DURABILITY OF POLYESTER POLYMER CONCRETES BASED ON METALLURGICAL WASTES FOR THE MANUFACTURE OF CONSTRUCTION AND BUILDING PRODUCTS

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ABSTRACT

Varied target materials can be incorporated into polyester polymer concretes (PPC), as the polymeric matrix and target particles tend not to react. This allows natural aggregates to be substituted for different waste products in non-structural polymeric construction and building materials. Many studies have explored the mechanical properties of waste-based PPC, but the durability and surface resistance of these materials is not fully known. Therefore, in this study, we compared the suitability of two metallurgical wastes to that of two natural aggregates for manufacturing durable PPC products. Three aspects were tested: durability against environmental conditions, durability against chemical products, and surface strength against physical damage. Durability against environmental conditions was characterised according to visual damage and mechanical strength losses after freezing-thawing; no PPC combinations exhibited surface damage following the ageing cycles. The ladle slag (LS) samples exhibited the best pre- and post-test flexural and compressive strength. The properties of the alumina filler (AF) combinations were initially similar to those of the natural aggregates, but they exhibited the highest flexural and compressive strength losses after freezing-thawing. Resistance against chemical substances was related to the target material used. The calcareous sand combinations were damaged most severely by acid, while LS and AF exhibited good resistance against chemical substances. The LS combinations exhibited the highest surface strength against impacts in the rebound number test, while the results obtained for the AF combinations were close to those of natural aggregates. Finally, the surface resistance against scratching

depended on the resin, not on the target material. These results demonstrate the potential of these recycled target materials in manufacturing durable and more sustainable PPC products.

KEYWORDS

Polyester polymer concrete; resin; ladle slag; alumina filler; metallurgical waste; construction material durability

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1. INTRODUCTION

The use of polyester polymer concrete (PPC) for manufacturing construction and building products is increasing due to their quick setting and hardening times, high mechanical strength, low permeability, and excellent resistance against chemical products [1–6]. A wide range of materials can also be incorporated into PPC as there is typically no reactivity between the surrounding polymer matrix and target particles. Therefore, the substitution of natural aggregates in PPC manufacturing is an effective method of the valorisation of waste materials, which conserves natural resources and promotes the production of environmentally sustainable construction products, in accordance with the circular economy principles [2,3,7–15]. Metallurgical slags have high potential for use in PPC manufacturing, as they have been employed in the production of conventional concrete and mortar [16–19]. . The manufacturing of non-structural construction and building pre-casts as urban furniture and façade panels, could benefit from waste-based PPC, as these products are expensive and widely consumed, and their manufacture likely consumes a high amount of natural aggregates. Importantly, these products are non-structural. As such, the concrete used in their manufacture is not subject to the strict regulations of structural concrete.

Extensive studies have been conducted on the relationship between the strength and durability of PPC and the use of wastes as target materials by comparing them to conventional concrete [8,10–12,15,20,21]. Durability against environmental conditions or chemical products and surface resistance against impact or scratches are important

characteristics of non-structural construction and building precast materials. Therefore, this study analyses the durability of metallurgical waste-based PPC for use in the manufacturing of non-structural construction and building precasts. An experiment was conducted using PPC mortar composed of natural and recycled target materials with a polyester resin. The physical and chemical resistance of PPC mortars was characterised by conducting conventional mortar-specific tests and tests specifically developed for other materials that could be representative of the durability requirements.

2. MATERIALS AND METHODS

In this study, different PPC combinations were considered. Laboratory tests were conducted using 40×40×160-mm prismatic samples that were manufactured using a commercial polyester mixture supplied by *Castro Composites Co*. The main liquid and cured characteristics of this resin are shown in Table 1.

