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## CHAPTER 12

# Performance on an Alkali-Activated Cement-Based Binder (AACB) for Coating of an OPC Infrastructure Exposed to Chemical Attack: A Case Study

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### 12.1 INTRODUCTION

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Premature degradation of ordinary Portland cement (OPC) concrete infrastructure is a current and serious problem related to the fact that OPC concrete presents a higher permeability that allows water and other aggressive elements to enter, leading to carbonation and chloride-ion attack, resulting in corrosion problems [1].

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Pacheco-Torgal et al. [2] mentioned the case of a tunnel in Dubai, which had been concluded in 1975 and needed to be completely repaired after just 11 years, a case of pile foundations that had disintegrated after just 12 years, and also a study on Norway OPC concrete bridges that indicated that several presented corrosion problems 24 years after they were built. As a consequence, worldwide concrete infrastructure rehabilitation costs are staggering. For example in the United States, where about 27% of all highway bridges are in need of repair or replacement, the needs are estimated to be over US\$1.6 trillion by 2021, and the corrosion deterioration cost due to deicing and sea salt effects is estimated at over US\$150 billion. In the European Union, nearly 84,000 reinforced and prestressed concrete bridges require maintenance, repair, and strengthening with an annual budget of £215 M, and that estimate does not include traffic management costs [3].

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Many of the degraded concrete structures were built decades ago when little attention was given to durability issues. Concrete durability means above all minimizing the possibility of aggressive elements to enter the



concrete, under certain environmental conditions for any of the following transport mechanisms: permeability, diffusion, or capillarity. The use of concrete surface treatments with waterproofing materials (also known as sealers) to prevent the access of aggressive substances is an important way of contributing to concrete durability. Almusallam et al. [4] studied several concrete coatings concluding that epoxy and polyurethane coatings performed better than acrylic, polymer, and chlorinated rubber coatings.

p0025 Other authors [5,6] showed that although some waterproof materials are effective for a particular transport mechanism (diffusion, capillarity, permeability), they may not be for another. They compared the waterproofing capacity of concrete with three polymeric resins (epoxy, silicone, acrylic) and mentioned that the silicone-based one is more effective (99.2%) in reducing water absorption by capillarity than the epoxy resin (93.6%), but in terms of chloride diffusion the epoxy resin is 100% effective, while the silicone varnish does not go beyond 67.5%. Epoxy coatings exhibited excellent durability under the laboratory and field-test conditions and are recommended for protecting concrete in cooling tower basins against sulfur-oxidizing or other acid-producing bacteria [7].

p0030 Medeiros and Helene [8] used a water-repellent material based on silane-siloxane, noticing that although it is effective to reduce the water absorption by capillarity of concrete (reduced from two to seven times), it only managed to achieve a reduction of the chloride diffusion from 11% to 17% and also failed to prevent the access of water by permeability.

p0035 Pacheco-Torgal and Jalali [9] confirm that the surface treatment of concrete with a water-repellent material is effective, but above all more cost effective when compared with the alternative of using a polymer additive in the composition of concrete.

p0040 In 2013, Brenna et al. [10] studied the efficiency of four commercial concrete coatings (a polymer-modified cementitious mortar and three elastomeric coatings) against chloride-induced corrosion, concluding that the polymer-containing mortar shows the best effect on delaying chloride penetration in concrete. In summary, the most common surface treatments use polymeric resins based on epoxy, silicone (siloxane), acrylics, chlorinated rubber, polyurethanes, or polymethacrylate.

p0045 Bijen [11] mentioned that the epoxy resins have low resistance to ultraviolet radiation and polyurethanes are sensitive to high-alkalinity environments. Polyurethane is obtained from isocyanates, known worldwide for their tragic association with the Bhopal disaster. As for chlorinated rubber it derives from reacting butyl rubber with chlorine; it is



important to remember that chlorine is associated with the production of dioxins and furans, which are extremely toxic and also bioaccumulative. Several scientist groups already suggest that chlorine-based industrial products should be prohibited [12].

p0050 Besides, the European Union has approved Regulation (EU) 305/2011, related to construction products regulation, which will replace the current Directive 89/106/CEE, already amended by Directive 1993/68/EEC, known as the Construction Products Directive. A crucial aspect of the new regulation relates to the information regarding hazardous substances [13].

p0055 Recent investigations on the geopolymer field [14] reveal a third category of mortars with high potential to enhance the durability of concrete structures. Investigations in the field of geopolymers have exponentially increased after the research results of Davidovits [15], who developed and patented binders obtained from the alkali activation of metakaolin, coining the term “geopolymer” in 1978. The technology of alkali activation, however, predates this terminology by several decades [16].

p0060 For the chemical designation of the geopolymer, Davidovits suggested the name “polysialates,” in which sialate is an abbreviation for aluminosilicate oxide. The sialate network is composed of tetrahedral anions  $[\text{SiO}_4]^{4-}$  and  $[\text{AlO}_4]^{5-}$  sharing the oxygen, which needs positive ions such as ( $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Li}^+$ ,  $\text{Ca}^{++}$ ,  $\text{Na}^+$ ,  $\text{Ba}^{++}$ ,  $\text{NH}_4^+$ ,  $\text{H}_3\text{O}^+$ ) to compensate for the electric charge of  $\text{Al}^{3+}$  in tetrahedral coordination (after dehydroxilation the aluminum changes from coordination 6 (octahedral) to coordination 4 (tetrahedral). However, Provis and Van Deventer [17] mentioned that the sialate nomenclature “implies certain aspects of the geopolymer gel structure which do not correspond to reality.”

p0065 In 2014, Provis presented a rigorous a useful definition of these materials: “alkali-activated materials are produced through the reaction of an aluminosilicate—normally supplied in powder form as an industrial by-product or other inexpensive material—with an alkaline activator, which is usually a concentrated aqueous solution of alkali hydroxide, silicate, carbonate or sulfate” [18].

p0070 In the last decade several authors have reported research in a large number of aspects related to geopolymers.

p0075 However, very few studies [19–21] have addressed the use of geopolymers for enhancement of concrete structures’ durability. Since geopolymer performance concerns the resistance to acid attack, is far better than that of Portland cement [16], which means that these materials could be an alternative low-toxicity coating material.



p0080 This paper presents results of an experimental investigation on the resistance to chemical attack (with sulfuric, hydrochloric, and nitric acid) of several materials: OPC concrete, high-performance concrete (HPC), epoxy resin, acrylic painting, and a fly ash-based geopolymeric mortar.

