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Use of fibres and surface treatment to improve the durability of concrete affected by sulphide mining

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The aim of this research is to evaluate solutions to improve the durability of structural concrete used in sulphide mining. This type of environment is one of the most aggressive conditions in which a concrete structure can be placed, with pH values below 3 and high sulphate contents. These environments cause significant degradation of metallic and structural materials, resulting in mass loss and alteration of mechanical properties, a process that is accelerated by the intervention of acidophilic bacteria. In this work it is proposed to reinforce the concrete by incorporating polypropylene fibres and silica fume and to protect the specimens by applying two types of surface materials, one polyurethane base and the other asphalt base. Several laboratory tests were carried out to evaluate the fundamental mechanical properties and durability of concrete, including tensile and compressive strength tests In addition, Slake tests were carried out to analyse the degradation of different fragments, water permeability tests and pressure sandblasting tests to measure the behaviour of the concrete against abrasion. The results confirm an increase in the tensile strength of fibre-reinforced concrete of about 8%, while the use of both types of surface materials has been shown a zero water penetration depth, while at the same time significantly improves the performance against impact degradation and abrasion with a reduction in weight loss that obtained in the reference samples with Slake test which is reduced to only 0.45% with surface treatment with polyurethane, 1.01% with asphaltic treatment and 2.48% with fibres, and even no weight loss in the sandblasting tests on the samples treated with asphalt material.

Keywords Acid attack, Sulphates, Degradation, Durability properties, Concrete's mechanical properties

Sulphide mining generates one of the most aggressive environments for the concrete structural elements, mainly due to the high levels of acidity and high sulphate and magnesium content, among others. The processes that explain this phenomenon have been widely described in scientific literature, in which pyrite (FeS_2) reacts with oxygen and water, generating sulphates and Fe^{2+}. The latter is oxidised to Fe^{3+}, in a process accelerated by the acidophilic bacteria found in this type of environment. The ideal conditions for the development of these bacteria are a pH range between 2.5 and 3.5, and temperatures between 15 and 35 °C, conditions that exist for most of the year in the Iberian Pyrite Belt, as well as in other mining environments throughout the five continents.

This process generates waters with pH values that can even be negative[1] and high levels of dissolved metals and metalloids, such as Cu^{2+}, Pb^{2+}, Cd^{2+}, SO_4^{2-}, Fe^{3+}, etc., which are generally referred to as acid mine drainage (AMD). These waters have chemical and biochemical characteristics with a high corrosive power on materials containing Fe or Al[2], which are commonly used in structural and mechanical elements in mining installations.

The durability of metallic and structural materials, and concrete in particular, is affected in these extreme environments by various processes, including physical, chemical and biological phenomena. The severity of the attack on concrete is directly related to the concentration and aggressiveness of the acidic characteristics of AMD and the high concentration of sulphates, which react with the calcium hydroxide ($Ca(OH)_2$) present in the cement[3], leading to the formation of a gypsum layer ($CaSO_4$ $2H_2O$) on the surface of the concrete. The gypsum formed can react with the hydrated tricalcium aluminate to form ettringite. This process causes expansion, cracking, structural weakening of the concrete and consequently a reduction in the overall quality of the material. The dissolution of calcium in the concrete matrix increases the porosity in the outer layers, facilitating the precipitation of gypsum, the erosion of aggregates and the access of water to the interior of the concrete.

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These combined effects lead to a significant loss of the mechanical strength in the concrete, which is exacerbated by the action of microorganisms that accelerate the process of corrosion and deterioration[4].

In addition to the deterioration of concrete and metallic materials when interacting with AMD media, these waters have been shown to be highly polluting, affecting the flora and fauna living in streams and rivers originating from pyrite and coal mining sites[5,6].

Despite significant advances in the study of the durability of structural materials, the interaction between acid mine drainage (AMD) and concrete remains an under-researched area. Although codes for structural concrete have begun to include more stringent requirements to improve durability, few studies specifically address the effects of AMD on concrete.

The few studies that have looked at the issue in depth have produced results that highlight the need to find systems to protect concrete from AMD. Studies show that concrete exposed to acidic leachate in a riverbed suffers a reduction in compressive strength of almost 43% in just six months of exposure[7]. When concrete is exposed to the same type of acidic water, but in a static scenario where there is no water movement, the compressive strength is reduced by 9% and the tensile strength is reduced by 24% for the same exposure time[8].

Although specific studies analysing the effects of AMD on concrete are scarce, it is possible to find work looking at the effects of acidic environments and sulphate exposure on concrete structures. For example, one study investigated the damage caused by sulphate erosion and the use of a silane coating as a mitigation strategy, which significantly reduced the damage[9]. Another paper reviewed the literature on sulphate attack on cement-based materials and concluded that the durability of concrete in acidic environments is a problem that requires special attention[10]. Other publications have examined the problem in specific cases, such as bridges[11] or underground installations of nuclear power plants[12].

This research provides a valuable context for understanding the mechanisms of deterioration in hostile environments and highlights the importance of developing effective methods to improve the durability of concrete in extreme conditions.

There has been some research into alternative protection for concrete in acidic environments, proposing the use of additions such as silica fume, rice husks, aggregates and recycled rubber[13–15]. However, the results obtained to date are neither conclusive nor definitive in terms of improving the resistance of concrete to this type of exposure or providing effective protection, even producing a reduction in compressive and tensile strength, and lower values of the modulus of elasticity were found[16].

Several solutions have also been proposed to improve the mechanical properties and durability of concrete in aggressive environments, such as the addition of fly ash, microorganisms or concrete repair agents, which have shown improvements in the strength of the material[17–19]. Other work has investigated the use of fibres, which have shown some increase in tensile strength and acid resistance[20–22]. However, these solutions, although promising in general acidic environments, have not been sufficiently tested in real AMD conditions. Furthermore, some modifications, such as the partial replacement of fine aggregates with glass cullet[23], have shown improvements in mechanical properties, but at the cost of a reduction in concrete durability, which poses additional challenges. Interesting results have been found in asphalt concrete in an acid water[24], although they showed reductions in tensile strength.

