



# Microstructural and Durability Assessment of Various Concrete Types Under Different Chemical Environments

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## Abstract

The research investigates the microstructural analyses and durability performances of a geopolymer concrete (GPC), two different engineered cementitious concretes (ECC), and an ordinary Portland cement concrete (NC) under 3.5% seawater (SW), 5% magnesium sulfate (MS), and 5% sulfuric acid (SA) environments. The nanosilica (NS) and F-type fly ash incorporated GPC specimen was activated using a mixture of sodium silicate and sodium hydroxide solution with a sodium silicate/hydroxide ratio of 2.5. The low-calcium (LCFA-ECC) and high-calcium (HCFA-ECC) fly ash-based ECC specimens were produced, and the NC was also cast for comparison. The durability performances of concretes were assessed by visual inspections, weight changes, and compressive strength tests, and microstructures of the chemically exposed specimens were compared to that of the unexposed specimens by Scanning Electron Microscopy (SEM) micrographs. The results showed that LCFA-ECC samples showed the best performance, while NC samples performed the worst performance against all chemical attacks. The GPC specimens showed a better durability performance than HCFA-ECC specimens under SW and MS attacks, while HCFA-ECC specimens exhibited better chemical resistance against SA attack. SEM images confirmed that sulfuric acid is the most hazardous environment since increased dosage of sulfates was observed on the surfaces, interfacial transition zone (ITZ), and polyvinyl alcohol (PVA) fibers.

**Keywords** Geopolymer concrete · Engineered cementitious composite · Sulfuric acid attack · Magnesium sulfate attack · Seawater attack

## 1 Introduction

Structures should have sufficient durability properties in addition to adequate strength and stiffness during their service life. Chemical attacks such as seawater (SW), magnesium sulfate (MS), and sulfuric acid (SA) can be regarded as the main durability issue for the structural elements. The chemical attacks are responsible for the degradation of different structural elements by reducing the bond between matrix and aggregate. As a result, low mechanical strength and service life are obtained (Gülşan et al. 2018). Due to the increase in the prices of manpower and materials, the service life of the structures should be extended from 50 years to at least 100 years. Not only the structural building elements but also the infrastructure concrete elements in the urban areas, i.e., sewage pipes, drainage pipes, and seawater pipes should resist the chemical attacks for improved service life.

Researchers continue to find superior concrete types to obtain high mechanical strength and durability properties. Generally, engineering cementitious composite (ECC) is

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used for crack control and enhanced strain characteristics. The polyvinyl alcohol (PVA) incorporating ECC tensile strain capacity can be 300–500 times more than that of usual concrete (Xi et al. 2020) due to the multiple micro crack formation (Tastani et al. 2016). The ECC prevents the macro crack formation and thus reduces the penetration of aggressive ions (sulfate ions) to the inner parts of concrete materials, which results in improved chemical resistance and service life of the structures (Tastani et al. 2016; Liu et al. 2017).

The utilization of supplementary cementitious materials, i.e., fly ash has been widespread in the concrete production stages due to the reduction in the environmental impact of concrete. Fly ash can be regarded as an aluminosilicate source (Nis and Altundal 2019; Kadhim et al. 2022; Nis and Al-Antaki 2022), and it can be divided as low-calcium fly ash—F type and high-calcium fly ash—C type depends on its source. Both of them are less reactive than the cement material, and their use enhances the durability properties of the concrete (Aljanabi et al. 2022). Fly ash can be used as raw materials (Hasar et al. 2022; Nis and Cevik 2022) due to its finer size, chemical ingredients, and ease of access (Mehta and Siddique 2017; Tenapalli et al. 2018). The annual fly ash production content of Türkiye is approximately 15 million tonnes (Their and Ozakca 2018), and the utilization of this material instead of cement in concrete structures becomes significant in terms of environmental pollution. ECC concrete can be also produced by partial utilization of fly ash instead of cement. With the help of fly ash, a novel eco-friendly concrete, geopolymer concrete emerges due to a reduced amount of energy (Okoye et al. 2017) and decreased carbon dioxide emissions (Aljanabi et al. 2022). Recently, ongoing studies have been made to use geopolymer concrete (GPC) instead of ordinary concrete. GPC has superior mechanical properties and chemical resistance, and it performs lower shrinkage and creep than conventional concrete in addition to its environmental nature (Nis and Altundal 2021; Alzebaree et al. 2021; Niş et al. 2023). Considering the reduced energy consumption, cost, and CO<sub>2</sub> emission, fly ash-based geopolymer concrete can be more sustainable than OPC concrete as green material (Duxson et al. 2007; McLellan et al. 2011; Zhuang et al. 2016). Therefore, fly ash-based GPC has potential use in structural design applications (Ge et al. 2023; Niş and Altundal 2023; Sing et al. 2023; Yang et al. 2023).

The durability of concrete is comprehensively affected by its permeability, crack formation, and volume change (Philip and Syriac 2023). GPC should have better resistance against changing temperatures, water, acids, corrosion, and other harsh factors than usual concrete (Manzoor et al. 2023). Also, it was reported that GPC specimens show higher resistance than conventional concrete specimens against seawater conditions, sodium sulfate attack, and magnesium

sulfate attack (Kumar et al. 2022). However, there is a lack of research regarding the durability of ECC, GPC, and NC together.

The ECC and GPC are novel types of concrete, and researchers generally focus on the production parts and their mechanical properties with respect to ordinary concrete. So far, the standardization of the GPC and ECC types of concretes for structural use cannot be reached, and the lack of durability studies on these concretes can be the main obstruction to the standardization of widespread use. The sulfate attacks that arise from the groundwater, soil, and seawater can be detrimental to structural safety, and hence durability performance of structural concrete elements exposed to a sulfate environment becomes significant for the service life of the structures (Taylor et al. 2001; Tulliani et al. 2002). In addition, acid attacks can also arise from groundwater, sewage pipes, industrial waste, and acid rains, and therefore, the acid resistance of concretes is also significant for the structural elements (Bakharev 2005; Matalkah et al. 2018).

Although there are several studies regarding the seawater, magnesium sulfate, and sulfuric acid resistance of ordinary concrete, there are very limited numbers of studies regarding the chemical resistance of the geopolymer and engineered cementitious composite concretes. To date, no study has been realized to investigate the three chemical attacks on GPC and ECC together. The research aims to investigate the performances of the GPC and two types of ECC (HCFA-ECC and LCFA-ECC) under chemical environments and to find superior concrete under chemical attacks. For comparison, ordinary concrete (NC) was also produced and subjected to identical chemical environments. The effects of seawater (SW), magnesium sulfate (MS), and sulfuric acid (SA) attacks on the physical and mechanical performance were investigated via visual inspection, weight change, and compressive strength variation. The microstructural analyses were executed to see the effects of the chemical deterioration on the inner parts of GPC, HCFA-ECC, LCFA-ECC, and NC specimens. The results found in this research can give significant insight to protect structural elements against chemical attacks. Thus, NC, GPC, LCFA-ECC, and HCFA-ECC concretes were cast, and a comprehensive evaluation was carried out under seawater, magnesium sulfate, and sulfuric acid attacks.

