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Study of the reduction of resistance over time in specimens tested by uniaxial compressive strength for paste filling

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Abstract

This paper presents the results of a case analysis which was the product of a conceptual study of the paste fill mix design carried out for a polymetallic mine in southern Peru. Paste fill is a mixture of water, cement and tailings in a high density. It is used in underground mines to fill cavities resulting from rock extraction (galleries, chimneys, pits, among others) to provide the support needed to continue mining and avoid a possible collapse. The strength requirements for the primary paste fill were between 1.0 to 2.0 MPa, and for the secondary filler, between 0.7 to 1.0 MPa after 28 days of setting. The physical, chemical and mineralogical characterisation of the tailings to be used was carried out, as well as uniaxial compressive strength tests (UCS.) of the paste. The mineralogical characterisation allowed us to identify a high presence of siderite (26%) and kaolinite (12%), minerals that do not benefit from obtaining high resistance values. On the other hand, the UCS allowed us to identify that, after 28 days of curing, the resistance of the analysed specimens presented a decrease concerning the values obtained at 7 and 14 days of curing. Given this scenario, tests were carried out at 60 and 90 days of curing and detected that the decrease in resistance fell to values well below those required (a decrease in resistance of more than 40%). Analysing the results identified that the presence of sulfates in the tailings possibly generated gypsum crystals during mixing. When they grow, these crystals break the cement bonds over time, affecting the integrity and resistance of the tested paste.

Keywords: *paste fill, mining, sulfates*

1 Introduction

Paste fill is a mixture of water and tailings with additives such as Portland cement, lime and slag, which react as binding agents (Belem & Benzaazoua 2008). This mix of components is used in underground mines to fill cavities resulting from the extraction of the mineral/s, allowing mining to continue and ensuring structural stability, thereby avoiding a possible collapse and improving the mine's productivity. To guarantee that the mix of these components provides a composition with the necessary properties, it is essential to characterise the tailings to be used, the water and the necessary additives. Also, due to modern mining's commitment to mine safety, the design of the paste backfill must ensure that it achieves the strength requirements according to the geomechanical study of the stability of the extraction zone. The purpose of this article is to present the results of UCS tests that contributed to the conceptual design of a paste filling plant for a polymetallic mine in southern Peru, in order to study the reduction in resistance over time of the specimens tested by UCS. As part of the tests, resistance measurements of specimens with days of curing greater than 28 days (up to 90 days) were carried out in order to understand the trend over time. They provided atypical results because the resistance after more than 28 days decreases to almost half of the target resistance. Due to this, it was proposed to evaluate different alternatives of mixtures by adding different types of cement and slag (in various concentrations) in order to define the mixture that best maintains resistance over time.

2 Methodology

To guarantee compliance with the necessary design parameters of the paste fill to be used, different tests were applied to the tailings in order to define their physical, chemical and mineralogical properties. Additionally, mechanical tests were carried out to determine the behaviour under loads of the paste filling.

2.1 Preparation of sample

Taking a representative sample for the development of the tests provides the most important guarantee that the results found will be useful for the development of engineering. Once the representative sample of the process has been obtained, the sample preparation stage is reached. Here the sample is homogenised and distributed for the different tests to be considered in the study. These newly generated subsamples must be from the same representative sample.

2.2 Physical characterisation of tailings

2.2.1 Particle size distribution

The particle size distribution (PSD) was carried out on the tailings considering the ASTM Standard D422 (ASTM International 2014a) using sieves of different sizes. For the finest sizes, the hydrometer methodology was used.

2.2.2 Specific gravity of solids

The specific gravity of the tailings solids was carried out considering the ASTM Standard D854 (ASTM International 2014b). For this test, a pycnometer was used.

2.3 Chemical and mineralogical characterisation of tailings

For the chemical and mineralogical analysis, the samples were sent to the Bizalab laboratory in Lima, Peru, where fluorescence and X-ray diffraction (XRF) tests were carried out.

2.3.1 Chemical analysis by XRF

In the chemical analysis by XRF, the semiquantitative determination of the elements from sodium (Z=11) to uranium (Z=92) was performed. The elements that are not reported were not detected by the equipment because they were below the detection limit of the respective element. Elemental results have been stoichiometrically expressed in oxides. The preparation method applied to the samples was by pressed tablet. The report includes the loss on ignition (LOI – calcination at 950°C for 2 hours), which corresponds to the weight loss, expressed as a percentage, and which reflects the content of volatile components such as water, carbonates (decomposition releasing CO_2), sulfates and sulfides (decomposition releasing SO_2), among others, in the samples.