TABLE 1

The commercial resin was specifically developed for manufacturing PPC and is marketed as CRYSTIC 406 NT. Two types of metallurgical waste were considered as non-conventional aggregates: Ladle Slag (LS) and Alumina Filler (AF). LS is also referred to as “secondary refining” or “white” slag, and is a sandy slag produced in transport ladles during the production of iron or steel from scrap-iron in electric arc furnaces. In Spain, the annual production of LS exceeds 340,000 tonnes. The LS used in this study was provided by *Construcciones y Auxiliar de Ferrocarriles Co.* (Spain). AF is a secondary waste obtained during the valorisation of aluminium salt slag. Approximately 300,000 tonnes of AF waste are generated in Europe per year. The AF used in this study was provided by *BEFESA Co*. (Spain). There are no effective methods of valorisation and

large volumes of both wastes are discarded in landfills each year, as this is the only available system of managing them [22,23]. As references, siliceous and calcareous sands obtained by crushing rocks were used as natural target materials. Figure 1 shows the granulometric curves of the considered target materials.

FIGURE 1

All target materials were dried in a laboratory oven at 100 ºC for 24 hours to fully remove moisture from the samples as polyester resins and water are incompatible.

The samples were manufactured as follows: the resin and target material were thoroughly mixed using an electric drill for five minutes. The cobalt catalyst was then added, and the sample was mixed for a further five minutes to ensure homogenization. The sample was then poured into 40×40×160-mm prismatic moulds coated with a commercial mould-release agent. The filled moulds were shaken on a vibrating table for three minutes to remove any air bubbles from the samples and to ensure that the moulds were properly filled. To determine the optimum resin dosage range for each target material, samples with different resin and target material combinations were prepared by changing the resin content by 5% of the total sample mass. After preparation, the samples were stored under typical room conditions for one hour before de-moulding. Once they had been unmoulded, the samples were cured for 24 h in an oven at 40 ºC to ensure complete resin polymerisation and consistent curing conditions. The samples were then stored at room temperature for seven days before testing. Each combination was denoted with letters that identified the aggregate type (S for siliceous sand, C for calcareous sand, LS for ladle slag, and AF for alumina filler), followed by the resin content, which was expressed as the mass percentage of the total mix. Table 2 shows the manufactured PPC combinations

as well as their main physical properties and strength, which was measured in our previous investigation. The resin contents varied due to the different workability requirements of each target material.

TABLE 2

The strength of PPC exceeds the non-structural construction and building precast requirements; however, its durability and surface resistance remain unknown. Therefore, three test categories were considered to characterise the PPC samples: (i) durability against environment, (ii) resistance to chemical products, and (iii) surface resistance to physical damage. Durability against environmental conditions is often characterised by conducting freezing-thawing tests, according to European Standard EN 14617-5. In this test, six mortar samples were subjected to 25 cycles of freezing followed by thawing in water. After the ageing cycles, the samples were visually surveyed to identify any physical damage. Flexural and compressive strength tests were conducted to determine any changes in the mechanical strength of the materials according to standards EN 12390-5 and EN 12390-13, respectively.

The resistance of PPC to chemicals was characterised according to standard EN ISO 10545-13 which was developed for ceramic tiles and considers how different dissolutions and concentrations of aggressive chemical substances affect the surface of a material. To avoid the expected protection offered to the PPC surface by the resin and to ensure that the chemical treatments were representative, these tests were conducted by immersing 3.5 mm-thick slices of the prismatic samples in the different solutions. After the PPC samples were immersed in the chemical product solutions, they were cleaned with deionised water, dried with a cloth, and stored for 24 h under laboratory conditions. Damage to the

samples was identified by visual inspection and categorised into five levels: No surface damage (0), slight change in surface colour (1), change in surface colour (2), change in surface colour with particle loss (3), and change in surface colour with particle loss and efflorescence (4).

The surface strength against physical damage represents the resistance of the surface of the material to impacts and scratching. Therefore, resistance against impacts was characterised based on the rebound number test according to EN 12504-2. This test was conducted using a Smidt hammer (esclerometer), which measures the surface strength of materials based on the rebounds observed when a sample is struck by a spring-loaded piston. To ensure representativeness, three different positions on three faces of five samples were tested. The surface resistance against scratching was determined based on the scratch hardness according to the Mohs scale test, as defined in EN 15771. Therefore, the Mohs hardness was denoted as the first substance in increasing order that could scratch three different parts of the surfaces of three PPC samples.

3. RESULTS AND DISCUSSION

3.1. DURABILITY AGAINST THE ENVIRONMENT

Following the freezing and thawing cycles, none of the samples exhibited any surface damage. Figure 2 shows the flexural mean strength and standard deviations of the samples before and after the accelerated ageing test.