## 2 Experimental Methods

### 2.1 Material

In the scope of the study, various concrete mixes with different water-to-cement and water-to-binder ratios were produced. The high-calcium FA-based ECC mix (HCFA-ECC), low-calcium FA-based ECC mix (LCFA-ECC), nanosilica

**Table 1** Chemical composition and physical properties of the binder materials

| Component     | CaO   | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | MgO  | SO <sub>3</sub> | K <sub>2</sub> O | Na <sub>2</sub> O | LOI  | SG   | BF (m <sup>2</sup> /kg) |
|---------------|-------|------------------|--------------------------------|--------------------------------|------|-----------------|------------------|-------------------|------|------|-------------------------|
| Fly ash-F (%) | 1,60  | 62,53            | 21,14                          | 7,85                           | 2,4  | 0,10            | 0,73             | 2,45              | 2,07 | 2,30 | 387                     |
| Fly ash-C (%) | 15,5  | 46,97            | 11,86                          | 7,98                           | 6,51 | 3,47            | 3,23             | 2,33              | 0,45 | 2,27 | 306                     |
| OPC (%)       | 62,58 | 20,25            | 5,31                           | 4,04                           | 2,82 | 2,73            | 0,92             | 0,22              | 3,02 | 3,15 | 326                     |
| NS (%)        | –     | 99,8             | –                              | –                              | –    | –               | –                | –                 | ≤1   | 2,2  | –                       |

**Table 2** PVA fiber properties

| Fiber | Tensile strength (MPa) | Modulus of Elasticity (GPa) | Elongation (%) | Diameter (μm) | Length (mm) |
|-------|------------------------|-----------------------------|----------------|---------------|-------------|
| PVA   | 1620                   | 42.8                        | 6              | 39            | 8           |

including geopolymer concrete mix (GPC), and an ordinary concrete mix (NC) were produced, which were exposed to different chemical attacks to evaluate durability performance of these mixes. For the materials, F types of FA, C types of FA, ordinary Portland cement (OPC), nanosilica (NS) for GPC mix, micro-silica sand for ECC mix, polyvinyl alcohol fibers (PVA) for ECC mixes, water, and high range water reducing admixture (HRWRA) were used in the production of these concretes. Based on ASTM C 618 standard, F types of fly ash is called low lime fly ash since the CaO content is below 10%. In addition, the total of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> is higher than 70% in F types of FA. The C types of fly ash is called as high lime FA since the CaO content is higher than 10% and the total of SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> is higher than 50%. Table 1 illustrates the physical properties and chemical compositions of the F and C types of fly ashes, NS, and OPC.

Table 2 presents the Polyvinyl alcohol (PVA) fiber properties used in this research. Generally, PVA fibers obtained from a local supplier are used by 2% volume in ECC for increased toughness and strain-hardening properties (Mohammedameen et al. 2019), and higher content of PVA fibers (> 2%) does not affect composite toughness (Pakravan et al. 2018). To reach a good bonding among PVA fibers and matrix and to have superior ECC behavior, the fiber surfaces were coated with 1.2% by a hydrophobic oiling agent mentioned by previous research (Li et al. 2002). The grain size of micro-silica sand can affect the ductility performance of

ECC (Sahmaran et al. 2009); thus, the maximum and average sizes of the micro-silica sand were 200 μm (0.008 in.) and 110 μm (0.004 in.), respectively.

## 2.2 Mix Design

Table 3 presents the mix proportions for the ECC mixes. For ECC mixes, water-to-cement (*w/c*) and water-to-binder ratios (*w/b*) were utilized as 0.56 and 0.27, respectively. Researchers pointed out that a *w/b* ratio of 0.3 was found as the best ratio for optimum rheological behavior and desirable strain-hardening performance of ECC (Meng et al. 2017). Therefore, the *w/b* ratio was selected as 0.27 to exhibit an enhanced strain-hardening response stated in the previous research (Yang et al. 2009). A high-range water-reducing admixture (HRWRA) was utilized to obtain the required fresh-state properties for ECC mixes. Two types of ECC were produced; one of them was produced with low calcium (F-type) fly ash and called LCFA-ECC, and another one was cast using a high calcium (C-type) fly ash and called HCFA-ECC.

For the production of the GPC series, F-type fly ash and nanosilica (NS) were obtained from a thermal power plant located in the Mediterranean region of Türkiye and Norway, respectively. In literature, the favorable effects of NS on the compressive strength of fly ash-based GPC (Adak et al. 2014; Gao et al. 2015), slag-based GPC (Çevik et al. 2018; Gülşan et al. 2019), and chemical durability (Çevik et al. 2018) were reported. For comparison, an ordinary concrete (NC) produced with CEM I 42.5 R type cement (OPC) with a water-to-cement ratio of 0.45 was also utilized. For the GPC geopolymerization, the Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio should be around 1.5–2.5 for economic reasons (Olivia and Nikraz 2012); thus, this ratio is chosen as 2.5. The Na<sub>2</sub>SiO<sub>3</sub> solutions (Na<sub>2</sub>O = 13.7%, SiO<sub>2</sub> = 29.4, water = 55.9%) and the NaOH solid in pellets having about 98% purity were taken at

**Table 3** The mix components of ECC mixtures

|      | Cement kg/m <sup>3</sup> | Water kg/m <sup>3</sup> | Micro silica sand kg/m <sup>3</sup> | F-type fly ash kg/m <sup>3</sup> | C-type fly ash kg/m <sup>3</sup> | HRWRA* kg/m <sup>3</sup> | Fiber kg/m <sup>3</sup> |
|------|--------------------------|-------------------------|-------------------------------------|----------------------------------|----------------------------------|--------------------------|-------------------------|
| LCFA | 570                      | 338                     | 455                                 | 684                              | –                                | 5.1                      | 26                      |
| HCFA | 570                      | 338                     | 455                                 | –                                | 684                              | 5.1                      | 26                      |

\*HRWRA: High-range water-reducing admixture; all ingredients amount, by weight, except for PVA fibers

a local firm. The NaOH molarity is adjusted to obtain 14 M one day before casting day. This molarity shows the lowest resistance against chemical attacks (Kumaravel and Girija 2013). The 14 M sodium hydroxide mixture was mixed with the sodium silicate liquid with a modulus ( $M_s$ ) of 2.14 at least 24 h before casting. Master Glenium RMC 303 type high-range water reducing admixture (HRWRA) was also utilized to obtain the required fresh state performance. Table 4 illustrates the mix ingredients of NC and GPC with a water-to-cement and alkali-to-fly ash ratio of 0.45. After several trial batches, the mix components were determined as given in Table 4.