2.3.2 Mineralogical analysis (X-ray diffraction XRD)

In the mineralogical analysis of clay minerals by X-ray diffraction XRD, the determination of the global mineralogy (random powder preparation) was carried out, in addition to finding the differentiations between the types of laminar minerals through the application of processes such as granulometric separation (preparation of oriented sheet). According to the type of clay found, the identification and its semiquantitative evaluation of the total sample have been reported with a detection limit (LD) of 1% on average. For the case of amorphous phases, the detection limit is approximately 20%. The identification and quantification of copper silicate phases are limited because they lack structural ordering (low crystallinity), in accordance with the results found in the analysis carried out by the Bizalab laboratory.

2.4 Uniaxial compressive strength tests

Uniaxial compressive strength (UCS) tests were carried out using tailings from a mining process. A test program for paste filler samples was performed to evaluate the strength of the filler using 50.8 × 101.6 mm cylinders. Figure 1 shows the reference moulds for the conformation of specimens. The cylinders were cured in a high humidity environment maintained at between 20 and 25°C. Three cylinders were cast per curing period (at 7, 14 and 28 days) and the results were averaged.

Figure 1 Equipment for the preparation of test tubes

3 Data and results

3.1 Physical characterisation of tailings

3.1.1 Particle size distribution

Figure 2 shows the PSD of the tailings found via testing developed under the ASTM Standard D422 (ASTM International 2014a).

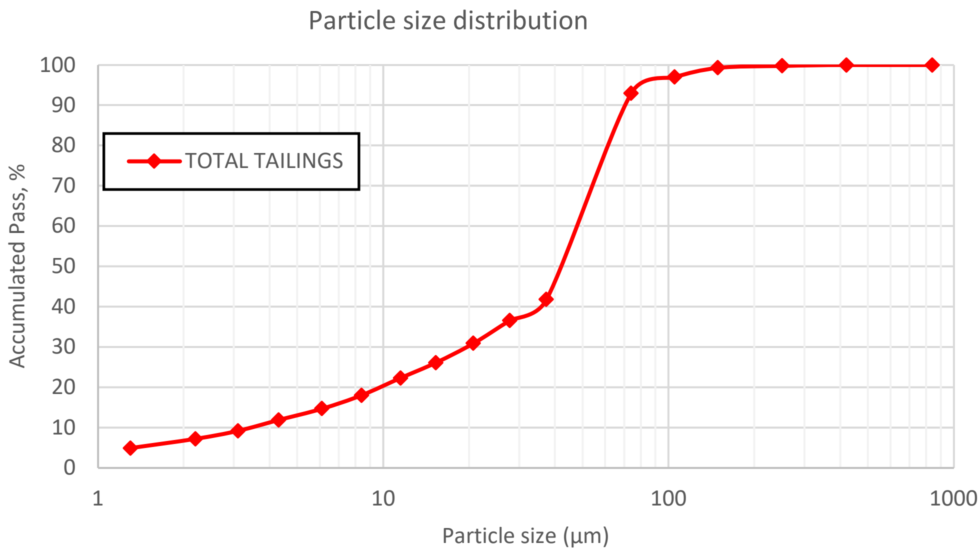

Figure 2 Total tailings – particle size distribution

According to the ASTM Standard D422 (ASTM International 2014a), the classification of fine particles is silts and clays, and the fines are considered for sizes smaller than 0.074 mm. The silts were found to be between 0.005 to 0.074 mm, while the clays were found in sizes smaller than 0.005 mm. Particles with sizes smaller than 0.001 mm are called colloids.

The granulometric distribution shows that the fraction passing through 20 µm mesh was in the recommended range between 20 and 40%, and the maximum size of the particles was below 12.7 mm. These two characteristics resulted in the paste obtained having good properties, however, the fraction of ultra-fine particles was 16%, which caused a decrease in the resistance of the filling (Safarizadeh, A & Taheri).

3.1.2 Specific gravity of solids

Table 1 shows the result of the specific gravity of the tailings found using the test developed under the ASTM Standard D854 (ASTM International 2014b).