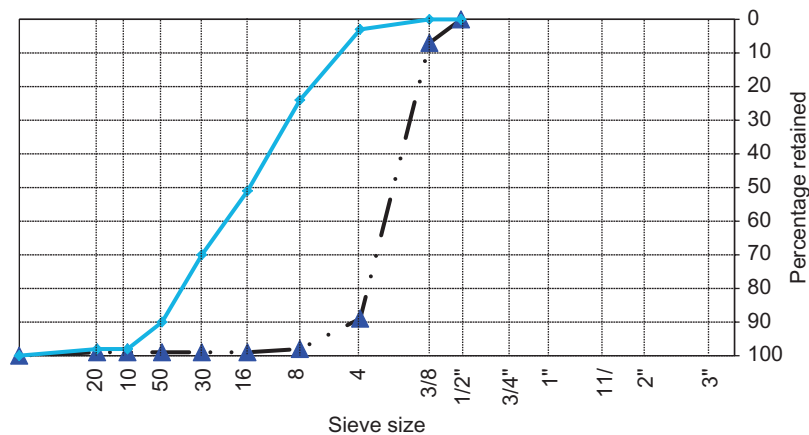
s0015 **12.2 EXPERIMENTAL WORK**

s0020 **12.2.1 Materials, Mix Design, Mortar and Concrete Mixing, and Concrete Coating**

p0085 The characteristics of the aggregates (coarse and sand) used are shown in Table 12.1 and in Fig. 12.1. The fly ash used in the geopolymeric mortars was supplied by Sines-EDP and according to the NP EN 450-1 it belongs to the B-class and has an N-class fineness modulus. Geopolymeric mortars were a mixture of aggregates, fly ash, calcium hydroxide, and alkaline silicate solution. The mass ratio for aggregates/fly ash and activator was 2/1/0.6. A 10% percentage substitution of fly ash by calcium hydroxide in the mixture was also used. This is because the use of minor

t0010 **Table 12.1** Characteristics of the aggregates

	Max dimension	Fine content	Density (kg/m <sup>3</sup> )	Water absorption
Sand	4.0	≤3	2660	0.2
Coarse aggregates	8.0	≤1.5	2620	0.6



f0010 **Figure 12.1** Aggregate particle-size distribution of the sand and of the coarse aggregate.

calcium hydroxide percentages is pivotal for the strength and durability of geopolymers [22,23]. The alkaline activator was prepared prior to use. An activator with sodium hydroxide and sodium silicate solution ( $\text{Na}_2\text{O} = 13.5\%$ ,  $\text{SiO}_2 = 58.7\%$ , and water = 45.2%) was used with a mass ratio of 1:2.5. Previous investigations showed that this ratio lead to the highest compressive strength results in geopolymeric mortars [14]. The sand, fly ash, and calcium hydroxide were dry mixed before being added to the activator. Three different sodium hydroxide concentrations (10, 14, and 18 M) were used. The fresh mortar was cast and allowed to set at room temperature for 24 h before being removed from the molds and kept at room temperature (20°C) until tested in compression and flexural strength. An OPC (CEM I 42,5 N) was used to prepare the concrete mixtures. Two concrete mixes (normal and HPC) were designed using the Faury concrete mix design method (Table 12.2). The concrete mixing starts with the introduction of the coarse aggregates in the mixer, followed by the sand for 2 min; then OPC is introduced and mixed to the aggregates for 2 more minutes. Then, 70% of the water is introduced in the mixer and all the ingredients are mixed for 2 min. Finally, the remaining water is added for 2 min and everything is mixed for 2 more minutes.

The concrete specimens were conditioned at a temperature equal to  $21 \pm 2^\circ\text{C}$  cured in a moist chamber until they have reached 28 days. An epoxy resin often used as concrete coating protection against acid attack with a commercial reference Sikagard 62 PT was used for coating of the two concrete mixtures. The epoxy adhesive is a two-component system (resin and hardener) with a bulk density of  $1.35 \text{ kg/dm}^3$ . After mixing the two components, the mixture remains workable for 20 min at 20°C or just 0 min at 30°C. An acrylic paint often used as concrete coating protection to prevent the access of aggressive substances with a commercial reference Sikagard 660 ES was also used for coating of the two concrete mixtures. This material has a bulk density of  $1.30 \text{ kg/dm}^3$  and is provided by the manufacturer as ready to be used.

t0015 **Table 12.2** Concrete mix proportions per cubic meter of concrete

	Cement (kg)	Sand (kg)	Coarse aggregates (kg)	Water	W/C
NC	270	1135	732	182	0.65
BED	442	876	782	205	0.45

s0025 **12.3 EXPERIMENTAL PROCEDURES**

s0030 **12.3.1 Compressive Strength**

p0095 The compressive strength was performed under NP EN 206-1. Tests were performed on  $100 \times 100 \times 100 \text{ mm}^3$  concrete specimens. The compressive and flexural strength data of geopolymeric mortars was obtained using  $160 \times 40 \times 40 \text{ mm}^3$  specimens according to EN 1015-11. Compressive strength for each mixture was obtained from an average of three cubic specimens determined at the age of 28 days of curing.

s0035 **12.3.2 Water Absorption by Immersion**

p0100 Tests were performed on  $40 \times 400 \times 80 \text{ mm}^3$  specimens. Specimens were tested with 28 days curing. The specimens were immersed in water at room temperature for 24 h. First, the weight of the specimens while suspended by a thin wire and completely submerged in water is recorded as  $W_{\text{im}}$  (immersed weight). After that, the specimens were removed from water, and placed for 1 min on a wire mesh allowing water to drain; then visible surface water is removed with a damp cloth and weight is recorded as  $W_{\text{sat}}$  (saturated weight). All specimens were placed in a ventilated oven at  $105^\circ\text{C}$  for not less than 24 h and allowing that two successive weighings at intervals of 2 h show an increment of loss not greater than 0.1% of the last previously determined weight of the specimen. The weight of the dried specimens is recorded as  $W_{\text{dry}}$  (oven-dry weight). The absorption coefficient is determined as following equation:

$$A(\%) = \frac{W_{\text{sat}} - W_{\text{dry}}}{W_{\text{sat}} - W_{\text{im}}} \times 100 \quad (12.1)$$

s0040 **12.3.3 Capillary Water Absorption**

p0105 Capillary water absorption was carried out using  $40 \times 400 \times 80 \text{ mm}^3$  specimens in the case of geopolymeric mortars and  $100 \times 100 \times 100 \text{ mm}^3$  specimens for concrete. After 28 days in a moist chamber the specimens were placed in a  $105^\circ\text{C}$  oven for 24 h. The test consists of placing the specimens in a container with enough water so that one side of the sample will remain immersed. This test is carried out according to Standard LNEC E393. Water absorption has been measured after 5, 10, 20, 30, 60, 90, 120, 180, 240, 300, 360, 420, and 480 min. Capillarity water absorption was obtained from an average of three specimens.



s0045 **12.3.4 Resistance to Chemical Attack**

p0110 The resistance to chemical attack followed a variation of the ASTM C-267 (Standard test methods for chemical resistance of mortars, grouts, and monolithic surfacing's and polymer concretes). The test used in the present investigation consists of the immersion of  $100 \times 100 \times 100 \text{ mm}^3$  concrete (NC, HPC, coated concrete specimens) and fly ash geopolymeric mortar specimens with 28 days curing in acid solution. Three different acids were used (sulfuric, hydrochloric, and nitric). Three acid concentrations were used (10%, 20%, and 30%) to simulate long-term exposure at lower concentrations. Other authors used 5%  $\text{Na}_2\text{SO}_4$  concentrations and immersion for 12 months [24]. The resistance to acid attack was assessed by the differences in weight of dry specimens before and after acid attack at 1, 7, 14, 28, and 56 days. The chemical resistance was assessed by the differences in weight of dry specimens before and after acid attack, since compressive strength of specimens immersed in acid media could not be evaluated. The fly ash-based geopolymeric mortar used in the resistance to acid attack was the one associated with the highest compressive strength and low water absorption.



