

Assessment of geopolymer composites durability at one year age

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ABSTRACT

Geopolymers are important alternative materials for use in support of recycling and sustainability, and one of the most important properties is durability. While many studies perform durability tests 28 days after production, we carried out durability tests on a mixture of geopolymers starting 365 days after production. In this case, other conditions that occur until durability conditions are applied are taken into consideration. The geopolymer mixture consisted of metakaolin (90% by wt.) and boron waste colemanite (10% by wt.). The study investigated heat and wet-dry curing methods and polyolefin and polyamide fibre ratios (0.5, 1.0, and 1.5% by vol.) on durability. Durability issues related to concrete, such as long-term exposure to hydrochloric acid, freeze-thaw cycles, and abrasion were recorded. Results show that polyamide fibres provide better results than polyolefin fibres, the optimum polyamide fibre ratio was 1%, and the wet-dry curing method increased geopolymerisation more than heat curing. The Si-O-Al bonds were found to be stronger after wet-dry curing. All durability studies showed that the compact structure of the geopolymers withstood the durability tests performed one year after production. Scanning electron microscopy (SEM) analysis supports these results.

1. Introduction

Geopolymers are environmentally-friendly alternatives to traditional Portland cement [1]. Their production uses less energy and emits fewer greenhouse gases than and they are more resistant to common durability issues. Geopolymers are inorganic polymers with three-dimensional Si-O-Al frameworks synthesized from aluminosilicates that can be dissolved in an alkaline medium [2]. When the durability properties were examined, they have been found to have a high rate of resistance to fire and chemicals [3,4]. This suggests that geopolymers may be used as construction material in buildings, refractories, and wastewater treatment facilities [5–8]. While many studies carried out durability tests starting 28 days after production, we conducted tests one year after production to evaluate the long-term durability based under various conditions.

Resistance to hydrochloric acid is a significant durability issue for concrete used in environments with caustic conditions. Concrete structures may be exposed to acids as a result of urban, industrial and agricultural activities [9–13]. The quality and type of concrete, type of acid, and the solution's concentration, pH, and fluidity influence acid resistance [14,15]. Acid attacks increase permeability and porosity, resulting in loss of alkalinity, weight, and strength. Geopolymers have emerged as

a promising solution to acid damage due to their ceramic-like micro-structure. While many studies have been done on the durability of fly ash and slag based geopolymers, data is needed to determine the acid resistance of metakaolin-based geopolymers [16–19]. Also, there are a limited number of studies on the resistance of geopolymers to the freeze-thaw cycle. Cai et al. [20] performed the freeze-thaw test for a slag based geopolymer concrete. The resistance coefficient was near 90% and the specimens had high resistance. Yunsheng et al. [21] produced polyvinyl alcohol reinforced geopolymer samples for a 20 cycle test; hardness tests and impact strength exhibited no influence on the specimens. The use of polyolefin and polyamide fibres in geopolymer production should also be investigated. When previous studies were examined, it was observed that bending strength and cracks were reduced by these fibres [22,23].

Numerous studies have been conducted on the type and duration of curing in the production of geopolymers. Aygörmöz et al. [24] used different temperatures and durations, and found that the most suitable curing condition was 72 h at 60 °C. Arslan et al. [25] applied the wet-dry method as a curing condition with the heat curing method. Improvement in mechanical properties was observed with this treatment. While the hydration reactions of the high calcium compounds are supported during wet curing, the strength of the geopolymerisation products

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increases and the bonding improves during the dry curing. Also, it was observed that metakaolin and colemanite are suitable for geopolymer production. Uysal et al. [26] substituted up to 40% colemanite with metakaolin and found that 10% of colemanite substitution had positive effects [27].

The main purpose of this study was to start the tests after 365 days to evaluate long term durability. Three different durability tests (hydrochloric acid, freeze-thaw, and abrasion) were selected. A general evaluation was made instead of evaluations by individual tests. Metakaolin and boron waste colemanite were used in a mixture of 90% metakaolin plus 10% boron waste colemanite by weight. Also, three different ratios (0.5, 1.0, and 1.5% by volume) of polyolefin and polyamide fibres were used. Two different curing methods (heat curing and wet-dry curing) were applied. After the freeze-thaw test of 300 cycles and the hydrochloric acid test (10% by volume) for six months, weight loss and strength results were examined. SEM analyses were performed after the durability tests. The abrasion test was applied to the samples twice before and after 500 °C temperature. The effect of the double durability test was investigated by applying the abrasion test together with the high-temperature test. Thus, the abrasion degree after the high temperature was studied. Also, SEM analyses were performed after the durability tests.