Table 1 Specific gravity results

Description	Average value	Units
Specific gravity of solids	3.04	Adimensional

3.2 Chemical and mineralogical characterisation of tailings

3.2.1 Chemical analysis by fluorescence (XRF)

Table 2 shows the results of the XRF chemical analysis test performed on the tailings.

Table 2 Results of chemical analysis by XRF

Formula	%
SiO_2	45.76
Fe_2O_3	20.12
Al_2O_3	9.31
SO_3	6.42
CaO	1.31
MnO	1.26
MgO	0.77
ZnO	0.59
K_2O	0.57
TiO_2	0.35
P_2O_5	0.12
PbO	0.06
As_2O_3	0.04
Na_2O	0.03
Cl	0.03
ZrO_2	0.02
V_2O_5	0.02
CuO	0.02

3.2.2 *Mineralogical analysis (X-ray diffraction XRD)*

Table 3 shows the results of the mineralogical analysis by X-ray diffraction XRD, test performed on the tailings.

Table 3 Results of mineralogical analysis by XRD

Name of mineral	General formula	Results (%)
Quartz	SiO_2	50.0
Siderite	$FeCO_3$	26.0
Kaolinite	$Al_2Si_2O_5(OH)_4$	12.0
Pyrite	FeS_2	6.0
Feldspar – K (Orthoclase)	$KAlSi_3O_8$	2.0
Sphalerite	(Zn, Fe) S	<LD*
Gypsum	$CaSO_4.2(H_20)$	<LD*

*Below limit of detection

From the results found, the most abundant mineral is quartz at 50% followed by siderite at 26%, kaolinite at 12%, pyrite at 6% and feldspar at 2%. The sphalerite and gypsum are below the detection limit.

The sample is made up of free and almost equigranularity grains of plagioclase (incipiently altered to sericite and clays), quartz, carbonates I, potassium feldspars (incipiently altered to sericite, clays and carbonates) and opaque minerals, as well as different types of agglomerates. The free and agglomerated grains are encompassed and supported by a matrix composed of clays with microscale aggregates of sericite, micritic aggregates of carbonates, and chlorites, with the presence of impregnations of iron oxides and clays.

Free quartz (CZ) and plagioclase (PGL) grains, as well as agglomerates (some formed by quartz and others by carbonates), were identified in a matrix formed by aggregates of carbonates, clays, chlorites and impregnated oxides (see Figure 3).

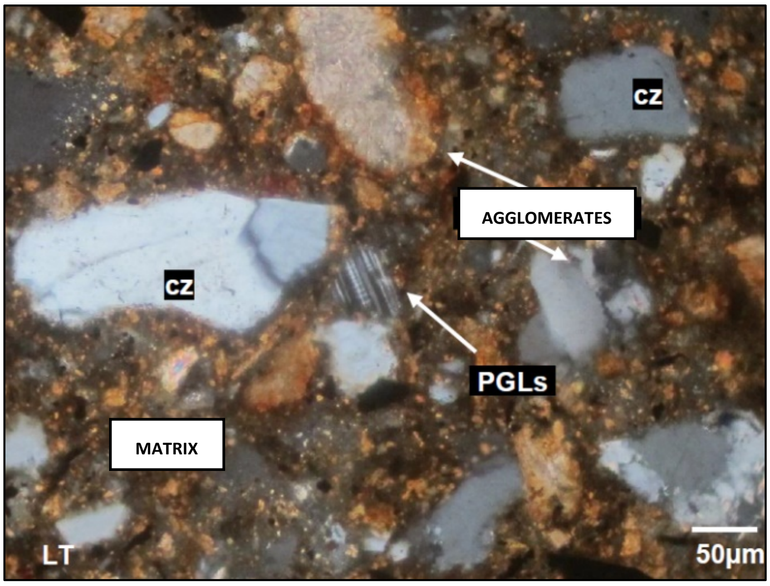

Figure 3 Image of the mineralogy of the mixture

According to the mineralogical results of the tailings, there is a high presence of siderite (26%) and kaolinite (12%), two of the least favourable minerals for obtaining high resistance values. Siderite is carbonate with low hardness and is quite fragile, while kaolinite is a clay mineral that attracts a lot of water so it tends to increase the required water-to-cement ratio.