FIGURE 2

The flexural strength of all samples containing the target materials before freezing and thawing was lower than that of the resin. These results are consistent with those obtained by Fink [24], who stated that different fillers could influence the mechanical properties of cured materials. The lower flexural strength could be related to the size, shape, and distribution of the aggregates, which would create discontinuities in the resistant polymer matrix[25]. Zhang and Singh [26] suggested that these discontinuities could be increased by the poor adhesion of target particles to the resin. After the thawing and freezing cycles, the flexural strength of resin samples decreased from 53.0 to 25.1 MPa, demonstrating that the flexibility of resin is highly sensitive to freezing and thawing. The loss of the flexural strength of the samples containing siliceous and calcareous sand slightly decreased as the resin content of the samples increased. The flexural strengths of samples S-20, S-25, and S-30 before and after the ageing test were 22.0 and 19.5 MPa, 25.8 and 23.5 MPa, and 25.1 and 24.5 MPa, respectively. Therefore, S-30 was the most durable, with a decrease in the flexural strength of 2.4%. The flexural strengths of samples C-20, C-25, and C-30 before and after ageing were 24.7 and 22.7 MPa, 23.5 and 21.9 MPa, and 21.0 and 21.3 MPa, respectively. Similar to the siliceous sand samples, the loss of flexural strength in the calcareous sand samples decreased as the resin content increased. The flexural strength of sample C-30 increased by 1.6%, which is likely due to differences in the manufacture of the samples. The good flexural strength of the siliceous and calcareous sand samples could be related to their lower resin and fine particle contents, which would create a dense and stable matrix under changes in temperature. Among the target materials, the LS samples exhibited the highest flexural strengths. Before testing, the flexural strengths of LS-35, LS-40, and LS-45 were 43.6, 44.5, and 45.2 MPa, respectively. After ageing, these strength values decreased by 25.2%, 12.4%, and 23.8%, respectively. However, they remained the most resistant combinations, with flexural

strengths of 32.6, 39.0, and 34.5 MPa for LS-35, LS-40, and LS-45, respectively. Before the ageing test, the flexural strengths of samples AF-35, AF-40, and AF-45 were 23.7, 25.5, and 24.1 MPa, respectively. After the freezing-thawing cycles, their flexural strengths decreased to 10.5, 13.7, and 17.0 MPa, which are resistance losses of 55.7%, 46.3%, and 29.5%. This indicates an indirect relationship with the resin content. The decreases in the flexural strength of the LS and AF samples could be related to their higher fine particle and resin contents, which would result in materials that are less durable.

FIGURE 3

Figure 3 shows the mean compressive strengths of the samples and standard deviations before and after the freezing and thawing cycles. Before the ageing test, the compressive strength of the calcareous sand and LS samples exceeded that of resin, while that of the siliceous sand and AF samples was lower. These differences could be due to the particle size, shape, and distribution of the target materials, as well as the resin content [27,28]. After freezing and thawing, the compressive strength of the resin sample increased by 25.2% from 105.3 to 131.8 MPa. Considering the flexural strength, this suggests that freezing and thawing caused the resin matrix to become more rigid and resistant to compression. This beneficial effect of ageing was observed for all target materials excluding AF, as their compressive strengths were maintained or slightly increased after the ageing test. The compressive strength of the siliceous sand samples varied between 93.4 and 106.2 MPa before the ageing cycles, indicating a direct relationship between the resin content and compressive strength. Following the ageing cycles, the compressive strength of S-20 decreased by 3.2%, while that of S-25 and S-30 increased by 0.8% and