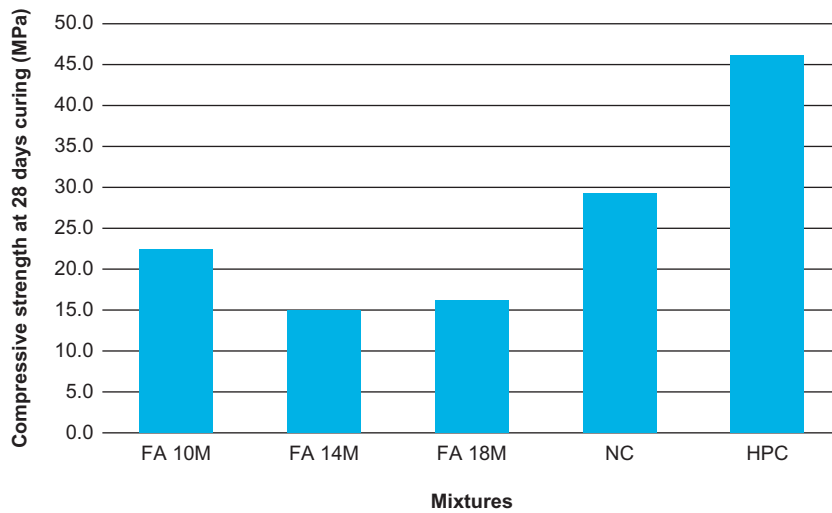
s0050 **12.4 RESULTS AND DISCUSSION**

s0055 **12.4.1 Compressive Strength**

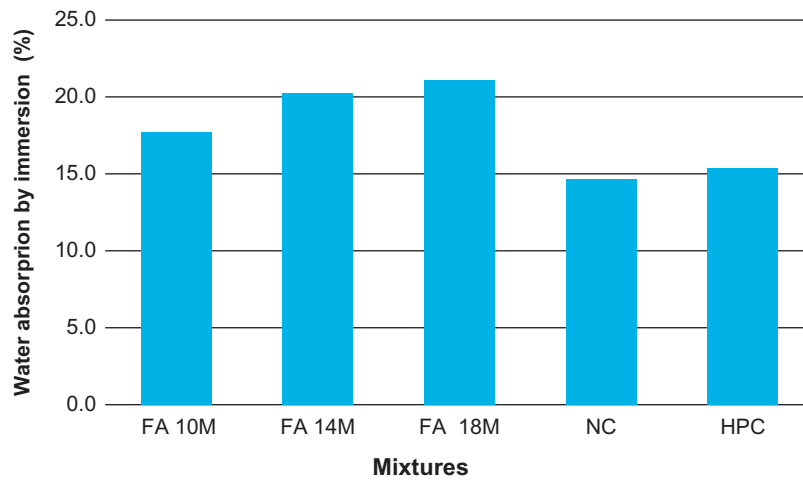
p0115 Fig. 12.2 shows the results of the compressive strength of the fly ash-based geopolymeric mortars after 28 days curing as well as of the two concrete mixtures. The results show that the compressive strength of geopolymeric mortars is very dependent on the molarity of the sodium hydroxide. Increasing the molarity from 10 to 14M leads to a relevant compressive strength loss.

p0120 However, further increase from 14 to 18M shows no noticeable effects. Previous investigations [25] have shown that although a high alkali content favors the dissolution of Al and Si species of fly ash it can also negatively affect its strength. Pacheco-Torgal et al. [26], who studied the geopolymerization of mine wastes, noticed the opposite phenomenon. Other authors [27] mentioned that when  $\text{OH}^-$  concentration was high enough, dissolution of fly ash was accelerated, but polycondensation was hindered. Normal concrete (NC) has a compressive strength around 30 MPa while HPC compressive strength slightly exceeds 45 MPa. The standard deviation was low and the coefficient of variation does not exceed 12% meaning that the results were statistical relevant.





f0015 **Figure 12.2** Compressive strength.

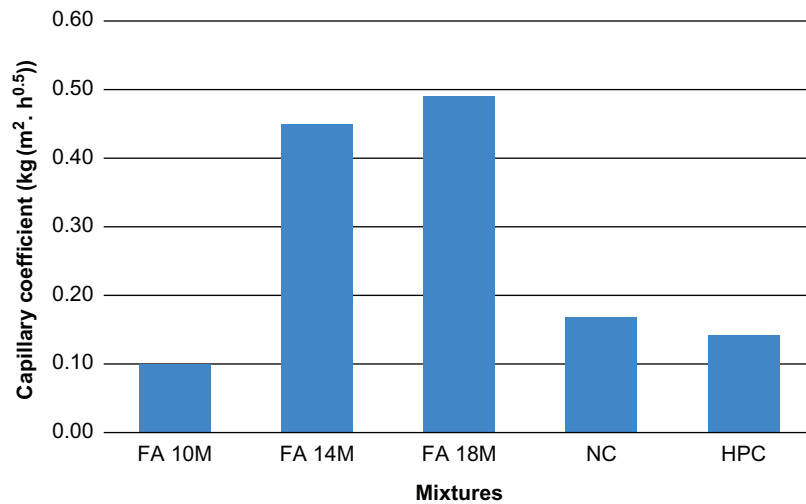


f0020 **Figure 12.3** Water absorption by immersion.

#### s0060 12.4.2 Water Absorption by Immersion

p0125 The results of water absorption by immersion are showed in [Fig. 12.3](#). These results are aligned with compressive strength performance. The fly ash geopolymeric mortar with the least water absorption by immersion is the one with the highest compressive strength.





f0025 **Figure 12.4** Water absorption capillary coefficients.

p0130 The geopolymeric mortars with a sodium hydroxide molarity of 14 and 18M show a water absorption around 20%. This means that compressive strength is directly influenced by open porosity. However, previous investigations [28] on the field of geopolymers showed that low porosity does not always mean high compressive strength; being that compressive strength is more influenced by NaOH concentration than it is from porosity. Both NC and HPC show a water absorption around 15%. This falls in the current water absorption by immersion range of current OPC concretes used by the construction industry (compressive strength at 28 days curing between 25 and 45 MPa), of 12–16%.