In addition to adding components to the concrete mix to improve its performance against AMD, it is also important to find surface protection systems that limit the interaction of AMD with concrete. Several solutions have been proposed, such as the use of magnesium-based coatings, particularly magnesium oxide and magnesium hydroxide, which have been shown to be effective against sulphuric acid biocorrosion. Several studies[25,26] have evaluated different coatings with different proportions of MgO and $Mg(OH)_2$ exposed to sulphuric acid. The results showed that these coatings maintained the surface pH at alkaline levels (pH values between 9 and 10). These studies show promising results for the protection of concrete against acidic environments; however, they were carried out simulating environments similar to those to which sewer pipes are exposed, with AMD media being even more aggressive. In some of them, the permeability of concrete is analysed as a parameter to evaluate its durability[27] and in the research carried out by[28], graphene oxide was applied as a coating to concrete to reduce the penetration of water and chloride ions. The results showed that a surface-applied graphene oxide coating can reduce water absorption by up to 57% and chloride penetration by 25%, making it a promising solution for protecting concrete structures exposed to wet or marine environments, although its behaviour in acidic environments remains to be investigated. Coatings with a higher NCO/OH ratio showed improved adhesion and film thickness, as well as providing superior protection against acids such as sulphuric acid and saline sodium chloride solutions, making them a viable option for harsh industrial environments. As with previous solutions, no studies were found applying this coating to concrete affected by AMD.

The main objective of this research is to develop and evaluate the effectiveness of three innovative protection systems for concrete exposed to acid mine drainage. The first of these systems will focus on improving the internal composition of the concrete by incorporating fibres to increase the resistance of the concrete mass to acid attack. The other two systems will consist of surface protection treatments designed to act as a barrier to minimise or prevent the direct interaction of AMD with the concrete. These systems will be evaluated in terms of their ability to reduce the chemical and physical degradation of the concrete, as well as their impact on medium and long-term durability.

Materials and methods

Materials used

The base material used was a mass concrete, which has been used in previous research to study the degradation of concrete in extremely acidic environments such as acid mine drainage[7,8]. Polypropylene fibre was used as the reinforcing material and two alternatives were proposed for the protection system through the application of surface materials, one based on polyurethane and the other on asphalt.

Following the indications of the current Building Code, a mass concrete was designed that had to achieve a minimum resistance of 35 MPa, a minimum cement content of 325 kg/m^3 and a maximum w/c ratio of 0.45, as well as the mandatory use of sulphur-resistant cement. To obtain these values required by the current regulations, Eq. 1 of the de la Peña method (1995) was used, starting from the required compressive strength - see Eq. (1) -. All these parameters allow the designed concrete to comply with the requirements of the current UNE-EN 206:2013 + A2:2021, which classifies AMD media as environment XA3, as shown in Table 1.

$$Z = K\ f_{cm} +\ 0.5 \tag{1}$$

.

Where Z is cement-water ratio (c/w) in weight, f_{cm} is the average of concrete strength in MPa at 28 days and K is a parameter that depends on the type of cement and aggregate. In our case it takes the value of 0.045 for the type of cement 42.5.

Once the amount of water in the mix has been determined using Eq. 1, and taking into account the consistency and maximum aggregate size, the amount of aggregate required is determined using Fuller's parabola according to Eq. 2[31].

$$P = 100\sqrt{\frac{d}{D}} \tag{2}$$

.

Where p is the percentage by weight that passes through the sieve, d is the diameter of each sieve, and D is the maximum size of the aggregate.

Using these criteria, a w/c ratio of 0.45 was obtained, the minimum strength obtained in practice was 55 MPa, and the cement content used was 435 kg/m^3, using sulphur-resistant cement of type 42.5. In the specimens made with polypropylene fibre reinforcement, microsilica was used at a rate of 7% of the cement content, fibres at concentrations of 1.7, 3.4 and 6.8 kg/m^3 and superfluidifier at a rate of 1% of the cement weight. All specimens were made with a cylindrical geometry of 100 mm diameter and 200 mm height and cured for 28 days in a temperature-controlled tank. They were then dried at ambient temperature.

Two coatings were used for the external protection. The first was a bitumen emulsion 'Soprema Textop' bituminous emulsion of bitumen and polyurethane with a density of 1.05 kg/L and a tensile adhesion of 0.80 MPa. For the second coat, 'Smart POL' polyurethane with a density of 1.10–1.60 kg/L was used, after having been treated with a primer of an epoxy resin type Sikafloor-150. This resin consists of two components with a density of 1.08 kg/L and a tensile adhesion of more than 1.5 MPa. The polyurethane material itself also consists of two components, one of xylene and the other of a mixture of hexamethylene diisocyanate, xylene, butyl acetate and ethyl benzene. The bituminous material was applied in two coats, while the polyurethane material required three finish coats in addition to the primer, according to the manufacturer's instructions.

SikaFiber M-12 polypropylene fibres, which are manufactured in 12 mm long and 31 µm diameter filaments, were used as concrete reinforcement. These fibres have a density of 0.91 g/cm^3, a tensile strength of 31.9 cN/tex (the strength corresponds to approximately 285 MPa at the specified density. The tensile strength of fibres is defined in cN/tex because it depends on the specific density of the fibres - cN is centinewton; tex represents the mass of 1000 m of fibre in grams) and a modulus of elasticity of 15,000 kg/m^2. According to the result of[32], the use of polypropylene fibres is mainly to improve tensile strength and abrasion resistance. However, the manufacturer states that it also contributes to improved compressive strength.