6.7%, respectively. Before ageing, the compressive strengths of C-20, C-25, and C-30 were 111.3, 116.7, and 118.4 MPa, respectively. After ageing, their compressive strengths were 113.1, 117.8, and 116.0 MPa, respectively, indicating increases of 1.6% and 0.9% for C-20 and C-25, respectively, and a decrease of 2% for C-30. Therefore, there was a weak, indirect relationship between the durability and resin content of these samples. Similar to the flexural strength, the LS samples achieved the best compressive strength before and after ageing, with values of 153.7 and 152.6 MPa, 155.6 and 153.9 MPa, and 160.5 and 156.1 MPa for LS-35, LS-40, and LS-45, respectively, which are decreases of 0.7%. 1.1%, and 2.7%. Therefore, there was a direct relationship between the resin content and compressive strength of the samples. The compressive strengths of the AF samples were between 104.2 and 87.4 MPa, and decreased by 61%, 45.3%, and 12.7% after freezing and thawing for samples AF-35, AF-40, and AF-45, respectively. Therefore, similar to the flexural strength, the compressive strength decreased significantly, which was indirectly related to the resin content of the samples and indicated that the PPC-AF matrix was not durable.

3.2. DURABILITY AGAINST CHEMICAL PRODUCTS

The results obtained in the chemical durability test are shown in Table 3.

TABLE 3

The resin samples were not damaged by any of the chemical solutions, demonstrating that this polymer is resistant to chemical attacks. The siliceous sand samples resisted most chemical products, excluding the KOH solutions, which caused the colour of the surface

of the samples to change. Therefore, basic environments would affect such aggregates. The calcareous sand samples only resisted the low-concentration KOH solutions. The high-concentration NH_4Cl and KOH solutions caused a slight change in the surface colour of these samples, and the colour changed more clearly in the NaClO solution. The HCl solutions caused surface colour changes and particle losses in the calcareous samples, while efflorescence was also observed in the samples treated with $C_6H_8O_7$ and $C_3H_6O_3$. As expected these results demonstrate that calcareous aggregates are weak to acidic environments. The LS samples were moderately damaged by all chemical products, exhibiting surface colour changes. Finally, the surface of the AF samples changed colour when treated with most of the chemical solutions, excluding NH_4Cl and NaClO, which slightly changed the surface colour of AF-35 and did not damage AF-40 and AF-45. The lack of damage to the resin samples, as well as the damage to all samples of each target material, excluding those of AF, highlight the impact of the target material on the durability of PPC products in chemically aggressive environments. Figure 4 presents microscopic images of the results of the application of citric acid, which caused the greatest damage, to different samples with low resin contents.

FIGURE 4

In accordance with the visual inspection, the microscopic image of the siliceous sand sample exhibited no damage, while that of the calcareous sand sample exhibited voids due to sand particle losses. Small particle losses were also observed for the LS sample, which only changed in colour. This is likely due to the presence of lime particles in the slag. Other than the change in colour, there was no surface damage to the AF sample.

3.3. SURFACE STRENGTH AGAINST PHYSICAL DAMAGE

Figure 5 shows the results of the rebound number test, expressed as the MPa of surface strength based on the standard EN 12504-2.

FIGURE 5

The surface strength of the resin samples reached 49 MPa, demonstrating good surface resistance against impacts; this is likely related to its good flexural strength. As the resin content increased, the average and high dispersion surface strength of the siliceous sand combinations decreased; samples S-20, S-25, and S-30 achieved average values of 40, 38, and 34 MPa, with standard deviations of 16.8, 4.5, and 10.9 respectively. These results contradict those obtained by Hameed et al. [29], who observed an increase in the surface strength against impacts as the resin content increase. This demonstrates that siliceous sand can resist impacts well. The behaviour of the other target samples was opposite to this, as their surface strength increased with increasing resin content. The surface strengths of C-20, C-25, and C-30 reached 34, 35, and 37 MPa; those of LS-35, LS-40, and LS-45 reached 39, 41, and 49 MPa; and those of AF-35, AF-40, and AF-45 reached 35, 36, and 42 MPa, respectively. As expected from the results obtained by Wang and Wan [30] and Kazemy et al. [31], the surface strength of the calcareous sand and LS samples was directly related to the compressive strength. However, the relationship between the surface and compressive strength of siliceous sand and AF was indirect. Remarkably, LS-45 exhibited the same surface strength as the resin sample, and all LS

samples exhibited low dispersion. These results demonstrate the relationship between the surface strength and the type and content of fine particles in the target material.

Figure 6 shows the procedure followed in the surface scratching test.