### s0065 12.4.3 Capillary Water Absorption

p0135 Fig. 12.4 shows the capillary water absorption coefficients. While the fly ash geopolymeric mortars with a sodium hydroxide molarity of 14 and 18M show a capillary water absorption around 0.45 kg/m<sup>2</sup>·h<sup>0.5</sup> the geopolymeric mortar with the lowest open porosity and the highest compressive strength has a 0.1 kg/m<sup>2</sup>·h<sup>0.5</sup> capillary water absorption coefficient.

p0140 The capillary water absorption of the two concrete mixes used in this investigation is very low, around 0.15 kg/m<sup>2</sup>·h<sup>0.5</sup>. As a comparison, a plain C30/37-strength class concrete has a capillarity coefficient of 0.251 kg/m<sup>2</sup>·h<sup>0.5</sup> for 28 days curing [29], while a plain C20/25-strength class

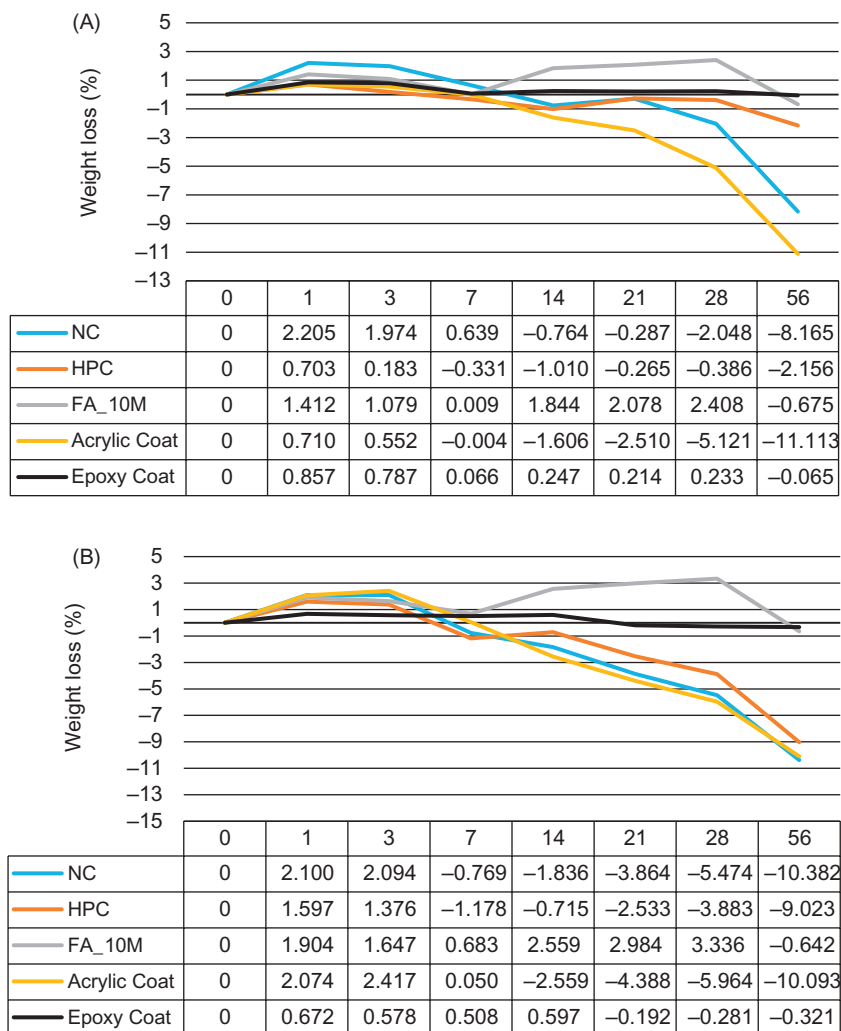
concrete (the most used strength class in Europe [30]) has capillarity coefficients between 0.85 and 2.6 kg/m<sup>2</sup>.h<sup>0.5</sup> [31].

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## 12.4.4 Resistance to Chemical Attack

### 12.4.4.1 Resistance to Sulfuric Acid Attack

Fig. 12.5 shows the weight loss after sulfuric acid attack for the different acid concentrations. NC coated with epoxy resin shows the most stable performance for all three acid concentrations confirming previous investigations.



f0030 **Figure 12.5** Weight loss due to sulfuric acid attack: (A) 10% acid concentration, (B) 20% acid concentration, and (C) 30% acid concentration.

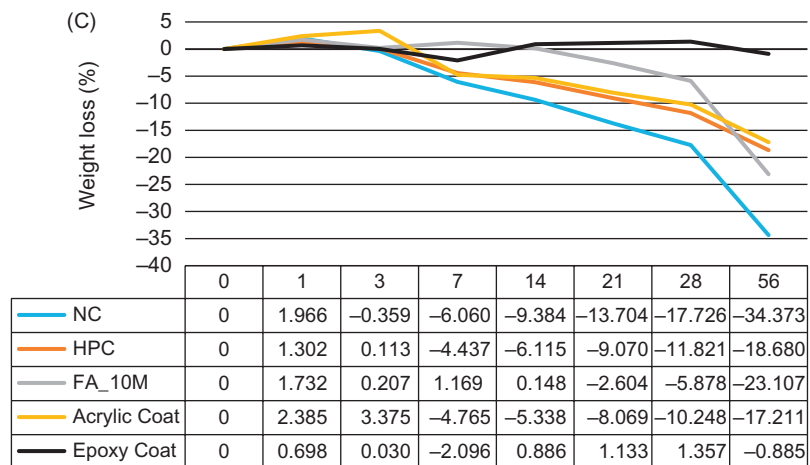
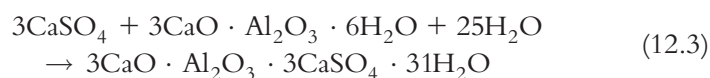