The concrete specimens prepared as described above were exposed to acidic leachate from the Tharsis mines (Iberian Pyrite Belt, SW Spain). These waters have a pH value below 2.8 and high concentrations of heavy metals, metalloids and sulphates throughout the hydrological year. The most representative average values of this highly aggressive medium are redox potential of 650 mV, electrical conductivity of 10.5 mS/cm, concentration of total dissolved solids of 5.6 mg/L and averages of 1100 mg/L of Fe, of which 400 mg/L are Fe^{2+}, as well as important concentrations of Pb^{2+}, Mg^{2+}, SO_4^{2-}, Cu^{2+}, etc[5].

Methods

The tests carried out were aimed at analysing and validating the protection and reinforcement proposals used in this research. Five types of tests were carried out, the first two of which are mechanical tests to characterise the concrete reinforcement proposals, mainly aimed at evaluating tensile strength, at this is the parameter most affected by AMD exposure[8]. The purpose of these tests is to evaluate the resistance to abrasion in order to assess this parameter with the proposals provided. To this end, a Slake type test was carried out and another by applying a F024 type corundum blast (size between 600 and 850 mm) to the samples, although this test is usually referred

Parameters	XA1 Weak chemical aggressiveness	XA2 Moderate chemical aggressiveness	XA3 Strong chemical aggressiveness	Used water
pH	6.5–5.5	5.5–4.5	< 4.5	< 2.6
Mg^{2+} (mg/L)	300–1,000	1,000–3,000	> 3,000	> 874
SO_4^{2-} (mg/L)	200–600	600–3,000	> 3,000	> 8,513

Table 1. Exposure class based on the chemical aggressiveness of water[29,30].

to as sand blasting. These tests simulate the erosive action of water particles[33,34], a situation that is amplified in AMD media due to the high total dissolved solids content of this type of water. A final test was carried out using a water permeability meter to assess the improvement in the permeability of the concrete, which is of paramount importance in the case of AMD, since the penetration of water through the pores is what facilitates the degradation of the binder and the corrosion of the reinforcement.

A total of 59 concrete specimens were tested, arranged as follows: for the tensile and compressive strength tests, 10 reference specimens and 10 specimens with fibre reinforcement at concentrations of 1.7, 3.4 and 6.8 kg/m^3 were tested. Slake tests were carried out on the reference specimens, on the 3.4 kg/m^3 fibre specimens and on the two external protection specimens. For the sandblasting tests, 2 reference samples, 2 samples with 3.4 kg/m^3 fibre reinforcement and 2 samples with each of the two external treatments were tested. For the water permeability tests, 2 reference specimens and 2 specimens with each of the two external treatments were tested.

Compressive and tensile strength tests

The mechanical characterisation tests were carried out using a Controls press, model Automax X5, which has a load capacity of 3,000 kN for compression tests. A sensitivity of 20 kN was used for these tests, with a speed of 0.6 MPa/s. These tests were used to determine the compressive strength and Young's modulus of the material using strain gauges. In the case of the indirect tensile tests, the load limit was 500 kN, with a sensitivity of 10 kN and a rate of 0.04 MPa/s. Prior to the compression tests, the specimens were ground on the top using a NORMATEST grinder. The whole procedure was carried out in accordance whit the requirements of standards UNE-EN 12390-3:20009 and UNE-EN 12390-6:2010.

Slake type tests

The Slake test is based on ASTM D4644 using Controls equipment. This test consists of placing 10 fragments of the specimens to be tested, each weighing between 40 and 60 g. The fragments are placed in a 100 mm diameter drum made of 2.0 mm stainless steel mesh. These drums rotate at a constant speed of 20 rpm (the total test specimen should weigh 450 to 550 g) immersed in the acid leachate in small tanks.

All fragments were dried at a temperature of 110 °C for 24 h and weighed to determine their water content before and after the test. According to ASTM standards, the test was carried out for 20 min in two cycles of 10 min each. However, in order to evaluate the protection alternatives over a longer period of time, the process was repeated for an additional 100 min to make the result more meaningful in terms of assessing differences. The temperature and pH of the exposure water were measured before and after the test as required by the standards used.

From the difference in weight, the durability index was determined using Eq. (3) according to ASTM D4644.

$$I_d = \left(\frac{W_F - C}{B - C}\right) \cdot C \tag{3}$$

.

Where I_d is the Slake durability index (second cycle until 20 min), B is the mass of drum plus oven-dried specimen before the first cycle, g, W_F *is* mass of drum plus oven-dried specimen retained at the end of the test, g, and C is the mass of drum, g.

Abrasive blast tests

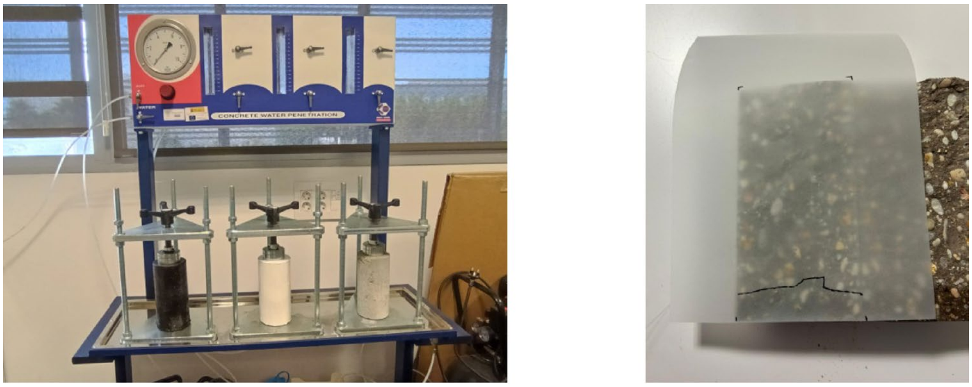
To evaluate the material degradation due to erosion corrosion, abrasive blast tests were carried out, following a procedure similar to that described in the ASTM C418 standard. A blast machine was used, which a compressor pressure of 5 bar maintained continuously for 30 min. The abrasive used was corundum with an Al_2O_3 content of 99.5%, a hardness of 9 Mohs, a bulk density between 1.5 and 2.1 g/cm^3 and a grain size between 600 and 850 µm. The material was applied at a flow rate of 600 g/min. applied at a distance of approximately 70 mm from the sample. This test was only carried out on the specimens with polypropylene fibre reinforcement where the maximum tensile strength was found. This test allowed the weight loss of the treated specimens to be determined and compared with the reference specimens.