FIGURE 6

The resin and mortar samples achieved a Mohs hardness of 3, corresponding to “calcite”, while siliceous and calcareous rocks have Mohs hardnesses of 9 and 3, respectively. These results indicate that the surface hardness depends on the resin, regardless of the target material.

4. CONCLUSIONS

This study demonstrates the suitability of two metallurgical wastes to replace natural aggregates in manufacturing durable PPC construction and building products. All waste-based samples were not visibly damaged by freezing and thawing cycles, although their mechanical properties decreased at variable magnitudes. The LS samples exhibited the best flexural and compressive strength before and after ageing. The AF samples exhibited close flexural and compressive strength properties to those of the natural aggregates before freezing and thawing, but they suffered the highest flexural and compressive strength losses after freezing and thawing. The resistance to chemical substances varied between the different target materials. The greatest damage was caused to the calcareous sand-containing samples by acids. Generally, LS and AF were sufficiently resistant to chemical substances, indicating their effectiveness and durability in chemically

aggressive environments. The surface strength of the LS samples against impact was highest, while that of the AF samples was close to that of natural sand. Finally, the surface resistance to scratching depended on the resin, and not on the target material. These results demonstrate the potential of these recycled target materials in manufacturing durable and more sustainable PPC products.

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DATA AVAILABILITY

The raw data required to reproduce these findings cannot be shared at this time due to technical and time limitations.

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TABLE 1. Liquid and cured characteristics of the CRYSTIC 406 NT Polyester ortoftalic tixotropic unsaturated not accelerated resin.

CHARACTERISTIC	UNITS	VALUE
Liquid resin		
Viscosity at 25 ºC	dPas	3.5-4.5
Specific gravity	g/cm^3	1.10
Volatile content	%	36-40
Acidity index	mg KOH/g	19-23
Cured resin		
Deformation temperature under load	ºC	65
Absorption after 24H of immersion in water	mg	15

Table 2. Characterization of the PPC samples. Composition, density and mechanical strength.

COMBINATION	TARGET MATERIAL	RESIN DOSAGE (%)	DENSITY (g/cm^3)	FLEXURAL STRENGTH (MPa)	COMPRESSIVE STRENGTH (MPa)
R	None	100	1.220	53.0	105.3
S-20	Siliceous Sand	20	1.897	22.0	93.4
S-25	Siliceous Sand	25	1.912	25.8	99.3
S-30	Siliceous Sand	30	1.870	25.1	106.2
C-20	Calcareous Sand	20	1.948	24.7	111.3
C-25	Calcareous Sand	25	1.961	23.5	116.7
C-30	Calcareous Sand	30	1.907	21.0	118.4
LS-35	Ladle Slag	35	1.902	43.6	153.7
LS-40	Ladle Slag	40	1.830	44.5	155.6
LS-45	Ladle Slag	45	1.749	45.2	160.5
AF-35	Aluminum Filler	35	1.749	23.7	104.2
AF-40	Aluminum Filler	40	1.690	25.5	101.6
AF-45	Aluminum Filler	45	1.589	24.1	87.4

Table 3. Durability against chemical products.

Product categories	Domestic cleaning product	Swimming pool product	Low concentration acid or base			High concentration aci		
Chemical products	Ammonium Chloride	Sodium Hypochlorite	Chloride acid	Citric acid	Potassium Hydroxide	Chloride acid	Lactic acid	
Composition	NH_4Cl	NaClO	HCl	$C_6H_8O_7$	KOH	HCl	$C_3H_6O_3$	
Concentration	100 g/L	20 mg/L	3%	100 g/L	30 g/L	18%	5%	
Testing time (Hours)	24	24	96	24	96	96	96	
R	0	0	0	0	0	0	0	
S-20	0	0	0	0	2	0	0	
S-25	0	0	0	0	2	0	0	
S-30	0	0	0	0	2	0	0	
C-20	1	2	3	4	0	3	4	
C-25	1	2	3	4	0	3	4	
C-30	1	2	3	4	0	3	4	
LS-35	2	2	2	2	2	2	2	
LS-40	2	2	2	2	2	2	2	
LS-45	2	2	2	2	2	2	2	
AF-35	1	1	2	2	2	2	2	
AF-40	0	0	2	2	2	2	2	
AF-45	0	0	2	2	2	2	2	

Key:

0	No surface damage
1	Slight change of surface color.
2	Change of surface color.
3	Change of surface color with particle loses.
4	Change of surface color with particle loses and efflorescences.