During mixing, solid materials were prepared in a mixer at low speeds for 4 min, and water was incorporated and mixed for an additional 2 min. Then, the liquid mixture was mixed at high speeds for 5 min, and a high range water reducing admixture (HRWRA) was added and again mixed for an additional 5 min. Later, PVA fibers were incorporated and mixed at high speeds for 5 min. Finally, this mixture was continued at high speeds for an additional 3 min to avoid fiber accumulation and obtain a uniform mix. For geopolymer mixtures, dry materials were incorporated into the mixer and mixed for 2.5 min. The alkali activators and HRWRA were incorporated in 1 min and further mixed for 2.5 min for homogeneity.

### 2.3 Curing Procedure

In this study, 100 × 200 mm cylinder specimens were produced using mixes reported earlier to evaluate mechanical strength deterioration of the concretes subjected to various chemical exposures. The water-curing was applied to the ECC and NC specimens, and oven-curing was applied to the GPC specimens, and all tested specimens were cylinder specimens with a diameter of 100 mm and a length of 200 mm.

The ECC and NC cylindrical specimens were demolded a day later upon casting and cured in water at  $23 \pm 2$  °C for 28 days. For the GPC specimens, the samples were covered by plastic bags and stored in the laboratory condition. Then, specimens without demolding were cured in an oven at 70 °C for 48 h. Then, specimens were removed from the oven, let to cool down, and demolded. After that, the samples were kept in the laboratory at  $23 \pm 2$  °C for up to 28 days.

### 2.4 Chemical Exposure

For the chemical exposure, there is a lack of procedure to investigate the effects of different environments on the durability performance of the concrete specimens. However, the ASTM C 267 standard proposed that specimens should be in water-saturated conditions before chemical solution exposures. Thus, all samples were soaked in drinkable tap water for 24 h stated by the standard before chemical exposures. Then, specimens were kept in the laboratory environment for 2 h for drying, and the weights of the specimens were measured as initial weights. For the immersion tests, the specimens are generally exposed to 3.5% seawater solutions to simulate saline/marine conditions in a corrosive environment (Okeniyi et al. 2016). Therefore, 3.5% seawater solutions were prepared first in the laboratory, and some of them were put inside containers after weight measurements, and 3.5% seawater solutions were poured into the containers.

For magnesium sulfate and sulfuric acid solutions, 5% magnesium sulfate (MS) and 5% sulfuric acid (SA) solutions are the most commonly used aggressive environments for concretes (Roy et al. 2001; Bakharev et al. 2002). Thus, 5% MS and SA solutions were prepared first, and specimens were located in containers, and these 5% prepared solutions were cast into the containers.

In addition, some of the specimens were held in the ambient environment for 60 days for comparison. Three identical specimens were produced for different chemical environments, and the average values were calculated for each concrete. The obtained results represent the average results of three companion specimens.

### 2.5 Chemical Evaluation

The compressive strengths of the specimens were measured using the ASTM C39 standard at the end of the chemical exposures, and the deterioration amount was determined due to the decrease in the compressive strength of the specimens. The compressive strength tests of the specimens were realized on cylinder specimens having a diameter of 100 mm and length of 200 mm at 90 days. All compressive strength tests were executed under displacement-controlled loading with a rate of 0.2 mm/min. Two linear variable displacement transducers (LVDT) were attached to measure axial deformations in the specimens

**Table 4** Mixtures proportions of GPC and NC (kg/m<sup>3</sup>)

| Mix | FA  | Cement | Na <sub>2</sub> SiO <sub>3</sub> + NaOH | Water | Fine Agg | Coarse Agg | NS | HRWRA | Na <sub>2</sub> SiO <sub>3</sub> /NaOH | A/B  | W/C  |
|-----|-----|--------|-----------------------------------------|-------|----------|------------|----|-------|----------------------------------------|------|------|
| GPC | 485 | —      | 225                                     | —     | 549.5    | 1098.8     | 15 | 7.5   | 2.5                                    | 0.45 | —    |
| NC  | —   | 476    | —                                       | 214.2 | 562.6    | 1125       | —  | 2.38  | —                                      | —    | 0.45 |

All numbers are weight of the materials (kg/m<sup>3</sup>) except Na<sub>2</sub>SiO<sub>3</sub>/NaOH, alkali/binder (A/B), W/C

**Fig. 1** Compressive strength determination of cylinder specimens



shown in Fig. 1. Three companion specimens were tested, and the average values were taken as compressive strength for each series. As well as compressive strength, visual inspection and scanning electron microscope (SEM) observations were also performed on exposed and unexposed specimens to observe the degradation amount of the different concretes in micro-scale due to SA, MS, and SW environments. The results were compared with the unexposed (control) specimens that were kept in an ambient environment. All ECC and NC specimens were cured in water for 28 days, GPC had an oven-curing at 70 °C for 48 h, and they were left for 26 days under a laboratory environment. Then, all specimens were soaked in water for 24 h to obtain a water-saturated condition. Then, specimens were kept in different chemical environments for 60 days and left for drying in a laboratory environment for 24 h before mechanical tests.

For the SEM observations, the chemically affected zone of cylinder specimens was found only 20 mm from the outer surface (20 mm out of 100 mm) in earlier research (Attiogbe and Rizkalla 1988), and this study also confirmed that the chemical deterioration of the specimen initiates from the surface and then spreads to the interior regions (Aitcin 2003). It was observed that sulfuric acid attack was hazardous at the outermost 20 mm diameter, while MS and SW attacks can be detrimental from the outermost 8–10 mm diameter. For this reason, the outer surfaces of the cylinder specimens were investigated in micro-scale. For the preparation of the SEM sample, one-inch slices (~25 mm) were cut from the top and bottom regions, and these slices were removed. Then, a thick circular slice of 1 cm from the interior regions was obtained. As a result, 1 cm × 1 cm × 1 cm concrete samples were utilized for the SEM investigations (0–1 cm from the outermost layer), similar to the previous investigation (Mohammedameen et al. 2019).