Water permeability tests

The concrete permeability test was based on the UNE-EN12390-8:2020 standard using PROETISA equipment, ETI H0330, which allows three tests to be carried out simultaneously (Fig. 1a). The test consists of passing pressurised water through the upper part of the specimen using a compressor. For this purpose, the specimens were previously dried at 110ºC for 24 h and the face of the specimen to be pressurised was devastated. The water pressure during the test was 5 bar and the test lasted 72 h. At the end of the test, the specimen was fractured using an indirect tensile test and depth of water penetration was measured using vegetable paper to represent the water penetration front on the face resulting from the fracture, as shown in Fig. 1b.

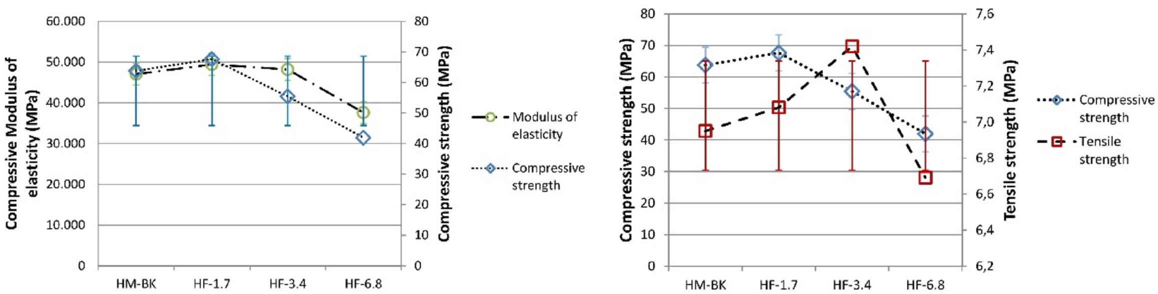
To determine the penetration depth, Eqs. (4) and (5) were used according to UNE-EN12390-8:2020, where *Pm* is the penetration depth rounded to the nearest mm, A_{pf} is the area of the penetration front measured in mm^2, d is the diameter of the samples, M_p is the mass of the initial paper, A_p is the dimensions of the paper (mm) and M_{pf} is the mass of the paper cut to the shape of the penetration front.

$$P_m = \frac{A_{pf}}{d} \tag{4}$$



a) b)

Fig. 1. (**a**) Water penetration test equipment, (**b**) Determination of water penetration front of the face.



a) b)

Fig. 2. Relationship between compressive strength of concrete with different fibre concentrations and the compressive modulus of elasticity (5a) and tensile strength (5b).

$$A_{pf} = \frac{(A_p \cdot M_{pf})}{M_p} \tag{5}$$

Results and discussion

Evaluation of the compressive and tensile strength of the reinforced specimens

Figure 2 shows the mean values obtained in the compressive and tensile strength tests for the reference specimens (HM-BK) and the specimens reinforced with polypropylene fibres at different concentrations (HF-1.7, HF-3.4 and HF-6.8). The figure compares the compressive strength with the modulus of elasticity (Fig. 2a) and the tensile strength (Fig. 2b) of the specimens tested.

The results show an increase in compressive strength and modulus of elasticity of about 6% of the reference value for specimens made with a concentration of 1.7 kg/m^3, and a decrease in strength when specimens are made with concentrations of 3.4 and 6.8 kg/m^3, both parameters following a similar evolution; these results present a small improvement compared to those found by Momotaz et al. in[35]. The tensile strength increases by approximately 7% of the reference value when the specimens are made with a fibre content of 3.4 kg/m^3 and decreases at higher fibre concentrations. Therefore, reinforced specimens with fibre concentrations higher than 3.4 kg/m^3 are no longer effective, the most suitable ones being those with fibre concentrations between 1.7 and 3.4 kg/m^3.

Regarding tensile strength, the results are similar to those obtained by other authors, with slight variations in the concentration at which the maximum resistance is obtained. However, the general behaviour remains consistent, with a maximum value starting at a certain fibre concentration, followed by a decrease in strength as the concentration increases further[22,36]. This is also consistent with the manufacturer's claims. However, regarding compressive strength, the manufacturer claims that it improves by a small percentage with the addition of polypropylene fibres. This is not in line with the results obtained in this study.

Evaluation of the wear degradation of the proposed methods

To study the wear that would occur in the concrete with the different proposed treatments, the results obtained in the Slake test and in the abrasive blast machine were evaluated.

Table 2 shows the results obtained in the Slake test for 10 fragments from the reference specimens (RefC), those made with fibres at a concentration of 3.4 kg/m^3 (FibC), the specimens with polyurethane coating (PolC) and those with asphalt coating (AsphC). The test was carried out on the samples belonging to the 3.4 kg/m^3

