected in the same manner by altering the level of the assessed factors. In other words, the increment in the dry unit weight, for example, positively influences G_0 and q_u . The same applies to the curing period and the amount of cement. The type of cement, in turn, has exerted a marginal influence on the mechanical response.

Those results sort of corroborate what has been previously shown when correlating G_0 or q_u with the porosity/cement index using power-type relationships (Table 2). That is, both responses are enhanced for denser media containing higher amounts of cement. Ultimately, the contact area between the tailing' particles increases due to the augment in either the dry unit weight or the amount of cement, causing an increment in the initial shear modulus (Fernandez and Santamarina 2001). In parallel, the degree of interlocking between the particles strengthens owing to the increase in the dry density and, likewise, in the amount of cement, thus favoring the unconfined compressive strength of the mixtures. Naturally, the cement hydrates over time, forming binding products, which explains the high influence exerted by the curing period (CP).

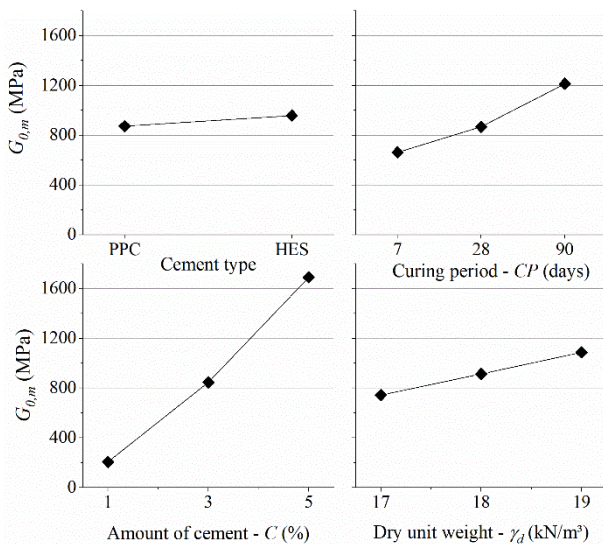


Figure 5. Main effects plot for the G_0 data.

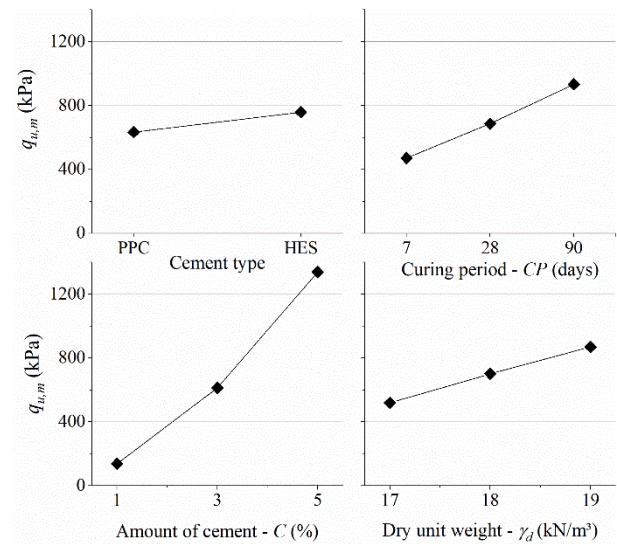


Figure 6. Main effects plot for the q_u data.

4. Conclusions

From the data presented throughout this manuscript and considering the boundaries of the study, the following conclusions can be drawn:

- The η/C_{iv} index has proved its usefulness in modeling both the small strain stiffness and the unconfined compressive strength of compacted iron ore tailings-cement blends, regardless of the cement type and the curing period. Thence, it might be a useful tool for dry stacking applications;
- The usage of pozzolana Portland cement, rather than the high early strength Portland cement, appears to be more interesting on a long-term basis as demonstrated by the greater performance of the former considering the 90 days curing period.
- The minimum amount of cement utilized herein (i.e., 1%) is not advisable for practical purposes owing to the lack of performance in comparison to the other used cement contents (3 and 5%).

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