2. Materials and methods

2.1. Materials

Metakaolin, the main raw material, was obtained from the Kaolin EAD company (Turkey). The $\text{Fe}_2\text{O}_3+\text{Al}_2\text{O}_3+\text{SiO}_2$ ratio of metakaolin was 97.18%. The pozzolanic activity index of the metakaolin additive determined by the supplier was 123.5% for the 7th day and 141.4% for the 28th day. Due to this situation, the metakaolin was determined to have a high pozzolanic activity ratio. Metakaolin is a very fine-grained material that easily reacts to form geopolymeric bonds. It has fine grains because the amount of metakaolin passing through a 45 μm sieve is 0.30%. Colemanite was supplied by the Eti Maden Boron and slag was provided by the Bolu cement industry A.S. company. The chemical composition of the raw materials is shown in Table 1. In this study, C, MK, and S are abbreviations for colemanite, metakaolin, and slag, respectively.

In this study, a mixture of sodium hydroxide and sodium silicate was used as the alkali activator and standard sand was used as the aggregate according to Rilem Cembureau. The $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio of sodium silicate solution obtained from Merck was equal to 3.29 and sodium hydroxide 99% pure was also obtained from Merck, dissolved in distilled water and prepared at a concentration of 12 M. Three different ratios (0.5, 1.0, and 1.5% by volume) of polyolefin and polyamide fibres were used. Table 2 presents the fibres' properties studied in the durability tests. PL and PA were used as abbreviations for polyolefin and polyamide fibres, respectively.

2.2. Mix design and specimen preparation

Materials used for this study were slag, metakaolin, colemanite, standard sand, sodium hydroxide, and sodium silicate. The amounts for the mortar mixture are given in Table 3 for 450 g (90% metakaolin+10% boron waste colemanite by weight). In Table 3, the binder/sand ratio was 1:2.5 and the binder/activator ratio was 1:1.

Table 1
The raw materials' chemical compositions.

Chemical compositions %	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	B_2O_3	L.O.I.
MK	56.10	40.25	0.85	0.55	0.19	0.16	0.55	0.24	–	1.11
S	40.60	12.83	1.37	0.75	36.08	6.87	0.68	0.79	–	0.03
C	5.00	0.40	0.08	–	26.02	3.00	–	0.50	40.00	25.00

Table 2
The fibres' properties.

Fiber Type	Length (mm)	Diameter (μm)	Specific Gravity	Nominal Tensile Strength (MPa)	Aspect Ratios
PL	10	63	0.90	275	101.89
PA	10	55	1.14	900	115.5

Table 3
Standart mixing quantities (g).

Metakaolin + Colemanite	Sand	Slag	NaOH (12 M)	Na_2SiO_3
450	1125	60	150	300

A detailed description of the mixture preparation can be described as follows. First, the sodium hydroxide solution was prepared as 12 M using distilled water the day before the mixture was prepared. Next, the sodium hydroxide solution was premixed with sodium silicate on the mixture day at least 1 h before use in the mortar to allow the liquids time to cool down. The activator solution was mixed with the binder material in a ratio of 1:1 using a mixer drill. Sixty grams of slag were added to increase the calcium content, at a rate of 13% of the binder material. Finally, standard sand at a quantity of 2.5 times that of the binder material was added. Preparation of the mixture was carried out for a total of 6 min—a 2-min dry mixture (metakaolin + colemanite + slag), a 2-min mixture with activator solution, and a mixture with aggregate in the last 2 min. The mortar sample was then placed in the mould and subjected to vibration.

The samples were cured at 60 °C for 72 h, 24 h after removal from the mould. The samples were then kept at room condition for up to 365 days or until test day. Wet-dry curing was applied along with heat curing. Although the initial periods were the same for the two curing methods, during wet-dry curing, one cycle of wet-dry was applied in addition to heat curing. One wet-dry cycle included submersion in water for 3 days followed by drying in oven for 3 days at 60 °C. After wet-dry curing, the samples were stored at room conditions until the end of 365 days.