3.3 UCS tests

The resistance tests considered the mining unit's sampled tailings, Portland cement Type I, process water and salt (or sea water), and the use of slag as an additive for optimisation in cement consumption.

The objective of resistance at 28 days of curing was defined as 1.0 MPa for the present study, and different parameters were raised to achieve the objective.

The UCS tests were carried out under two slump sizes: 177.8 and 254 mm, for which 77.5 and 75.2% solids concentration (% Cw) correspond, respectively.

Exploratory resistance trials were developed in the first stage (Stage 1) to find out how resistance increased or decreased under different conditions. Table 4 summarises the results of the UCS tests.

Table 4 UCS test results (Stage 1)

Stage 1										
No. of sample	Cement content (%)	Type of cement	Tailings	Slump (mm)	Aggregate type/content (%)	Type of water	UCS obtained (MPa)			
							Curing days			
							7	14	28	>45
D1	2	Portland Type I	Yes	177.8	–	Process water	0.40	0.43	0.46	0.36
D2	4	Portland Type I	Yes	177.8	–	Process water	0.89	0.97	0.99	0.51
D3	4	Portland Type I	Yes	254	–	Process water	0.59	0.61	0.62	0.34
D4	2	Portland Type I	Yes	254	Funsur (*) ground slag/2%	Process water	0.20	0.27	0.28	0.30
D5	4	Portland Type I	Yes	254	–	Salt water	0.55	0.62	0.68	0.38

*Slag from a smelter in Peru

Figure 4 graphically presents the resistance values' comparative results for the Stage 1 testing.

From the UCS tests (Stage 1) it was noted that after 28 days of curing, the resistance dropped for each of the designs evaluated. This could be due to a sulfate attack that generated gypsum crystals, which break the bonds of the cement once it grows to a certain size.

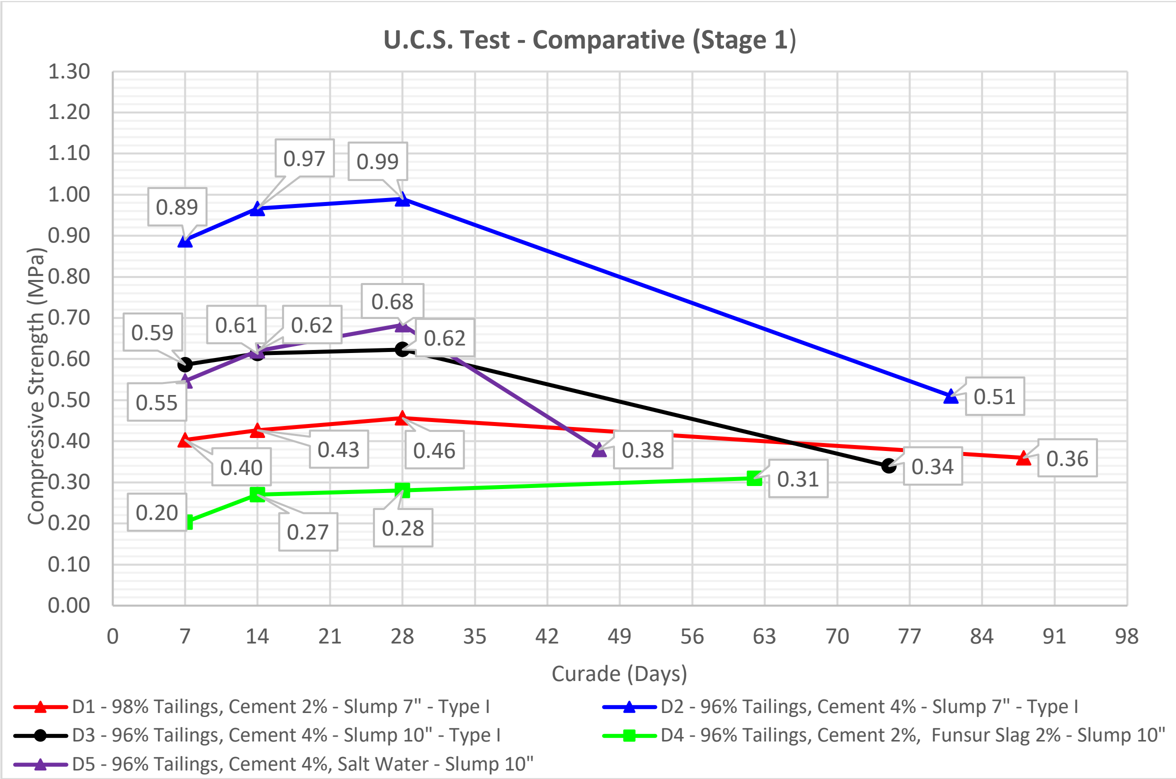

Figure 4 Results of UCS resistance tests (Stage 1)

It should be noted that during the first 28 days of curing, the resistance presented an upward trend.