### 3 Experimental Outcomes and Discussion

#### 3.1 Visual Inspection

Visual appearances of samples after 60 days of exposure to chemical solutions were evaluated, and the findings pointed out that the sulfuric acid environment was found most hazardous, while seawater solution was found the least dangerous environment to specimens. Figure 2 illustrates the visual appearances of the specimens under various chemical attacks. All specimens kept their initial conditions without color change under the SW solution. It was reported that concrete specimens subjected to the SW environment for many years were found to be in good condition, and no deterioration was observed (Kurtoglu et al. 2018). For the specimens exposed to the sulfate environment, no visual degradation was observed for the LCFA-ECC specimens, while a very thin layer of efflorescence was visible for the surfaces of the HCFA-ECC, NC, and GPC specimens, which caused softening in the outermost layer of the specimens. Despite the softening, the concretes remained intact from the structural point of view. Similar observations were reported in the earlier investigations (Gülşan et al. 2018; Matalkah et al. 2018). On the other hand, for the specimens subjected to the acid environment, a severe color change was observed in the HCFA-ECC and NC specimens, and the surface color of these specimens changed from grey to white. This color variation may be attributed to the high calcium oxide contents, which causes a formation of the white layer of gypsum crystals in the HCFA-ECC and NC specimens, leading to higher degradation. For the GPC specimens, the color of the specimens changed from grey to light green, and severe deterioration was also observed. Meanwhile, the least surface erosion and reduced number of gypsum crystals were observed for the LCFA-ECC specimens due to lower CaO content compared to HCFA-ECC and NC specimens. Also,

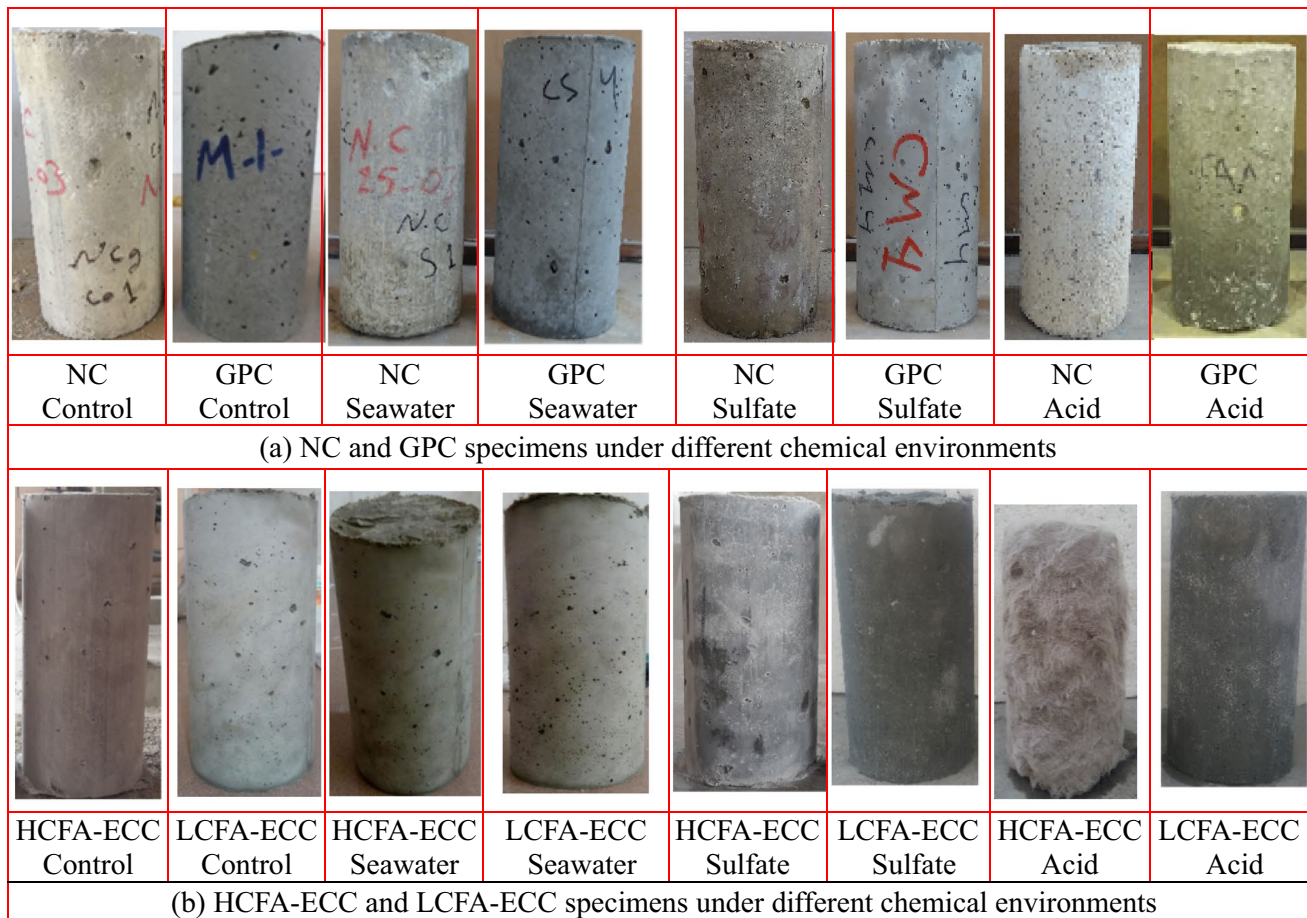


**Fig. 2** Visual inspection of specimen under different chemical environments

the surface color of the LCFA-ECC specimens remained grey at the end of the acid exposure. The higher surface deterioration was observed in the HCFA-ECC specimens under both MS and SA environments.