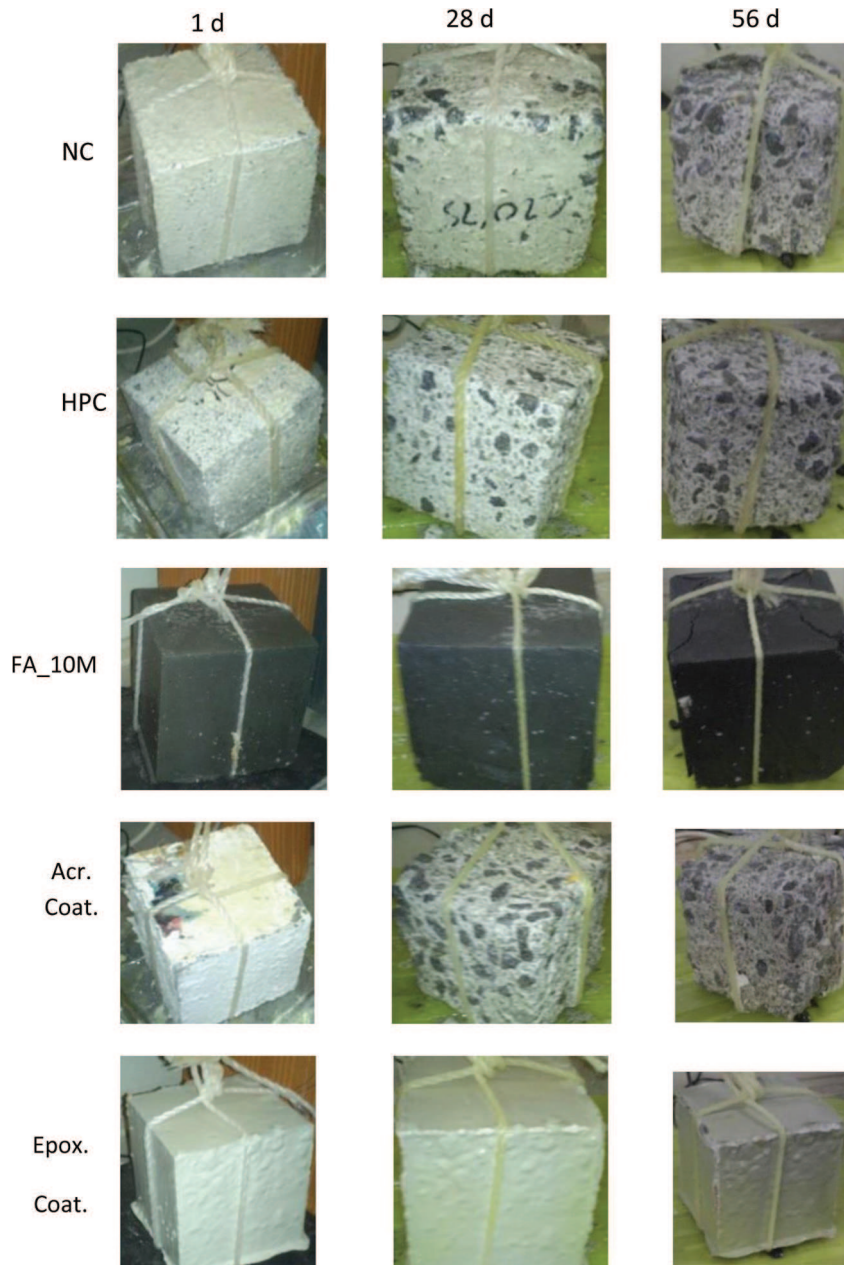
Figure 12.5 (Continued)

The fly ash geopolymeric mortar shows a good performance for both 10% and 20% sulfuric acid concentration. Fig. 12.6 shows photos of the different specimens after immersion in a 20% sulfuric acid concentration. Even for a 30% sulfuric acid concentration this mortar shows a good acid resistance for immersion until 14 days. HPC specimens show the third-best performance. It shows a minor weight loss after 56 days in a 10% sulfuric acid concentration. For a 20% sulfuric acid concentration the weight loss is clear beyond 14 days, reaching a maximum of 9%. When the concentration increases to 30%, the weight loss starts after 7 days immersion and reaches a maximum of 20% after 56 days. Specimens of NC coated with acrylic paint show the same performance of uncoated concrete specimens for both 10% and 20% sulfuric acid concentration. Only for the 30% acid concentration and long-term immersion can this coat be of some use.

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Since NC and HPC have almost similar capillary water absorption, the differences in acid resistance lie in the leaching of calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) from the pore solution and decalcification of CSH, which must be lower in the latter case due to a much higher Portland cement content. In the sulfuric acid attack, sulfate ions react with calcium hydroxide, forming calcium sulfate dihydrate-gypsum (Fig. 12.2), and with aluminate hydrates, forming ettringite (Fig. 12.3).



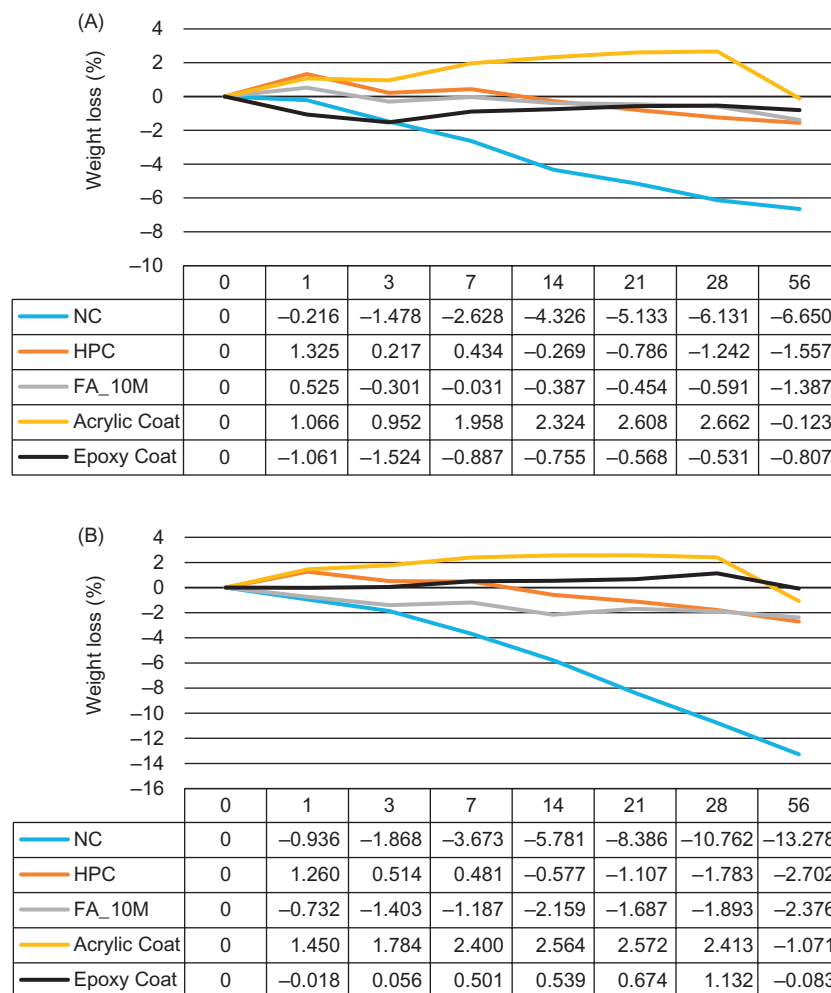


f0035 **Figure 12.6** Specimens after immersion in a 20% sulfuric acid solution.