Within the scope of the study, seven series were prepared and 90% MK + 10% C (by weight) was used as the control mixture. The 2nd, 3rd, and 4th series were produced by adding at volume fractions of 0.5, 1.0, and 1.5% polyolefin fibres to the control mixture, respectively. The 5th, 6th, and 7th series were produced by adding 0.5, 1.0 and 1.5% volume fractions of polyamide fibres to the control mixture, respectively. The first seven series were heat cured and the other seven series were produced by wet-dry curing to the same series, and denoted by the abbreviation W.

2.3. Test procedure

An ASTM C944 [28] rotary cutter was utilized for the abrasion test. The geopolymer samples were abraded for 2 min with a 98 N load. Cylinder samples of 10 cm diameter and 7.5 cm height were produced for this test. The same procedure was repeated four times per sample and the weight loss was measured each time, from which the average weight loss was determined. The samples were kept at 500 °C and then the abrasion test was performed again to see the effect of high-temperature together with the abrasion resistance. A total of 300 cycles were applied in all freeze-thaw tests. One freeze-thaw cycle includes temperatures of –20 °C for 12 h and +20 °C for 12 h.

The geopolymer samples were stored in a plastic box for six months at the end of 365 days to 10% (by volume) hydrochloric acid solution. The specimens were dried in the drying oven at 105 °C (24 h) before the test for stronger absorption of the solution. The solution was refreshed at the beginning of each month to maintain concentration and pH during the test period to simulate a continuous acid attack. Due to this situation, it was provided to examine the acid effect homogeneously. The amount of solution was adjusted to maintain a sample: solution ratio of 1:4. Samples were collected bimonthly and kept at room temperature. Flexural and compressive strengths and weight changes were recorded before and after the freeze-thaw and solution tests. A visual examination was performed after the acid test. Also, SEM analyses were conducted after the durability tests.

3. Results and discussion

3.1. Abrasion resistance

The abrasion test results are given in Fig. 1. Better results for weight loss were obtained with the addition of fibres because a durable layer formed [27]. When the results are examined, PA fibres provide better structural performance than PL fibres due to strong bonding within the geopolymeric matrix and high tensile strength properties [27]. When the results were examined after the abrasion test and before 500 °C, the average weight loss ranged from 0.31 g to 0.93 g. After heat treatment, the average weight loss of the sample ranged from 3.08 g to 3.88 g. Before treatment, according to the control sample, the average weight