However, for the following days, a decrease in resistance was clear.

For the second stage of tests, measurements were taken at 90 days of curing in order to discern how the resistance would trend with a longer curing time. Table 5 presents the summary of the results and the parameters considered for the resistance tests, while Figure 5 presents a comparison of the results.

Table 5 UCS test results (Stage 2)

Stage 2										
No. of sample	Cement content (%)	Type of cement	Tailings	Slump (mm)	Aggregate type/content (%)	Type of water	UCS obtained (MPa)			
							Curing days			
							7	14	28	90
D6	6	Portland Type I	Yes	177.8	–	Salt water	1.55	1.43	1.43	0.77
D7	3	Portland Type I	Yes	177.8	Funsur (*) ground slag/3%	Salt water	0.57	0.51	0.60	0.56
D8	6	Portland Type I	Yes	177.8	–	Process water	1.52	2.17	2.12	1.44
D9	3	Portland Type I	Yes	177.8	Funsur (*) ground slag/3%	Process water	0.72	0.76	0.80	0.76

(*) Slag from a smelter in Peru.

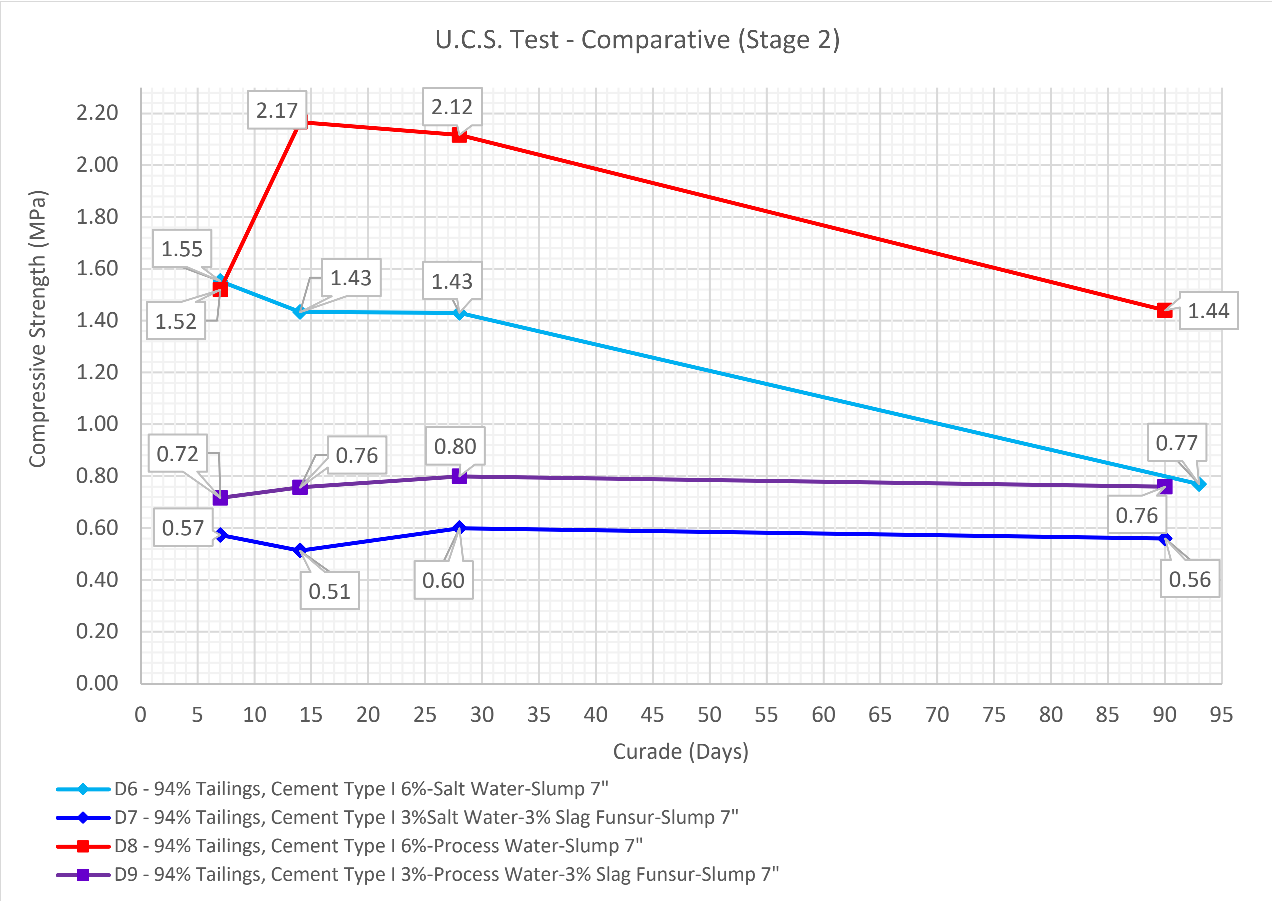

Figure 5 Results of UCS resistance tests (Stage 2)

From the UCS tests (Stage 2), Figure 5 shows that D8 (6% cement with process water) exceeded 2 MPa of resistance, however, at this stage, a small increase in resistance could be seen. After 28 days of curing there was a decrease in resistance, dropping to less than the resistance after 7 days of curing.

In the third stage of tests, an evaluation of Portland cement (ASTM International 2012) was proposed using the following types:

- HS-type cement.
- Portland I.
- Portland V.

It should be noted that different types of cements were evaluated to compare their behaviour of resistance over time, taking into account the properties of each type of cement.