### 3.2 Weight Change

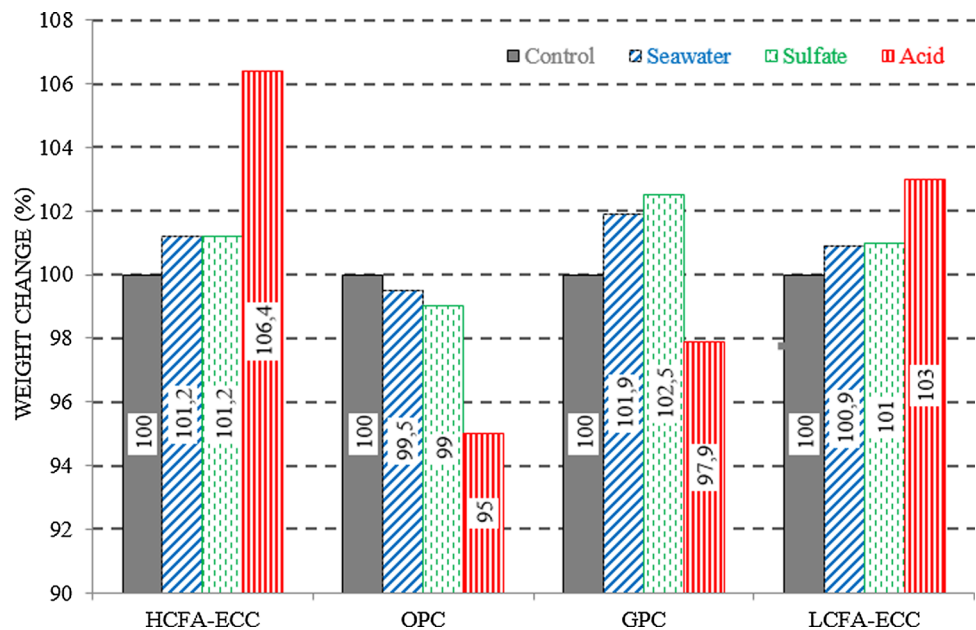
Figure 3 illustrates the variation in weight (%) of specimens due to chemical exposure compared to the control environment. First of all, weight gain was expected in the specimens due to the solution absorption. Then, weight loss started to be observed in the specimens due to the chemical reactions. The weight loss can be due to the loss of particles on the surfaces due to ongoing chemical reactions and ettringite formation (Li et al. 2023). The degradation starts to occur at surfaces and then progresses to the inner parts of the specimens.

For HCFA-ECC specimens, moisture uptake or weight gain was observed under all chemical environments. The rise in the weights of the HCFA-ECC samples was 1.2%, 1.22%, and 6.4% due to the SW, MS, and SA environments, respectively compared to ambient or control environments.

For NC specimens, weight loss was observed under all chemical attacks. The decrease in weight of NC samples was 0.5%, 1%, and 5% for SW, MS, and SA attacks, respectively. For the GPC specimens, 1.9% and 2.5% weight gain was observed due to the SW and MS environments, while 2.1% mass loss was observed due to the acid attacks. For the LCFA-ECC specimens, weight enhancement was observed for all chemical environments, and the amount of weight increase was found to be 0.9%, 0.98%, and 3% for SW, MS, and SA attacks, respectively. The higher weight gain and/or higher weight loss is an indicator of the high amount of deterioration. Therefore, the SA attack was the most detrimental, while the SW attack was the least hazardous environment for specimens. Among the concrete types, HCFA-ECC specimens (6.4%) showed a higher weight increase (the highest deterioration), and LCFA-ECC specimens (3%) exhibited the lowest weight gain (the lowest deterioration) under sulfuric acid attack.

For the acid environment, the highest weight increase for HCFA-ECC samples can be due to high CaO content in the C-type fly ash and the chemical absorption of PVA fibers. Due to the more calcium oxide contents, the ratio of

**Fig. 3** The change in weight for the samples exposed to various chemical environments for 60 days



Ca/Si rises, and the free calcium leads to the degradation of the cement-based paste and formations of ettringite and gypsum. Therefore, expansions, dimensional instabilities, cracks, spalling, and losses of mechanical performance were observed (Bassuoni and Nehdi 2007; Ariffin et al. 2013). The lowest weight increase for the LCFA-ECC samples can result from both low calcium oxide (CaO) content, decreased porosity, and reduced chemical absorption due to the high fineness ratio (Thokchom 2014). The weight gain resulting from the sulfuric acid environment is realized due to the chemical absorption, and this was also stated in the earlier investigations (Reddy et al. 2018). Due to chemical reactions between acidic medium and binder materials of concrete, calcium hydroxide may transform into calcium sulfate (gypsum) and calcium sulfoaluminate (ettringite) (Soroka 1980). The existence of calcium oxide (CaO) in concrete with a sufficient amount of chemical solutions or harmful ions from the surrounding environment can be detrimental to the service life of the concrete material. For NC and GPC specimens, weight losses were observed, and this can be due to the material and alkali dissolution from concrete to the surrounding acid environments. The highest weight loss was observed in NC specimens because of the increased CaO amount since calcium accelerates the ettringite and gypsum content, which becomes hazardous by increasing solid volume approximately twofold (Soroka 1980), raising tensile stresses inside the concrete material. As a result, cracking of concrete and material dissolution can be observed under the acidic medium.

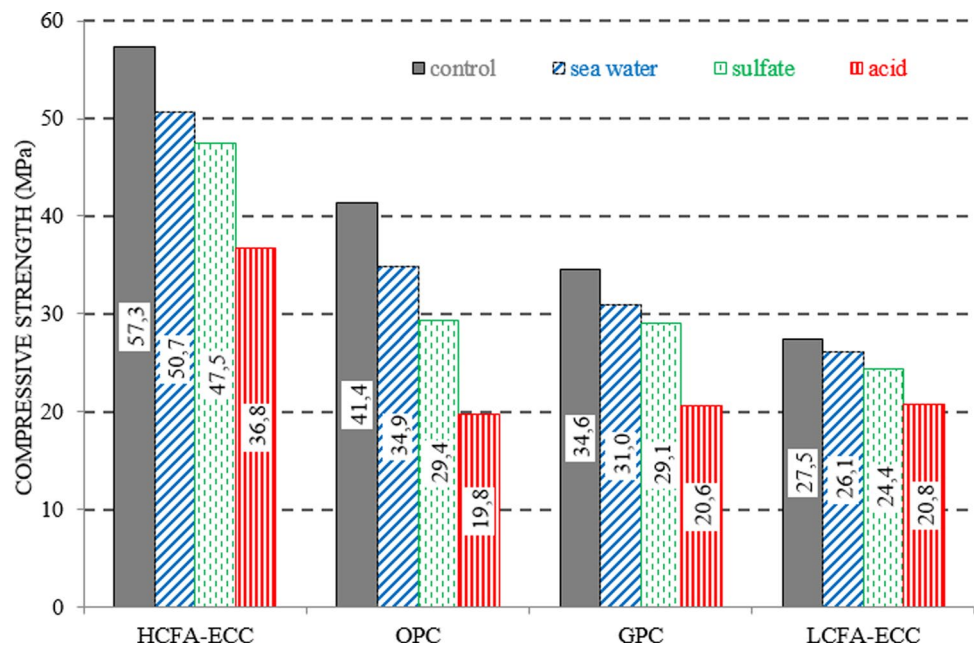
The samples under seawater and magnesium sulfate environments showed moisture uptake, except for the NC samples. The maximum weight increment was obtained

in geopolymer samples, which can result from the higher porosity due to the heat curing of GPC specimens. As a result of the heat curing (70 °C for 48 h), small micro-cracks were observed on specimen surfaces, and further chemical absorption was realized from sulfate or seawater medium to the inner region of the concrete. The LCFA-ECC specimens absorbed less chemical solution than the HCFA-ECC ones, which is due to the decreased porosity resulting from the high fineness ratio of F-type fly ash than C-type fly ash. Weight increment due to the absorption of chemicals was also stated earlier (Thokchom et al. 2010). Meanwhile, weight loss of 0.5% and 1% was observed in NC specimens due to seawater and magnesium sulfate environments, respectively. This may be attributed to the material dissolution due to gypsum formation from the specimen surfaces to the surrounding media. A similar result was also obtained in the earlier investigation (Çevik et al. 2018).