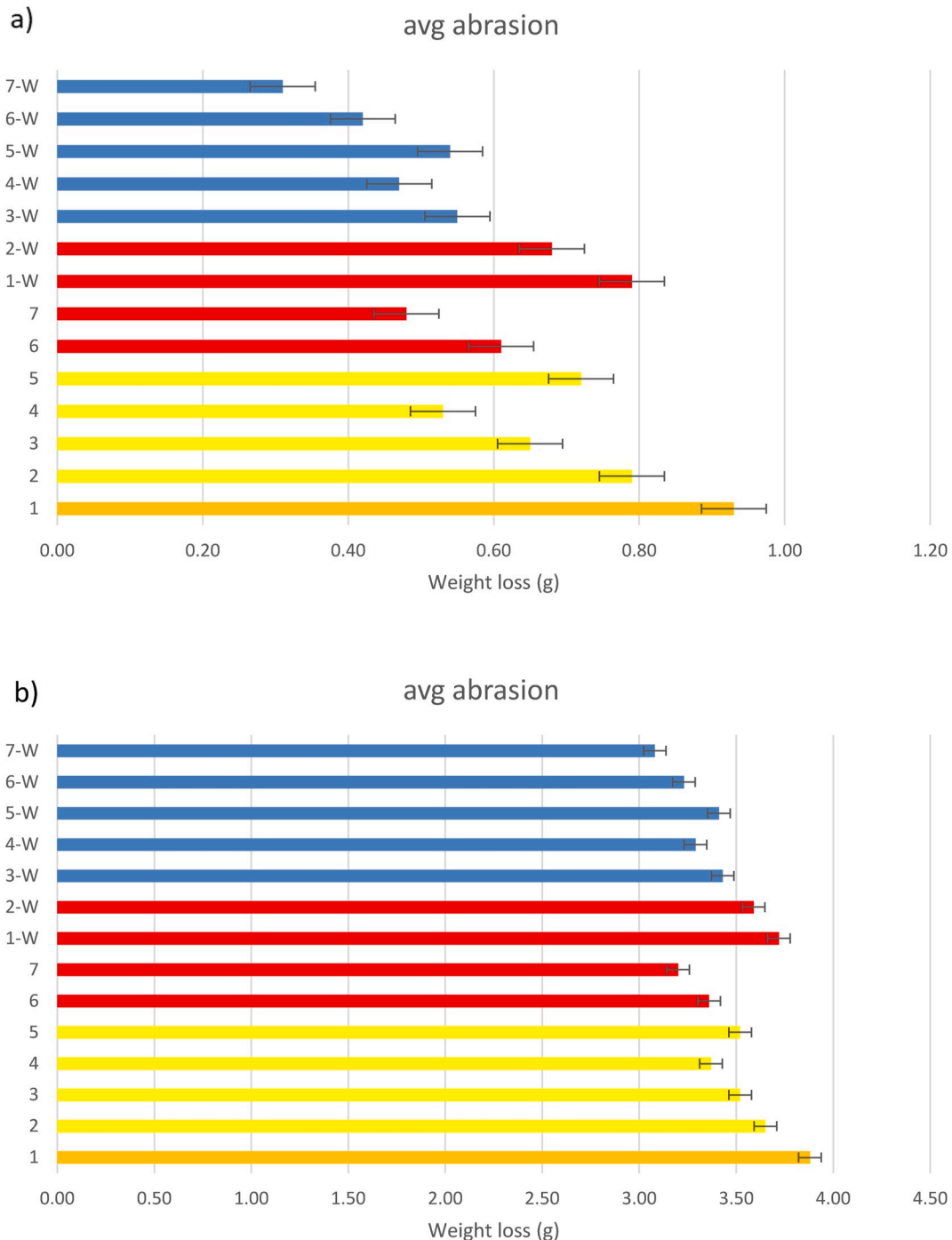


Fig. 1. Average weight loss due to abrasion: a) before 500 °C b) after 500 °C.

loss improved between 15.05 and 66.67%. After treatment, according to the control sample, average weight loss was improved between 4.12 and 20.62%.

These results are consistent with the literature [26,29]. The wet-dry effect strengthens geopolymerisation and forms a more stable structure [25], and the stable structure is maintained after heat treatment with minor weight loss. Kabay [30] conducted a study of abrasion resistance and fracture energy in concrete produced with basalt fibres. The fibres reduced abrasion between 2 and 18%.

3.2. Freeze-thaw test

3.2.1. Strength results

The engineering properties of the samples were observed after 300 freeze-thaw cycles (Figs. 2 and 3). Only minor losses were observed because the compact structure of the geopolymeric matrix is resistant to freeze-thaw effects due to good adhesion, and matrices also experience progress due to the cycles [20,21]. Its resistance is increased by the reaction products formed in the wet-dry curing process. Thus, the ability of wet-dry curing to maintain the integrity of the geopolymer was reflected in the durability tests.

While the freeze-thaw test had a positive effect over the short term, some scientists propose that the relative humidity of the experimental conditions improve certain concrete features such as strength and dynamic modulus [21,31,32], and it is thought that reaction products produced during the tests through activation of raw materials (slag, metakaolin, and fly ash) mitigate the effects of hard freezes. However, these findings do not evaluate the polymeric material's resistance to freeze-thaw events or the microstructural changes created by geopolymerisation or hydration generated by freeze-thaw cycles (especially in fly ash, which has low reaction kinetics). 300 cycles were used because a longer cycle was required to prevent this.

The addition of fibre to the polymeric material reduced losses in mechanical properties compared to other cases. In samples produced using fibres, similar behaviours were observed at the end of the cycles compared to behaviours before the test. Flexural strength results decreased more after 300 cycles than compressive strength results. When the samples were exposed to compressive loads, the cracks corresponded to the compressive load action, and the damage happened due to flexural stress at 90° to the compressive load. The resultant losses were similar to pre-test conditions.