	Reference concrete (RefC)			Concrete with fibres (3.4 kg/m^3 - FibC)			Treated concrete with polyurethane (PolC)			Treated concrete with asphaltic (AsphC)		
	W_0	W_{20}	W_{120}	W_0	W_{20}	W_{120}	W_0	W_{20}	W_{120}	W_0	W_{20}	W_{120}
M_1	40.00	38.82	38.46	40.05	38.81	37.95	46.47	46.32	46.22	40.76	40.52	40.42
M_2	40.10	39.27	38.69	41.00	39.85	39.24	46.92	46.80	46.72	44.66	44.36	44.21
M_3	54.49	53.6	52.44	45.50	45.11	44.91	48.85	48.69	48.55	46.37	45.99	45.84
M_4	55.31	54.54	54.14	50.54	49.38	48.72	49.30	49.18	49.1	46.66	46.25	46.11
M_5	56.63	55.78	54.28	55.64	54.78	54.42	52.69	52.56	52.47	49.21	48.91	48.80
M_6	57.62	55.81	54.55	56.13	55.26	54.73	55.29	55.15	55.05	52.98	52.61	52.50
M_7	57.65	56.52	55.30	56.44	55.74	55.21	58.55	58.41	58.31	53.92	53.52	53.27
M_8	57.67	56.61	55.86	57.24	56.69	56.08	58.83	58.68	58.56	56.82	56.38	56.25
M_9	57.93	56.86	56.59	57.42	56.41	56.09	59.76	59.59	59.49	59.44	59.03	58.83
M_{10}	59.24	58.29	57.73	57.44	56.76	56.34	59.89	59.74	59.65	59.60	59.21	59.04
Small fraction	0.00	0.64	2.51	0.00	0.17	0.89	0.00	0.00	0.00	0.00	0.00	0.00
Total weight (g.)	536.64	526.74	520.55	517.35	508.96	504.57	536.55	535.12	534.12	510.42	506.75	505.27

Table 2. Weights determined with the slake apparatus at different times, where W_0 is the initial weight of the samples, W_{20} is the weight after 20 min and W_{120} is the weight after 120 min of testing. All values are expressed in grams.

	W_0	W_{20}	W_{120}	$I_{d,20}$	$I_{d,120}$	pH_0	pH_{120}	T_0	T_{120}
RefC	536.64	526.74	520.55	98.16	97.00	2.59	2.86	19.4	18.9
FibC	517.39	508.96	504.57	98.37	97.52	2.60	2.79	20.4	20.9
PolC	536.55	535.12	534.12	99.73	99.55	2.54	2.54	20.1	21.2
AsphC	510.42	506.78	505.27	99.29	98.99	2.59	2.56	19.4	18.2

Table 3. Parameters obtained in the slake test. Mass values expressed in grams, temperature in ºC and slake index in %.

fibres specimens, as they had the highest tensile strength. The Table 2 shows the individual weights of each of the 10 fragments before the test, after 20 min and after 120 min of testing. The total weight of the smallest fraction retained in the drum (greater than 2 mm) remaining in the drum from the disintegration of the pieces initially introduced is also given, as is the total weight of the sum of the 10 resulting fragments.

After 20 min of testing, the reference material fragments lose between 1.4 and 3.1% of their mass, the fibre material between 0.8 and 3.1%, the polyurethane coating material between 0.2 and 0.3% and the asphalt coating material between 0.6 and 0.9%. After 120 min of testing the mass loss of the fragments is between 2.1 and 5.3% for the reference material, between 1.3 and 5.2% for the fibre material, between 0.4 and 0.6% for the polyurethane coating, and between 0.8 and 1.2% for the asphalt coating.

After 120 min of testing, the amount of disaggregated reference material larger than 2 mm is 0.5% and 2.5% of disaggregated particles smaller than 2 mm. For the material with fibres these values are 0.2% and 2.3% of particles larger and smaller than 2 mm, respectively. The coated fragments do not lose mass due to disintegration of particles larger than 2 mm, but do lose mass of smaller particles, being 0.5% and 1% for the polyurethane and asphalt coatings, respectively.

These results indicate a significant improvement in the wear of the material with the three proposed treatments, highlighting that in the two samples with external protection, the small disintegrated fraction was zero, demonstrating the effectiveness of the treatment against erosion processes. The smallest fractions are also significantly reduced with fibre reinforcement. This shows that there was no loss of concrete mass, especially with the external treatment, possibly due to a surface loss of the coating material.

Table 3 shows the results of the total mass of the fragments studied at 20 and 120 min, as well as the Slake indices obtained ($I_{d,20}$ and $I_{d,120}$), the temperatures measured (T_0 and T_{120}), and the evolution of the pH of the acidic water used (pH_0 and pH_{120}).

The values obtained for the Slake index at both 20 and 120 min indicate that the treatments with external protection reduce the weight loss with respect to that of unprotected mass concrete from 1.84% and 3.00–0.71% and 1.01% in the case of asphalt treatment and to only 0.27% and 0.45% using polyurethane, respectively for 20 and 120 min cycles. When the concrete is reinforced with polypropylene fibres, the weight loss is 1.63% and 2.48% for 20 and 120 min, respectively. If the volume loss of the samples is evaluated (eliminating the small fraction) and taking into account the specific weight of each material, the mass concrete fragments would lose 0.51%, those reinforced with fibres 0.39%, those treated with polyurethane 0.11% and, finally, those protected with asphalt material 0.32% of the initial volume. The calculated Slake indices indicate that the most effective treatment against concrete erosion is the one treated with polyurethane coating, followed by the one treated with