Figure 1. Granulometric curves of the target materials considered.

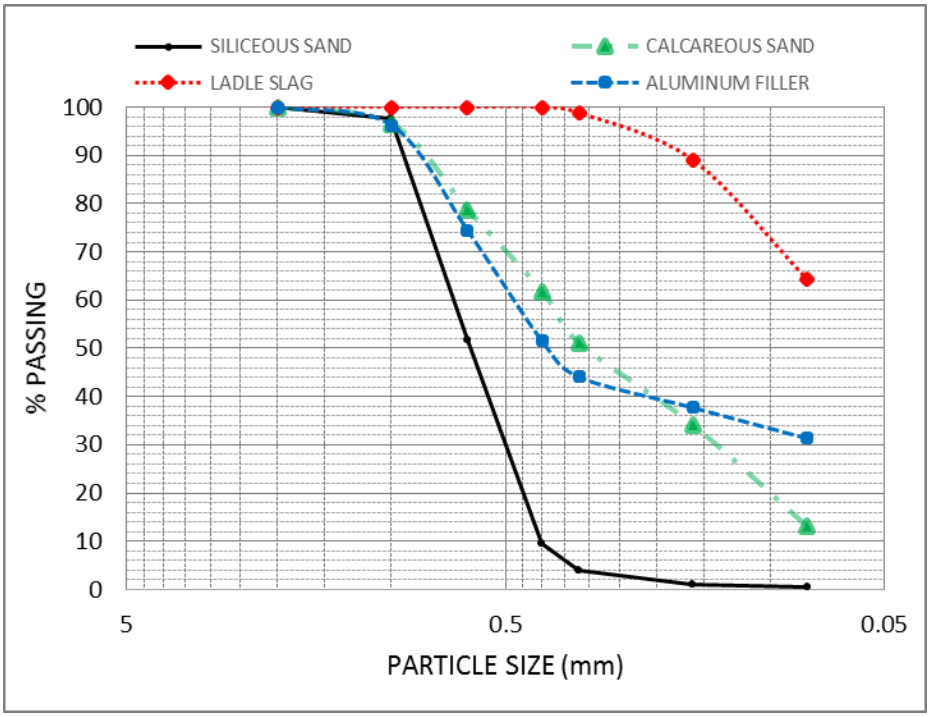

Figure 2. Flexural strength variations before and after the freezing and thawing cycles.

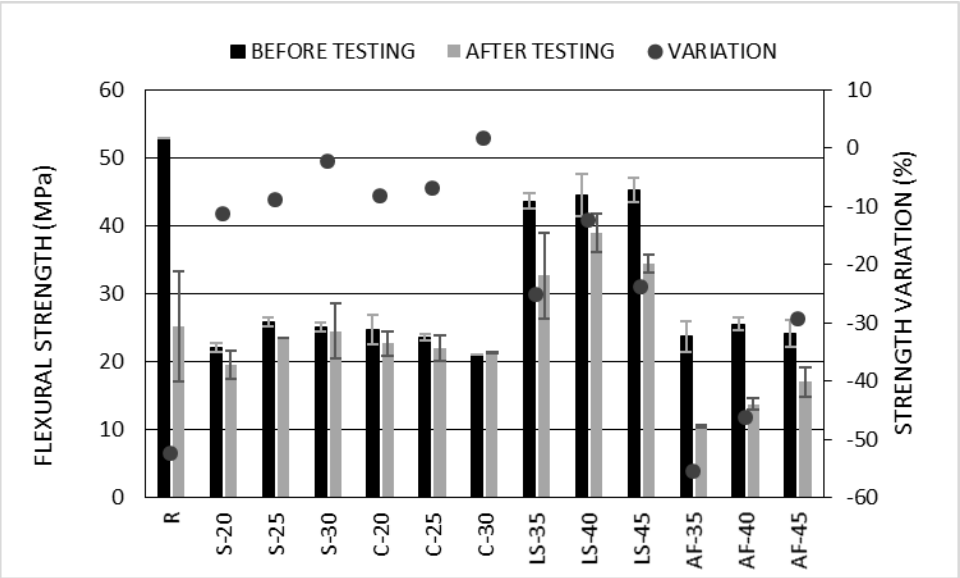

Figure 3. Compressive strength variations before and after the freezing and thawing cycles.