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#### 12.4.4.2 Resistance to Nitric Acid Attack

Weight loss after nitric acid attack is shown in Fig. 12.7. Again, NC coated with epoxy resin shows the most stable performance for all three acid concentrations. Nitric acid attack at 10% concentration is especially destructive for NC even after just 7 days immersion. Nitric acid reacts with calcium compounds, forming calcium nitrate, which has a solubility of 56%. All the other mixtures show a weight loss not exceeding 2% even after 56 days immersion. The behavior for a 20% nitric acid concentration



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Figure 12.7 Weight loss due to nitric acid attack: (A) 10% acid concentration, (B) 20% acid concentration, and (C) 30% acid concentration.

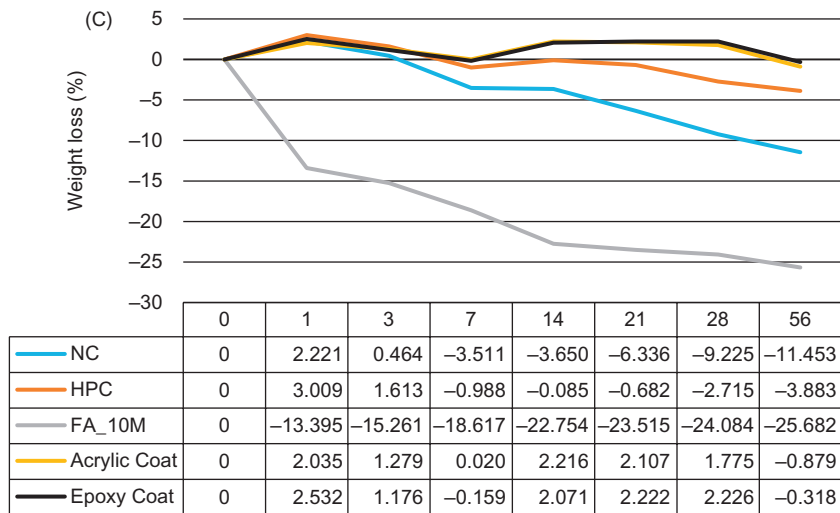


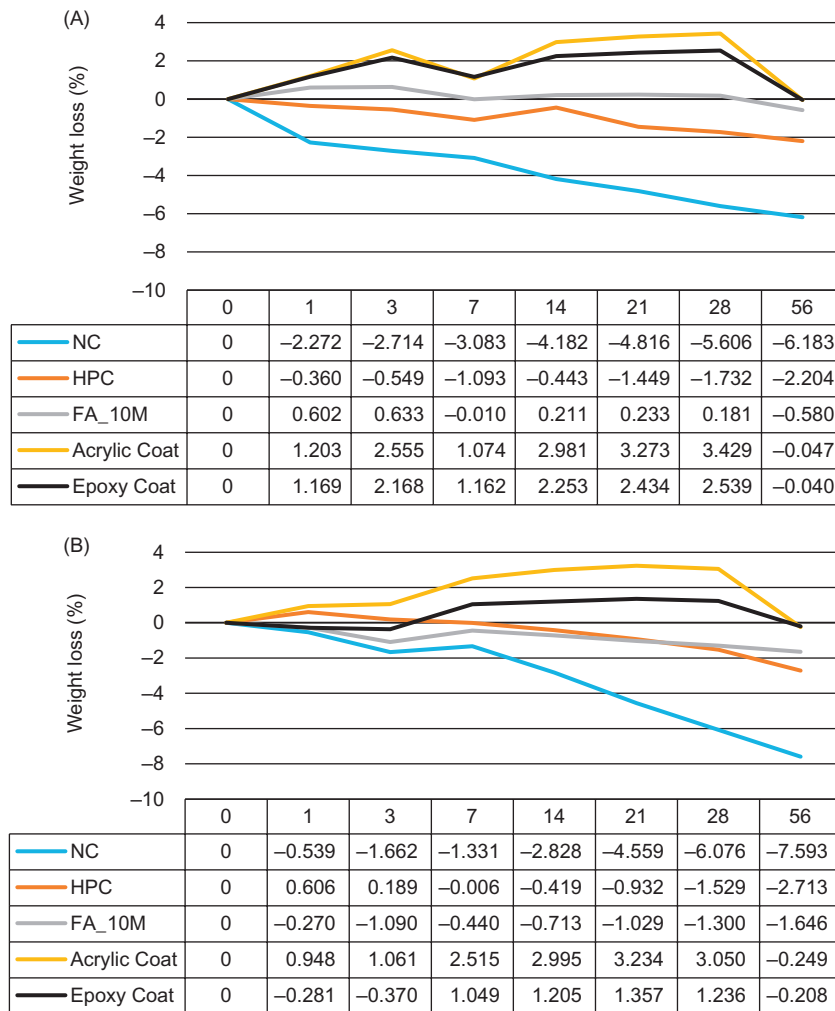
Figure 12.7 (Continued)

is almost the same, the difference being that NC shows a higher weight loss. When the acid concentration is increased to 30% NC does not show an increase in the weight loss. For this very high acid concentration the geopolymeric mortar shows a disappointing performance. Allahverdi and Škvára [32,33] suggested that the electrophilic attack of nitric acid protons results in the ejection of tetrahedral aluminum from the aluminosilicate framework and in the formation of an imperfect highly siliceous framework. Other authors [34] also suggested this aluminosilicate depolymerization.

s0085 **12.4.4.3 Resistance to Hydrochloric Acid Attack**

p0160 Fig. 12.8 shows the weight loss after hydrochloric acid attack for the different acid concentrations. The results are every similar to those of the nitric acid attack. A 10% hydrochloric acid concentration is responsible for a relevant NC weight loss even after just 7 days immersion. This type of acid reacts with calcium compounds, leading to the formation of calcium chloride, which has extremely high solubility (46.1 wt%) [35]. The behavior for a 20% nitric acid concentration is almost the same.

p0165 The difference being that NC shows a higher weight loss. All the other mixtures show a weight loss not exceeding 2% even after 56 days immersion. When the hydrochloric acid concentration is increased to 30%, NC does not show a relevant increase in the weight loss. However,



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**Figure 12.8** Weight loss due to hydrochloric acid attack: (A) 10% acid concentration, (B) 20% acid concentration, and (C) 30% acid concentration.

the geopolymeric mortar shows a high weight loss. Davidovits et al. [36] reported a 78% weight loss for OPC concrete specimens immersed for 4 weeks in a 5% hydrochloric acid solution, which is much higher than the weight loss of NC after immersion for 56 days in a 30% hydrochloric acid solution, which was lower than 10%. This difference is so high that it cannot be explained by the specimen's geometry or OPC concrete composition. A possible explanation could be related to the periodic replacement