### 3.3 Change in Compressive Strength

Figure 4 indicates the compressive strength variation of the specimens under various chemical environments. It should be remembered that water-to-cement and water-to-binder ratios were 0.56 and 0.27 for the HCFA-ECC and LCFA-ECC specimens, respectively. On the other hand, the water-to-cement ratio was 0.45 for NC specimens, and the alkaline-to-fly ash ratio was 0.45 for the GPC specimens. Compressive strength values of the unexposed (control) specimens were 57.3 MPa, 41.4 MPa, 34.6 MPa, and 27.5 MPa for HCFA-ECC, NC, GPC, and LCFA-ECC samples, respectively. The highest compressive strength was obtained for the HCFA-ECC sample, and the lowest

**Fig. 4** The change in compressive strength for the samples under chemical environments for 60 days



compressive strength was reached for the LCFA-ECC sample. In addition, NC specimens exhibited more compressive strengths than GPC samples under ambient environments.

Due to the softening and material dissolution, compressive strength values were reduced under all chemical environments. The results indicated that the amount of reduction in compressive strength was lowest for the seawater environment, while highest for the sulfuric acid environment. For the specimens exposed to seawater environment, compressive strength (CS) reductions were 12%, 16%, 10%, and 5% for the HCFA-ECC, NC, GPC, and LCFA-ECC specimens, respectively. For the specimens subjected to magnesium sulfate solution, CS reductions were 17%, 29%, 16%, and 11% for the HCFA-ECC, NC, GPC, and LCFA-ECC specimens, respectively. The reductions in compressive strengths due to sulfuric acid were 36%, 52%, 40%, and 24% for the HCFA-ECC, NC, GPC, and LCFA-ECC samples, respectively. The findings pointed out that the decrement in compressive strengths after exposure to seawater and magnesium sulfate environments followed a trend: NC > HCFA-ECC > GPC > LCFA-ECC. However, in the acidic medium, the strength reduction became in the order of NC > GPC > HCFA-ECC > LCFA-ECC. It should be emphasized that GPC specimens deteriorated more than HCFA-ECC specimens under acid attack, while GPC specimens deteriorated less than HCFA-ECC specimens under seawater and sulfate environments. This can be inferred that the alkaline solution (sodium silicate and sodium hydroxide) can be influenced adversely by the sulfuric acid attack.

The degradation mechanism under magnesium sulfate and seawater attack can be identified as diffusion of sulfate ions from an environment to the matrix reacting with

C<sub>3</sub>A to compose gypsum and ettringite, resulting in volume increase and degradation of concrete. As a result of the chemical attack, Brucite is formed and this prevents the hazardous effects of chemical attacks at the beginning phases (Bondar et al. 2015). However, in the upcoming stages, calcium silicate hydrate (CSH) gel transforms into magnesium silicate hydrate (MSH) gels thereby, softening and the reduced mechanical strength observed in concrete (Kurtoglu et al. 2018). Also, the alkalis in GPC specimens diffuse into the seawater or sulfate environment and calcium, magnesium, and sodium ions diffuse into the matrix to give a reaction with sodium hydroxide and sodium silicate. As a result of these reactions, delayed ettringite forms, cause a reduction in mechanical strength (Mataalkah et al. 2018).

The degradation mechanism of specimens produced with OPC concrete under a sulfuric acid environment can be clarified that calcium silicate hydrate (C-S-H) and sodium aluminate silicate hydrate (N-A-S-H) decalcify with the existence of sulfuric acid, thus the ratio of Ca/Si decreases (Zhang et al. 2017). Specimens including OPC include a high amount of Ca content; therefore, the ratio of Ca/Si rises, and free Ca can cause gypsum and ettringite formation, which results in spalling, cracking, and deterioration of mechanical strength (Türker et al. 1997; Ariffin et al. 2013). For the GPC specimens, the mechanical strength decreased due to the acid environment resulting from the destruction of the oxy-aluminum bridge of the geopolymeric gel, which is responsible for both strength and bond improvement in the GPC (Chindaprasirt et al. 2013; Karthik et al. 2017).

### 3.4 Scanning Electron Microscopy (SEM) Evaluation

Figures 5 and 6 illustrate the scanning electron microscopy (SEM) observations of the specimens under different chemical environments. SEM observations were performed to observe the changes in the microstructure of the chemically exposed specimens. It should be noted that SEM samples were taken from the chemical affected zone of 0–10 mm (from the surface to the interior regions) because chemical attacks initiate from the surfaces of the concrete and progress to the interior regions (Çevik et al. 2018). Results indicated that the white sediments or sulfates formed at the surface and interfacial transition zone (ITZ) of the specimens, and the resulting formation of gypsum and ettringite can be responsible for the expansion, cracking, spalling, and strength loss (Bassuoni and Nehdi 2007; Karthik et al. 2017). For the seawater (SW) attack, cracking was observed in NC and GPC specimens; however, the width of the crack (deterioration) was higher in NC specimens than the GPC specimens. In addition, PVA fibers were also influenced by the SW solution, and much more sulfates were observed on the ITZ zone and PVA fibers for HCFA-ECC samples than the LCFA-ECC ones. The higher deterioration of HCFA and NC specimens may be attributed to the high CaO contents, high Ca/Si ratios, and free calcium. The degradation effect of CaO was also reported in the earlier investigation (Çevik et al. 2018).

Figures 5e, f, 6e, and f illustrate the SEM observation of the specimens exposed to magnesium sulfate (MS) attack. According to the SEM results, the number of sulfates observed under the MS was much more than in the SW attack. The white sediments were seen to be more in NC samples, and the width of the formed crack was bigger (higher deterioration) than the crack width of the NC specimen under the SW attack. For the HCFA-ECC specimens, the increased number of white deposits on the ITZ zone and PVA fibers can be easily seen and these deposits reduced the bond and led to loss of mechanical performance. For GPC specimens, nanosilica and fly ash filled the crack and reduced the porosity and permeability. However, the formed sulfates on the crack locations deteriorated the interfacial bond. The least number of sulfates were observed in LCFA-ECC specimens due to the low CaO content and the low free calcium amount.