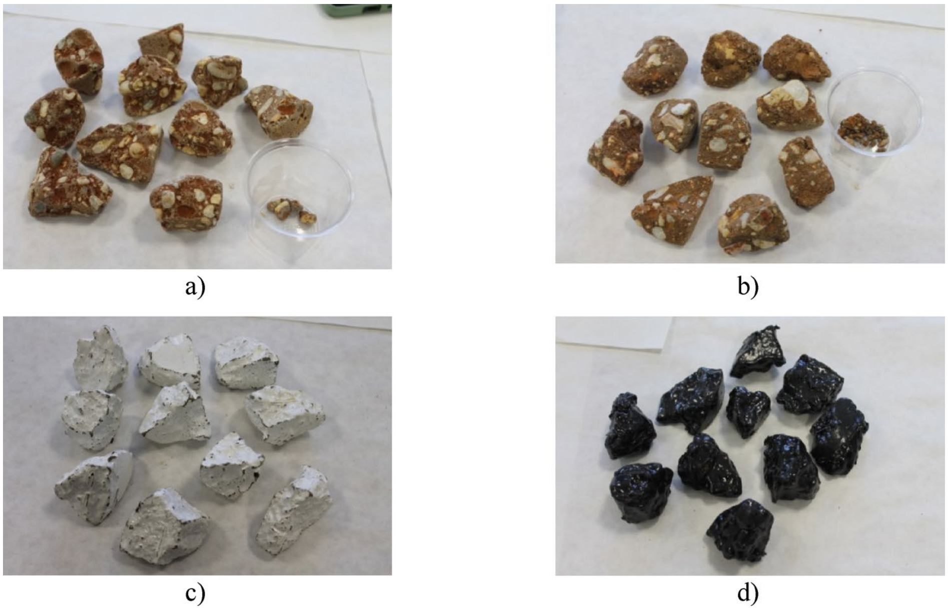


Fig. 3. Final condition of the simples in the Slake test: RefC (**a**); FibC (**b**); PolC (**c**); AsphC (**d**).

	RefC	FibC	PolC	AsphC
Slake	3.00%	2.48%	0.45%	1.01%
Slake no small fraction	3.47%	2.65%	0.45%	1.01%
Sandblast abrasion	0.56%	0.03%	0.13%	0.00%

Table 4. Slaking versus sandblasting results.

asphalt coating. Some research[37] has shown some improvement in the Slake Index by incorporating coal mine waste as a partial replacement for fine aggregate.

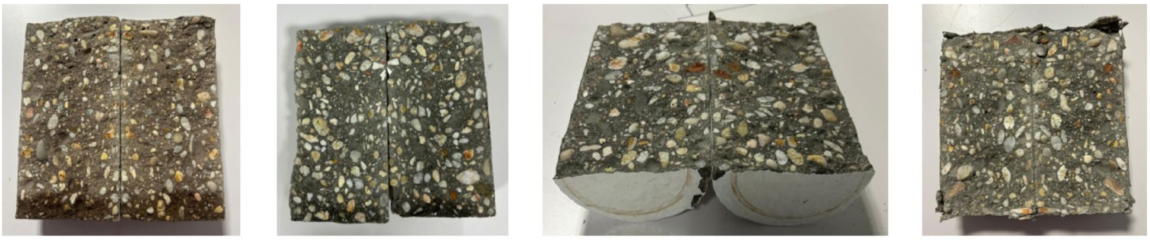
The photographs in Fig. 3 show the final state of the fragments after 120 min of testing. In the specimens without external protection, a reddish colour change can be seen after the test, due to the interaction with the AMD, possibly due to the precipitation of iron oxides[7,8], this effect being more pronounced in the reference specimens, a phenomenon that does not occur in the specimens with external protection. On the other hand, the samples without external protection have more rounded edges, whereas the edges of the samples protected with polyurethane and asphalt material are less affected. This is consistent with the amount of small material obtained in the tests and shown in Table 3.

After 30 min of exposure to the abrasive material in the sandblaster, the results were quite significant. The weight loss of the reference specimens was 0.56%, while the specimens reinforced with polypropylene fibre at a concentration of 3.4 kg/m^3 lost only 0.13% of their mass. The specimens with external protection suffered less wear, with a weight loss of 0.03% for those protected with polyurethane and zero weight loss for those protected with asphalt material.

These results indicate that all the proposed treatments significantly improve the abrasive wear of the concrete. Taking into account the specific weight of each material, the samples treated with polypropylene fibre would lose approximately 0.56 cm^3 of their volume, compared to a volume loss of 3.50 cm^3 for the samples protected with polyurethane and a loss of 8.75 cm^3 for the reference samples.

Table 4 shows the results obtained in the Slake test and in the sandblaster abrasion test. The results of the Slake test are also shown, excluding the weight of the small fraction obtained in the reference concrete samples and in the samples with fibre at the end of the 120-minute test period, since it is understood that this small material can no longer be considered concrete, nor would it offer any resistance.

Comparing the results obtained in the Slake test with those obtained in the sandblast abrasion test, it can be affirmed that, in general, the treatment with the best performance against degradation due to erosion is the concrete with external polyurethane protection. Therefore, if the samples with this treatment were also reinforced with polypropylene fibres at a concentration of 3.4 kg/m^3, an additional improvement against abrasion would be obtained, as well as an increase in the tensile strength. The results obtained in the sandblaster test on fibre-reinforced concrete with asphalt treatment are better than those found by[38] with the addition of silica fume and fly ash to the concrete, and slightly worse in the case of polyurethane treatment.



a) b) c) d)

Fig. 4. Indirect tensile test specimens showing the penetration front: RefC (**a**), FibC (**b**), PolC (**c**), AsphC (**d**).

Water permeability of the proposed treatments

The results obtained in the water permeability tesst of the treated concrete specimens were very significant. Figure 4 shows the photographs of the specimens after the indirect tensile test, where the penetration front can be observed.

In the reference samples, a penetration surface area of 2587.56 mm^2 and a penetration depth of 25.88 mm were obtained. For the samples treated with polypropylene fibres at a concentration of 3.4 kg/m^3, the penetration depth was 13.97 mm and the frontal area was 1396.72 mm^2, almost 46% less than for the reference sample. The sample with external protection showed a penetration depth of zero. Water permeability has been improved by using recycled concrete and aggregates from alluvial soil[39].

These results are considered to be of great importance, since preventing the access of acidic water to the interior of the concrete increases its durability by preventing not only the deterioration of the binder, but also the ingress of microorganisms that accelerate the opening and propagation of cracks[8].

Conclusions

In this work, different reinforcement and protection alternatives for mass concrete against the highly aggressive environment known as acid mine drainage have been evaluated: reinforcement with polypropylene fibres at different concentrations and external protection with polyurethane material and asphalt material.