Table 6 presents the summary of the results obtained in stage 3.

Table 6 UCS test results (Stage 3)

Stage 3											
No. of sample	Cement content (%)	Type of cement	Tailings	Slump (mm)	Aggregate type/content (%)	Type of water	UCS obtained (MPa)				
							Curing days				
							7	14	28	60	90
D10	4	Portland Type HS	Yes	177.8	–	Process water	0.50	0.67	0.69	0.21	0.25
D11	4	Portland Type I	Yes	177.8	–	Process water	0.77	0.81	0.84	0.51	0.37
D12	4	Portland Type V	Yes	177.8	–	Process water	0.70	0.86	0.88	0.54	0.43
D13	0.4	Portland Type V	Yes	177.8	Lafarge (*) slag/3.6%	Process water	N.C	N.C	0.7	1.34	0.63
D14	2	Portland Type V	Yes	177.8	Funsur (**) ground slag/2%	Process water	0.24	0.30	0.29	0.31	0.32
D15	2	Portland Type V	Yes	177.8	Lafarge slag/2%	Process water	0.27	0.50	0.90	0.46	0.46

*Slag from a cement plant

**Slag from a smelter in Peru

The UCS tests in Stage 3 were carried out with different types of anti-sulfate cement (Yin et al. 2017) and different types of slag. The results show that the sample (tailings/cement) which reached the highest resistance was type V cement. Nevertheless, the resistance fell after 28 days.

The test of cement, tailings and slag mixtures which had the lowest consumption of cement was the Lafarge slag. It was determined that Lafarge slag has better properties as a mixing additive since less is required compared to Funsur slag (Figure 6, samples D13 and D15). In the same way, Lafarge slag decreases the amount of cement required compared to Funsur. However, as Funsur slag is readily available for the project (unlike Lafarge slag), it provides significant savings.

Figure 6 shows the comparative results of Stage 3.