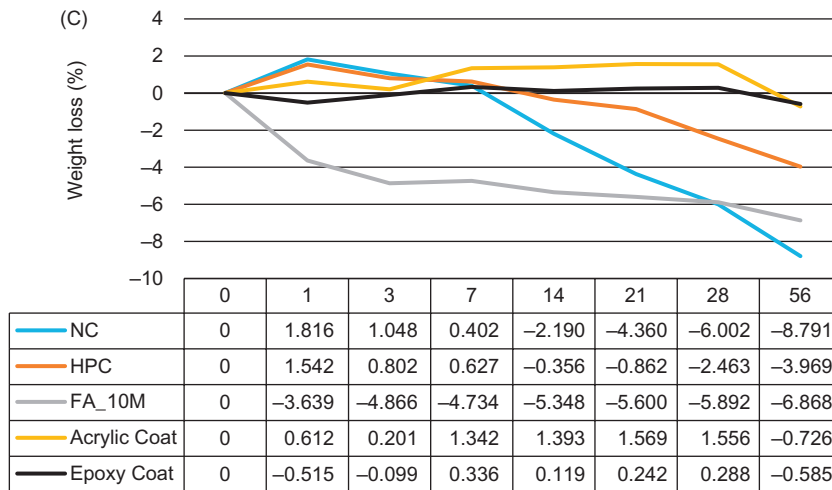


Figure 12.8 (Continued)

of the acid solution by the Davidovits study. Just because the pH is raised with time, for instance, a solution of sulfuric acid at 5% concentration evolves from pH = 1.05–6.95 after 28 days [37].

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## 12.5 COST ANALYSIS

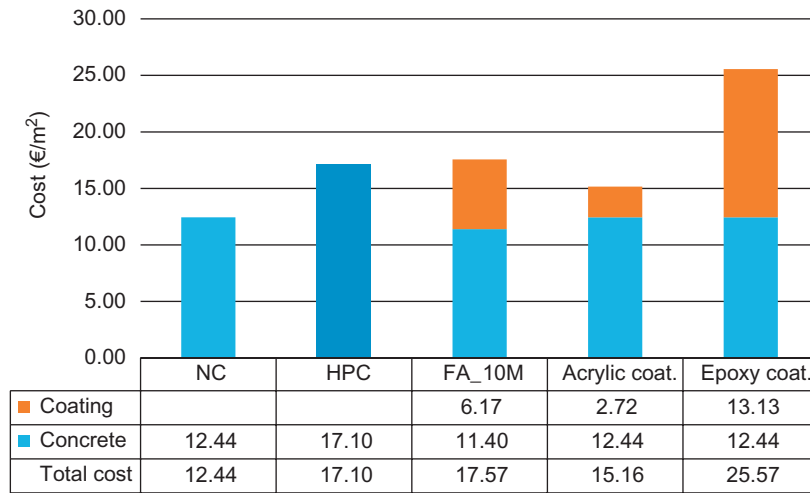
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In order to evaluate the economic efficiency of several structural solutions, comparisons between the costs of materials were made. The cost calculations were related to 1 m<sup>2</sup> of concrete pavement with 0.3 m thickness. Two noncoated solutions (NC, HPC), one with 0.275 m NC thickness coated with 0.025 m fly ash geopolymer and two coated with acrylic paint and epoxy resin were analyzed. Fig. 12.9 shows the costs of the different solutions. The concrete pavement coated by epoxy resin is by far the most costly solution. Epoxy coating costs exceed the NC solution costs by as much as 100%. Fig. 12.10 shows the cost to remaining mass (after acid attack) ratio according to acid concentration. The results show that for 10% and even 20% acid concentrations NC shows the best cost efficiency. The cost efficiency of the HPC-based solution is similar to the fly ash-based geopolymeric mortar except for a 30% acid concentration.

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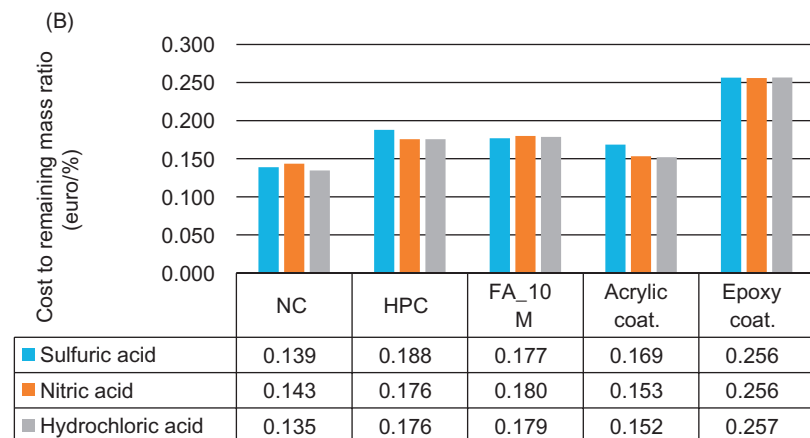
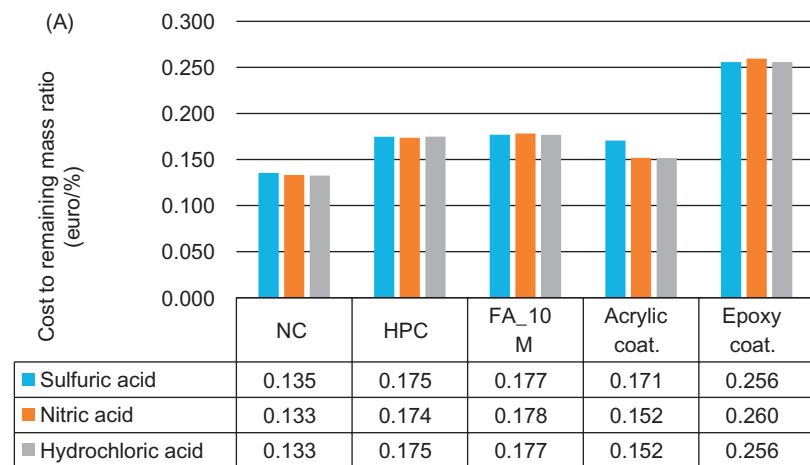
The results also show that no matter how well epoxy resin performs under acid attack its economic efficiency is the worst between all five solutions, being 70% above the cost efficiency of the fly ash-based geopolymeric mortar. Only for a 30% acid concentration does the





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Figure 12.9 Costs of the different concrete pavement solutions.



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Figure 12.10 Cost to remaining mass ratio (euro/%) (A) 10% acid concentration, (B) 20% acid concentration, and (C) 30% acid concentration.