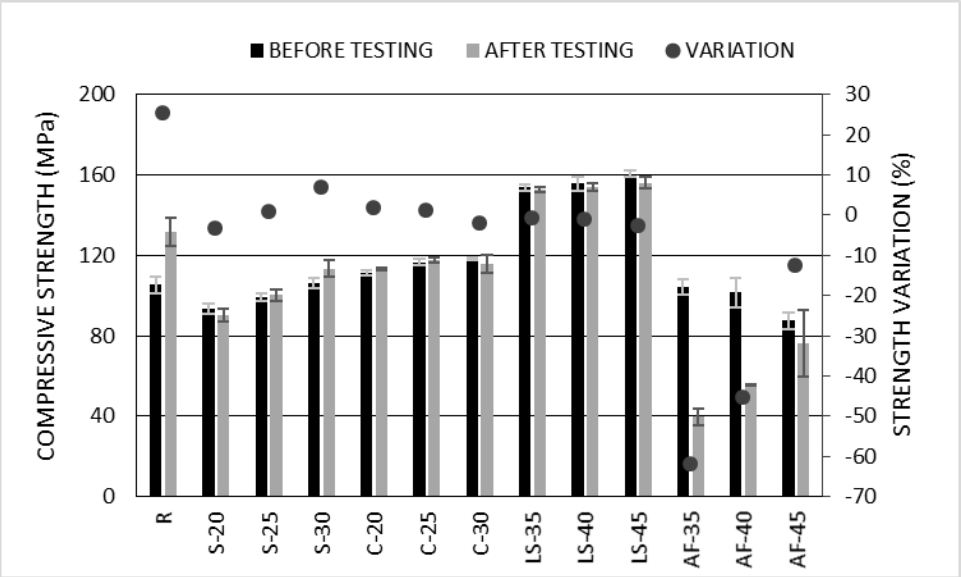

Figure 4. Durability against citric acid before and after the attack in different combinations. a) S-20, b) C-20, c) LS-35 and d) AF-35.