Figures 5g, h, 6g, and h illustrate the SEM investigations of the specimens subjected to sulfuric acid (SA) attack. From the SEM micrographs, it is easily understood that the SA is the most dangerous environment to the specimens since much more sulfates (ettringite) were observed on the surfaces, ITZ zone, and PVA fibers (for ECC matrix only). The formed ettringite is responsible for the deterioration of the specimens by increasing the weight of the specimens and deteriorating the bond between the matrix, aggregate/

matrix, and PVA fibers. The maximum amount of ettringite was observed for the NC sample. Then, much more sulfates were observed on the GPC specimens than on the HCFA-ECC specimens. Therefore, the GPC specimens exhibited more strength decrement than the HCFA-ECC ones. This may explain why the alkalis of the GPC specimens can be influenced more by the acid attack than the HCFA-ECC specimens. In addition, the PVA fibers may be dominant in the strength performance when the matrix is weakened due to acid attack. Meanwhile, LCFA-ECC specimens showed less deterioration due to the lower amount of free calcium content.

## 4 Conclusions

In the research, the durability performance of the high and low calcium fly ash-based ECC specimens (HCFA-ECC and LCFA-ECC), geopolymer concrete specimens including nanosilica (GPC), and ordinary concrete (NC) samples were investigated after 3.5% seawater (SW), 5% magnesium sulfate (MS) and 5% sulfuric acid (SA) attacks. The changes in concrete visual appearances, specimen weights, and mechanical strengths due to the various chemical attacks were evaluated on a macro-scale, and SEM observations were also realized to observe the effects of the various chemical attacks on a micro-scale. The following results are obtained:

- Visual inspection results under SA indicated that the color change and surface deterioration occurred on specimens exposed to the SA environment. Severe deterioration and color change were observed on the surfaces of the NC, HCFA-ECC, and GPC samples, whereas LCFA-ECC samples remained in their initial color and condition under SA attack.
- Visual inspection of the specimens under MS and SA attacks showed that low efflorescence was observed on the surfaces of some of the samples; however, they were found to be structurally intact and seem to be in good condition visually.
- Weight change results showed that weight gain was observed for HCFA-ECC and LCFA-ECC specimens under all chemical attacks and the amount of weight gain increased in the order of SA, MS, and SW environments. The highest weight increase was observed on HCFA-ECC specimens, whereas the lowest weight gain was observed on LCFA-ECC specimens.
- Weight change results also pointed out that for the GPC specimens, weight loss was observed under acid attack, while weight gain was observed under SW and SA environments, and the weight gain was more for the SA attack. For the NC specimens, the weight loss was observed under different environmental attacks, and the