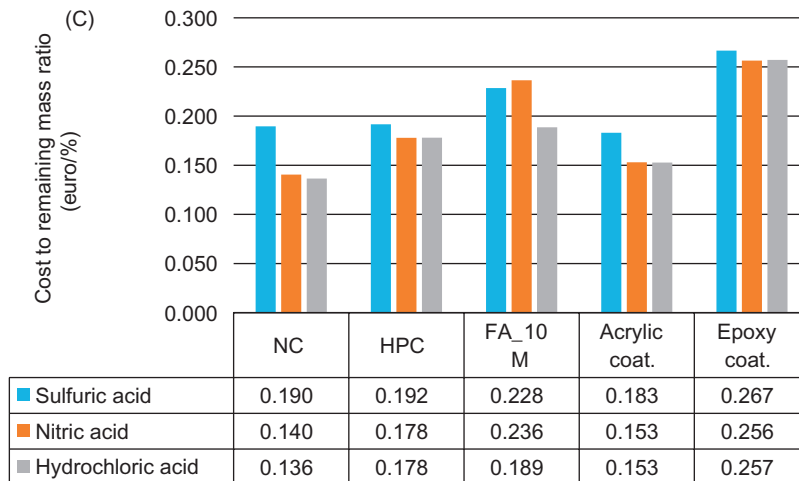


Figure 12.10 (Continued)

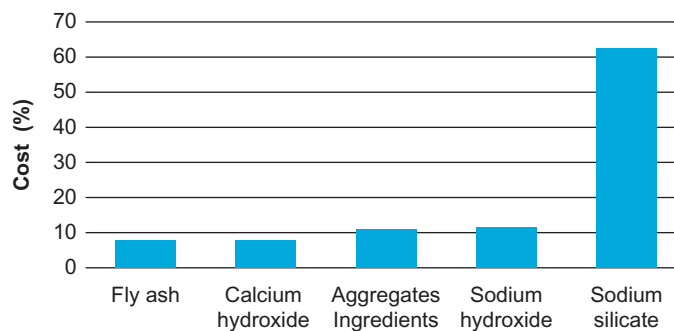
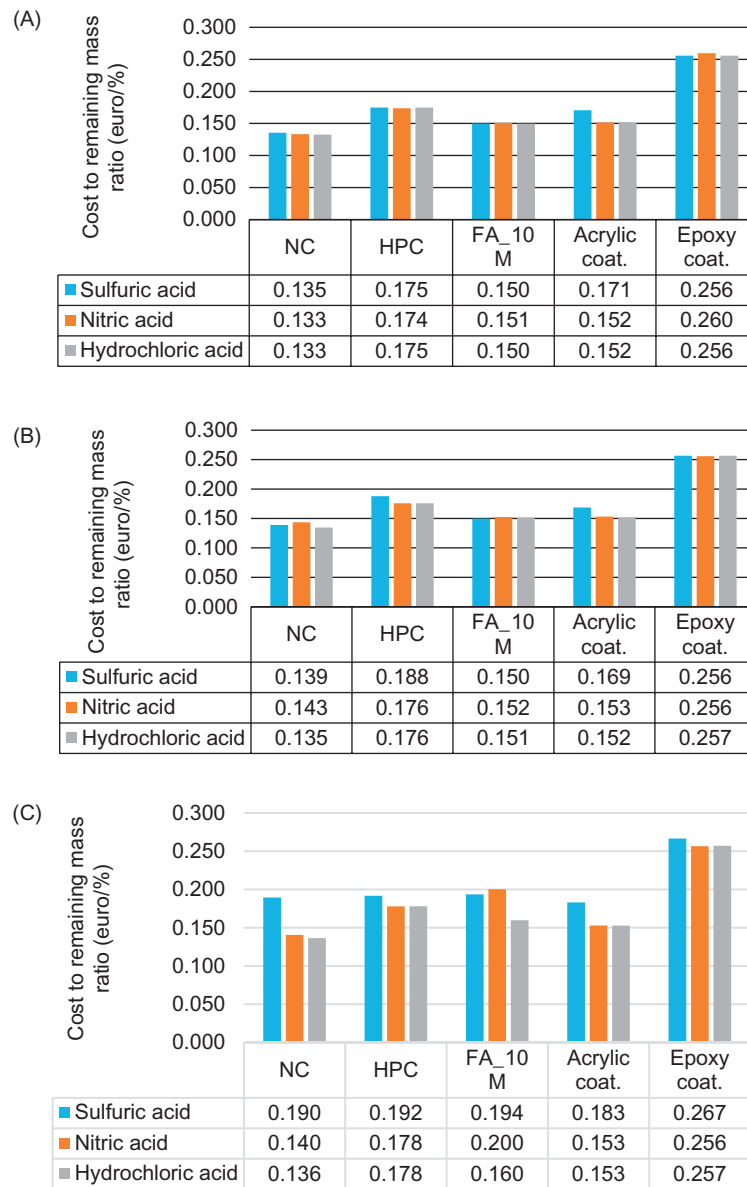


Figure 12.11 Cost percentage of fly ash geopolymeric mortar ingredients.

epoxy-based solution gain some interest. It is important to remember that the cost of the fly ash-based geopolymeric mortar is very dependent on the cost of sodium silicate (Fig. 12.11). Fig. 12.12 shows a simulation of the cost-to-remaining-mass (after acid attack) ratio according to acid concentration when the sodium silicate cost is around 30% of its current cost. This means that current investigations aiming to replace sodium silicate with low-cost waste glass [38] will increase the cost efficiency of the fly ash-based geopolymeric mortar as a coating material for OPC concrete infrastructures exposed to harsh chemical environments. Furthermore, the future use of waste glass as sodium silicate replacement fits the European zero-waste program COM 398 [39].



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**Figure 12.12** Cost to remaining mass ratio (euro/%) for a low-cost waste glass sodium silicate replacement simulation: (A) 10% acid concentration, (B) 20% acid concentration, and (C) 30% acid concentration.



s0095 **12.6 CONCLUSIONS**

p0180 Worldwide infrastructure rehabilitation costs are staggering. Premature degradation of OPC concrete infrastructure is a current and serious problem related to the fact that OPC concrete presents a higher permeability that allows water and other aggressive elements to enter, leading to carbonation and chloride-ion attack, resulting in corrosion problems. This article presents results of an experimental investigation on the resistance to chemical attack of several materials. NC coated with epoxy resin shows the most stable performance for all three acid types and acid concentrations. For a very high nitric acid concentration the geopolymeric mortar shows a disappointing performance that could be due to the ejection of tetrahedral aluminum from the aluminosilicate framework and in the formation of an imperfect highly siliceous framework. The results show that no matter how well epoxy resin performs under acid attack its economic efficiency is the worst between all the five solutions, being 70% above the cost efficiency of the fly ash-based geopolymeric mortar. Current investigations aiming to replace sodium silicate with low-cost waste glass will increase the cost efficiency of the fly ash-based geopolymeric mortar as coating material for OPC concrete infrastructure exposed to harsh chemical environments.



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