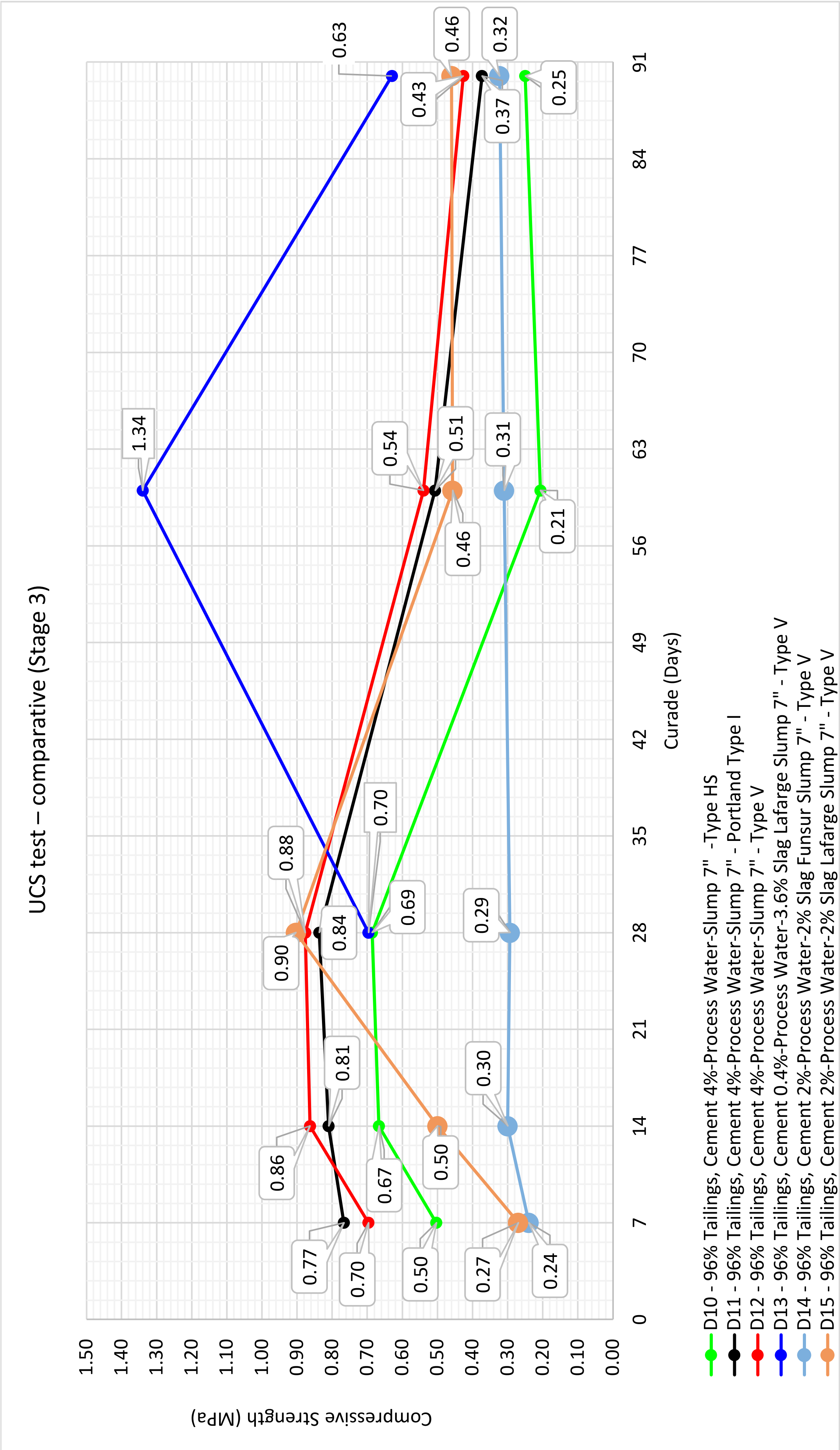

Figure 6 **Results of UCS resistance tests (Stage 3)**

In general, the results of the resistance tests show a tendency of increases during the first 28 days. However, after this time there is a decrease in resistance, bringing it below the initial resistance presented at 7 days of curing.

4 Conclusion

According to the UCS tests carried out, the use of cement alone in the tailings under study was not enough and the tailings needed to be accompanied by slag to keep the resistance reached after 28 days of stability (as can be seen in Figure 6).

In Stage 2 sea water was used, without favourable results, so the use of process water is considered the best mixing element (Figure 5, sample D6). It should be noted that when using process water as a mixing element, the resistance decreases as with the use of sea water, however, it does so to a lesser degree (Figure 5).

Finally, we can confirm that the most appropriate mixture for this type of tailings (which contain a number of sulfates) is tailings 77.5%, solids (%Cw), process water + 2% cement Type V (for high sulfate resistance) and 2% Funsur slag. With this mixture the resistance increases until 28 days of curing and then persists over time (see Figure 6, Test D14).

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