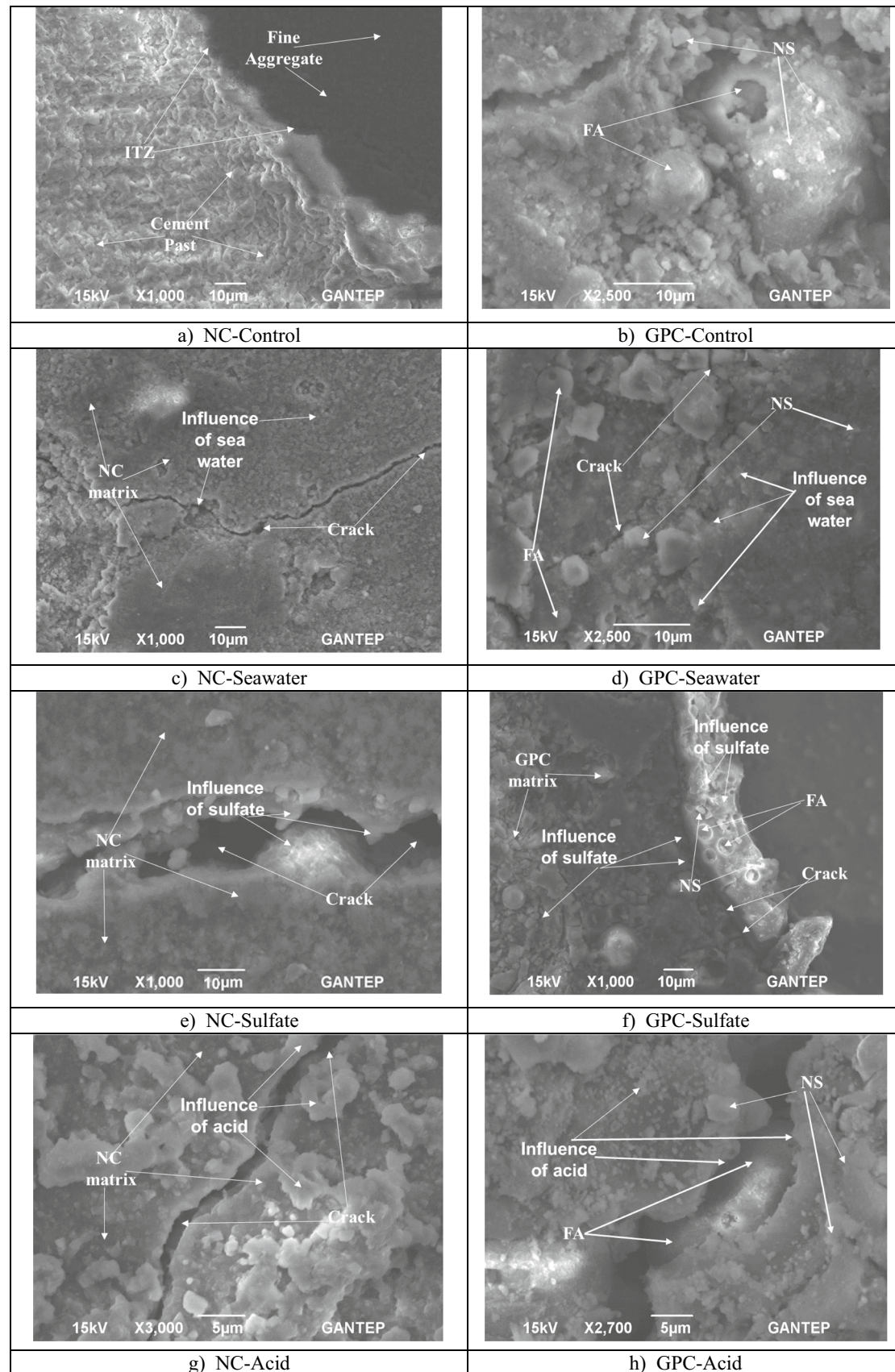


**Fig. 5** SEM observations of NC and GPC specimens under various chemical environment

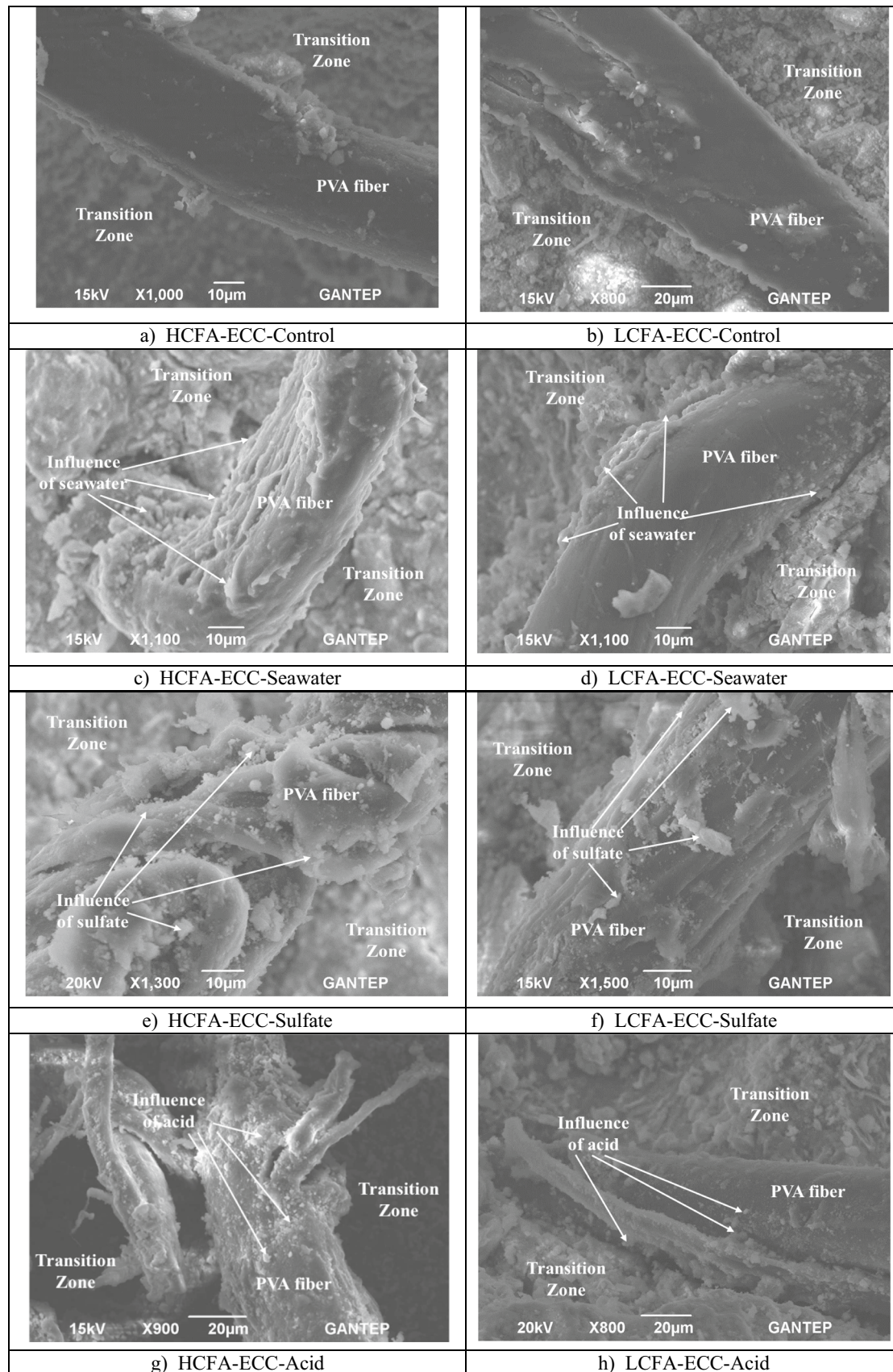


Fig. 6 SEM micrographs of HCFA-ECC and LCFA-ECC specimens under various chemical environment

highest weight loss was observed on specimens subjected to acid attack.

- The compressive strength results of the specimens at the age of 90 days under the ambient environment indicated that HCFA-ECC specimens showed the highest compressive strength and LCFA-ECC specimens showed the lowest compressive strength. The high strength can be attributed to the high CaO amount in the matrix. Also, the compressive strength of the NC specimens was found to be more than the compressive strength of the GPC specimens. The 90-day compressive strength of the specimens under SA attack declined by 52%, 40%, 36%, and 24% for the NC, GPC, HCFA-ECC, and LCFA-ECC specimens concerning control counterparts. The compressive strength of the specimens under MS attack was reduced by 29%, 17%, 16%, and 11% for the NC, HCFA-ECC, GPC, and LCFA-ECC specimens, respectively. For the SW attack, compressive strength decreased by 16%, 12%, 10%, and 5% for the NC, HCFA-ECC, GPC, and LCFA-ECC specimens, respectively.
- The compressive strength results indicated that the decrease in compressive strength due to the SA attack was 3–4 times more than the SW attack and approximately 2 times bigger than the MS attack.
- SEM observations illustrated that much more sulfates (ettringite) were observed on the surfaces, ITZ zone, and PVA fibers (for ECC matrix only) of the specimens exposed to SA environment than SW and MS environments, indicating that SA attack was the most hazardous attacks among the chemical attacks. The amount of sulfate increased with the following trend: seawater < magnesium sulfate < sulfuric acid environments.
- SEM images confirmed the compressive strength results that much more sulfates (ettringite) occurred on NC specimens, and lower sulfates were observed on LCFA-ECC specimens. Therefore, The LCFA-ECC specimens exhibited the superior, while the NC specimens performed the poorest performance against all chemical attacks. When HCFA-ECC and GPC specimens were compared, GPC specimens exhibited better performance against SW and MS attacks, while HCFA-ECC specimens performed better resistance against SA attacks.
- For the reason of the deterioration due to chemical attacks, the high CaO content is responsible for the reduction in the compressive strength or high deterioration of the cement incorporating specimens since CaO caused the formation of expansive gypsums and ettringites, which results in the degradation of the mechanical strength. For the GPC specimens, demolition of the oxy-aluminum bridge of the geopolymeric gel can be responsible for the loss of bond and the reduced mechanical strength.

**Author contributions** All authors reviewed the manuscript.

## Declarations

**Conflict of interest** The authors declare no competing interests.

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