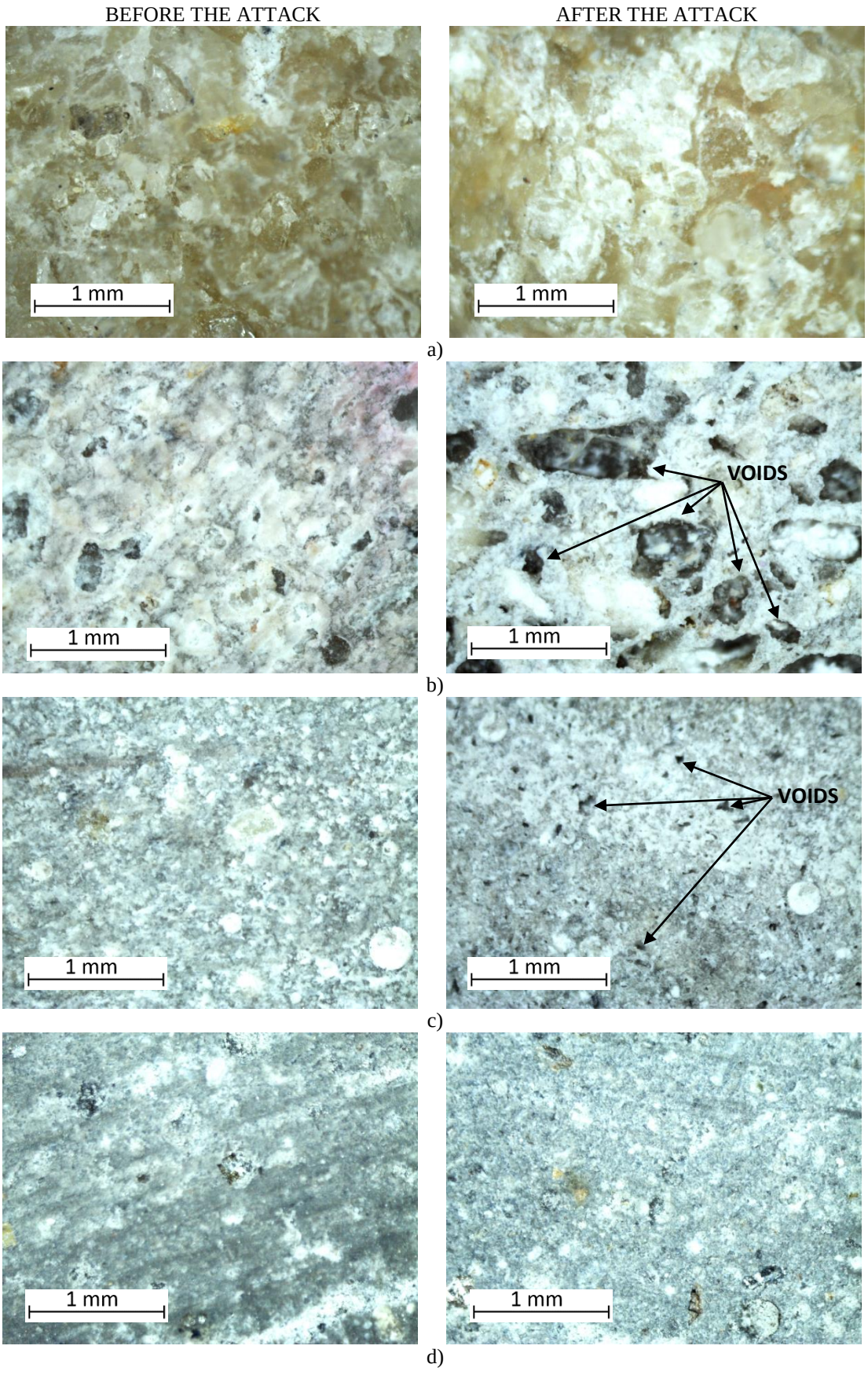

Figure 5. Rebound number test results.

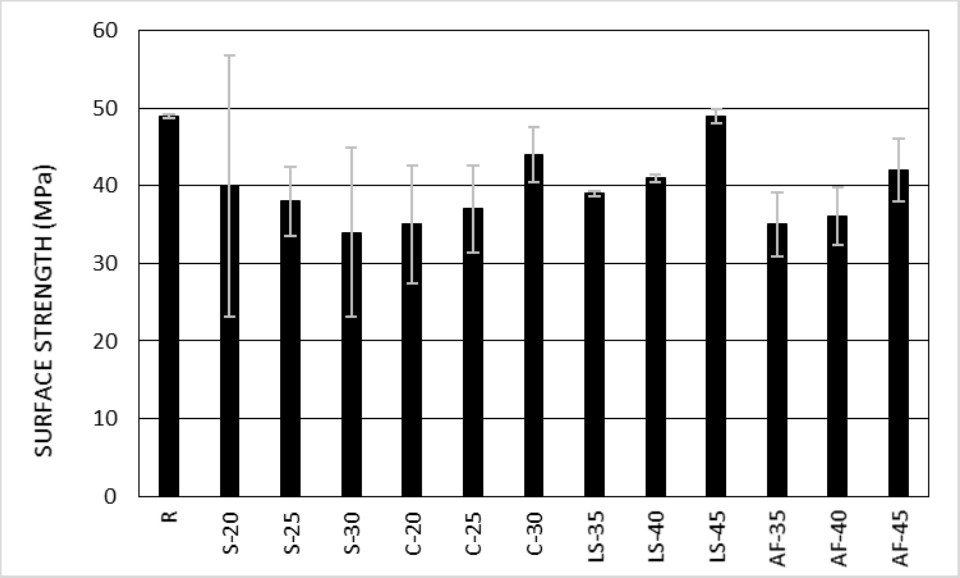

Figure 6. Procedure carried out for surface scratching test.