From the results obtained, it can be concluded that the samples reinforced with polypropylene fibres have a maximum tensile strength at a fibre concentration of 3.4 kg/m^3 and a modulus of elasticity very close to the maximum. For the compressive strength, the highest value was obtained with a fibre concentration of 1.7 kg/m^3. Although the increase in tensile strength is not very large, considering that this parameter is very low in concrete, any increase is significant, and other alternatives could even be tested in the future.

The best durability performance, as determined by the Slake test, was obtained by the polyurethane-protected concrete specimens with a Slake index at 120 min of 99.55%, followed by the asphalt-treated specimens with a Slake index of 98.99%. The specimens treated with external protection showed a zero penetration depth against water, which in both cases would guarantee that acidic water could not penetrate the concrete and therefore the material could not be attacked by this aggressive medium. In the case of fibre-reinforced concrete, a significant improvement against water penetration was also confirmed, as the penetration depth was reduced by half. In the case of abrasion, the best solution was found to be protection with asphalt material followed by treatment with fibre reinforcement, and a significant improvement was also found in the samples protected with polyurethane.

Although all the proposed treatments have been confirmed as valid solutions, the best alternative would be to add polypropylene fibre.03 reinforcement at a concentration of 3.4 kg/m3 to the exterior polyurethane treatment. This would improve both the tensile strength—a very important factor, considering that this parameter is the most affected in acidic environments—and the durability of the material, improving its abrasion resistance and preventing the penetration of water, creating a total barrier against the access of acidic water to the inside of the pores and therefore represent a very effective treatment against these highly aggressive AMD environments.

Data availability

All data generated or analysed during this study are included in this published article.

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References

1. Sarmiento, A. M. et al. Negative pH values in an open-air radical environment affected by acid mine drainage. Characterization and proposal of a hydrogeochemical model. *Sci. Total Environ.* **644**, 1244–1253. https://doi.org/10.1016/j.scitotenv.2018.06.381 (2018).
2. Nordstrom, D. K. & Alpers, C. N. Negative pH, efflorescent mineralogy, and consequences for environmental restoration at the Iron mountain superfund site, California. *Proc. Natl. Acad. Sci.* **96**, 3455–3462. https://doi.org/10.1073/pnas.96.7.3455 (1999).
3. Ekolu, S. O., Diop, S., Azene, F. Z. & Mkhize, N. Disintegration of concrete construction induced by acid mine drainage attack. *J. South. South. Afr. Institution Civil Eng.* **58**, 34–42. https://doi.org/10.17159/2309-8775/2016/v58n1a4 (2016).
4. Ekolu, S. O., Azene, F. Z. & Diop, S. A concrete reactive barrier for acid mine drainage treatment. *Proceding Institution Civil Eng. - Water Manage.* **167**, 373–380. https://doi.org/10.1680/wama.13.00035 (2014).

5. Sarmiento, A. M., Nieto, J. M., Olías, M. & Cánovas, C. R. Hydrochemical characteristics and seasonal influence on the pollution by acid mine drainage in the Odiel river basin (SW Spain). *Appl. Geochem.* **24**, 697–714. https://doi.org/10.1016/j.apgeochem.2008.12.025 (2009).
6. Galhardi, J. A. & Bonotto, D. M. Hydrogeochemical features of surface water and groundwater contaminated with acid mine drainage (AMD) in coal mining areas: a case study in Southern Brazil. *Environ. Sci. Pollut. Res.* **23**, 18911–18927. https://doi.org/10.1007/s11356-016-7077-3 (2016).
7. Davila, J. M. et al. Determination of the extreme reduction of concrete strength due to acid mine drainage by laboratory tests on specimens located in a real environment. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2020.121817 (2021).
8. Davila, J. M., Sarmiento, A. M., Grande, J. A. & Luís, A. T. Different evolution of tensile and compressive strength in concrete affected by acid mine drainage. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2023.132351 (2023).
9. Liu, D. et al. Comprehensive testing of sulfate erosion damage of concrete structures and analysis of silane coating protection effect. *Sensors* https://doi.org/10.3390/s22207991 (2022).
10. Zhang, C., Li, J., Yu, M., Lu, Y. & Liu, S. Mechanism and performance control methods of sulfate attack on concrete: A review. *Materials* https://doi.org/10.3390/ma17194836 (2024).
11. Zhang, Y. et al. Study on the resistance of concrete to high-concentration sulfate attack: A case study in Jinyan Bridge. *Materials* https://doi.org/10.3390/ma17143388 (2024).
12. Qiao, D., Matsushita, T., Maenaka, T. & Shimamoto, R. Long-term performance assessment of concrete exposed to acid attack and external sulfate attack. *J. Adv. Concr. Technol.* **19**, 796–810. https://doi.org/10.3151/jact.19.796 (2021).
13. Yasser, N., Abdelrahman, A., Kohail, M. & Moustafa, A. Experimental investigation of durability properties of rubberized concrete. *Ain Shams Eng. J.* https://doi.org/10.1016/j.asej.2022.102111 (2023).
14. Dezhampanah, S. et al. Environmental performance and durability of concrete incorporating waste tire rubber and steel fiber subjected to acid attack. *J. Clean. Prod.* https://doi.org/10.1016/j.jclepro.2020.122216 (2020).
15. Mahedi, M., Dayioglu, A. Y., Cetin, B. & Jones, S. Remediation of acid mine drainage with recycled concrete aggregates and fly ash. *Environ. Geotechnics*. **11**, 15–28. https://doi.org/10.1680/jenge.19.00150 (2024).
16. Sofi, A. Effect of waste tyre rubber on mechanical and durability properties of concrete – A review. *Ain Shams Eng. J.* https://doi.org/10.1016/j.asej.2017.08.007 (2018).
17. Han, M. & Li, J. Enhancement of compressive strength and durability of sulfate-attacked concrete. *Buildings*. https://doi.org/10.3390/buildings14072187 (2024).
18. Singaram, K. K., Khan, M. A. & Talakokula, V. Review on compressive strength and durability of fly–ash–based geopolymers using characterization techniques. *Archives Civil Mech. Eng.* https://doi.org/10.1007/s43452-025-01116-7 (2025).
19. Moghaddam, S. C., Madandoust, R., Jamshidi, M. & Nikbin, I. M. Mechanical properties of fly ash-based geopolymer concrete with crumb rubber and steel fiber under ambient and sulfuric acid conditions. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2021.122571 (2021).
20. Fode, T. A., Jande, Y. A. C., Kivevele, T. & Rahbar, N. Effect of waste water bottle and treated Sisal fibers on the durability and mechanical properties of concrete. *Sci. Rep.* https://doi.org/10.1038/s41598-025-92306-z (2025).
21. Dashti, J. & Nematzadeh, M. Compressive and direct tensile behavior of concrete containing Forta-Ferro fiber and calcium aluminate cement subjected to sulfuric d attack with optimized design. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2020.118999 (2020).
22. Zhang, X., Yin, R., Chen, Y. & Lou, C. Experimental study on the axial tensile properties of polypropylene fiber reinforced concrete. *Sci. Rep.* https://doi.org/10.1038/s41598-023-43723-5 (2023).
23. Tanwar, V., Bisht, K., Ahmed Kabeer, K. I. S. & Ramana, P. V. Experimental investigation of mechanical properties and resistance to acid and sulphate attack of GGBS based concrete mixes with beverage glass waste as fine aggregate. *J. Building Eng.* https://doi.org/10.1016/j.jobe.2021.102372 (2021).
24. Feng, S., Wang, W., Tang, H., Xue, M. & Höeg, K. Effects of acidic water on hydro-mechanical properties of asphalt concrete. *Case Stud. Constr Mater*. (2020). https://doi.org/10.1016/j.cscm.2020.e00445.
25. Merachtsaki, D., Tsardaka, E. C., Anastasiou, E. & Zouboulis, A. Anti-corrosion properties of magnesium oxide/magnesium hydroxide coatings for application on concrete surfaces (sewerage network pipes). *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2021.125441 (2021).
26. Merachtsaki, D., Tsardaka, E. C., Anastasiou, E. & Zouboulis, A. Evaluation of the protection ability of a magnesium hydroxide coating against the bio-corrosion of concrete sewer pipes, by using short and long duration accelerated acid spraying tests. *Materials* https://doi.org/10.3390/ma14174897 (2021).
27. Aydin, M. & Bulut, H. A. Evaluation of the effect of E–waste on the permeability properties of polymer concrete composites and their behavior in aggressive environments. *Archives Civil Mech. Eng.* https://doi.org/10.1007/s43452-024-01006-4 (2024).
28. Korayem, A. H., Ghoddousi, P., Javid, A. A. S., Oraie, M. A. & Ashegh, H. Graphene oxide for surface treatment of concrete: A novel method to protect concrete. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2020.118229 (2020).
29. Codigo Estructural. BOE núm. 190, de 10 de agosto de, Ministerio de la Presidencia, Relaciones con las Cortes y Memoria Democratica. Spain. (2021).
30. ACI Committee 318, Building code requirements for structural concrete (ACI318-14) and commentary. (American Concrete Institute, 2014).
31. Fernández, M. & Hormigón *5ª Edición Adaptado a La Instrucción De Cementos RC-97 Y a La Instrucción De Hormigón Estructural EHE-98* (1999).
32. Kakooei, A., Akil, H. M., Jamshidi, M. & Rouhi, J. The effects of polypropylene fibers on the properties of reinforced concrete structures. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2011.08.015 (2012).
33. Kumar, G. R. & Sharma, U. K. Standard test methods for determination of abrasion resistance of concrete. *Int. J. Civil Eng. Res.* **5**, 155–162 (2014).
34. Warudkar, A. & Elavenil, S. A comprehensive review on abrasion resistance of concrete. *J. Appl. Sci. Eng.* **17**, 23–43. https://doi.org/10.1016/10.6703/IJASE.202003_17(1).029 (2020).
35. Momotaz, H. et al. Changes in flexural, tensile and impact characteristics of Kerb concrete due to the addition of tyre-derived aggregates and polypropylene fibres. *J. Building Eng.* https://doi.org/10.1016/j.jobe.2024.108438 (2024).
36. Hasan, A. H., Maroof, N. R. & Ibrahim, Y. A. Effects of polypropylene fiber content on strength and workability properties of concrete. *Polytechnic J.* https://doi.org/10.25156/ptj.v9n1y2019.pp7-12 (2019).
37. Jena, B., Sarkar, P. & Karak, S. K. Effect of coal mine as partial replacement of fine aggregates on resistance and slake durability of hardened concrete. *Int. J. Coal Preparation Utilization*. https://doi.org/10.1080/19392699.2025.2495983 (2025).
38. Cai, X., He, Z., Tang, S. & Chen, X. Abrasion erosion characteristics of concrete made with moderate heat Portland cement, fly ash and silica fume using sandblasting test. *Constr. Build. Mater.* **127**, 804–814. https://doi.org/10.1016/j.conbuildmat.2016.09.117 (2016).
39. Lin, L. & Wu, B. Water permeability behavior of recycled lump/aggregate concrete. *Constr. Build. Mater.* https://doi.org/10.1016/j.conbuildmat.2022.126508 (2022).

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Author contributions

Davila JM: conceptualization, methodology, investigation, formal analysis; Rodriguez-Gomez C: acquisition of data, formal analysis, writing - original draft; Sarmiento AM: writing - review and editing, supervision, resources, project administration.

Declarations

Competing interests

The authors declare no competing interests.

Additional information

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