The highest residual compressive strength result was 41.46 MPa in sample 6-W, while the lowest residual compressive strength was 33.48 MPa in sample 1. The highest residual flexural strength was 6.96 MPa in sample 7-W, while the lowest residual flexural strength was 4.23 MPa in sample 1. When the results were examined, it was seen that using 1.5% polyamide fibre reduced the compressive strength. Therefore, compressive strength results obtained with 1.5% polyamide fibre were lower than 0.5 and 1% polyamide fibres and close to the control sample. When the flexural strength results were examined, a continuous increase was observed with increasing fibre ratio. This is compatible with Celik et al. [27].

3.2.2. Weight loss results

Weight loss remained below 1% in all samples (Fig. 4). The highest weight loss was in sample 1 at 0.88% and the lowest weight loss was in sample 7-W at 0.24%. The common condition observed when examining weight loss is that the freeze-thaw effect is limited. This situation differs according to the number of low cycles and high cycles. Microcracks form during low cycles. However, the humidity of the experimental environment ensured that the voids formed in the microcracks were filled in low cycles. Aygörmüş et al. [33] showed that the geopolymer matrix in a low cycle is resistant to freeze-thaw effects due to its compact structure. The geopolymer matrix has a good degree of adhesion. Due to these situations, a process of progress in geopolymerisation has occurred. This condition can also be considered as a continuation of the curing condition. Due to conditions created by wet and humid conditions, increases in weight values can be seen in low cycles. In the following cycles, this situation began to change and the effects of deterioration began to increase, microcracks increased and desquamation was seen on the surfaces of the samples. Due to these conditions, a reduction in weight was observed. But the compact nature of the geopolymer has limited this reduction. These results are compatible with other studies [33,34]. Stable, homogeneous porous networks limit weight loss. Also, the reaction products created in the wet-dry curing process reduced weight loss [25].

3.2.3. SEM analyses

Microstructural changes occurred in sample 7-W after 300 freeze-thaw cycles (Fig. 5). The presence of microcracks was determined from the SEM images. It was found that the microcracks generally passed through the interface between the gel and the metakaolin spheres,

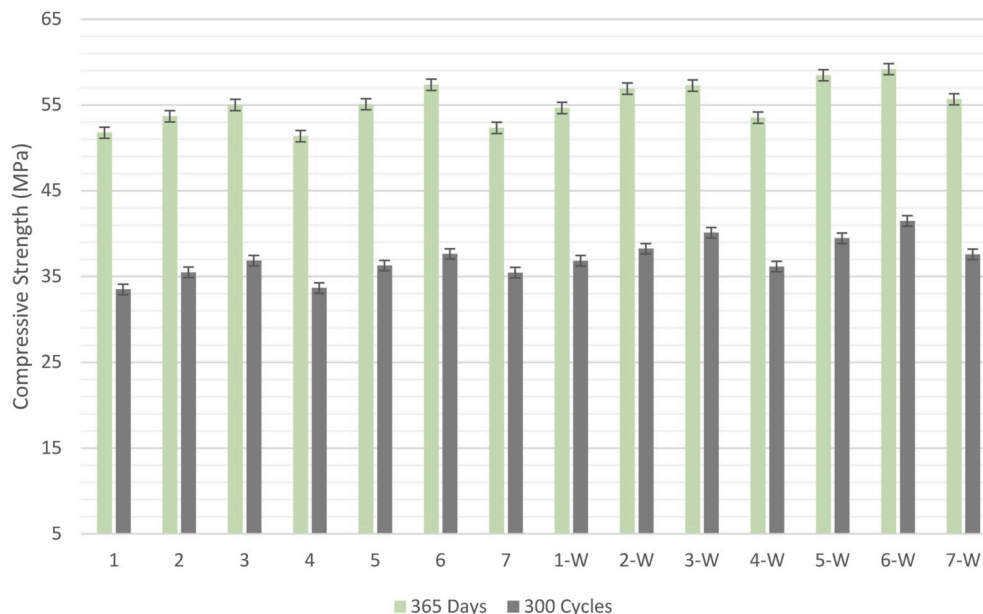


Fig. 2. Residual compressive strengths after the freeze-thaw test.

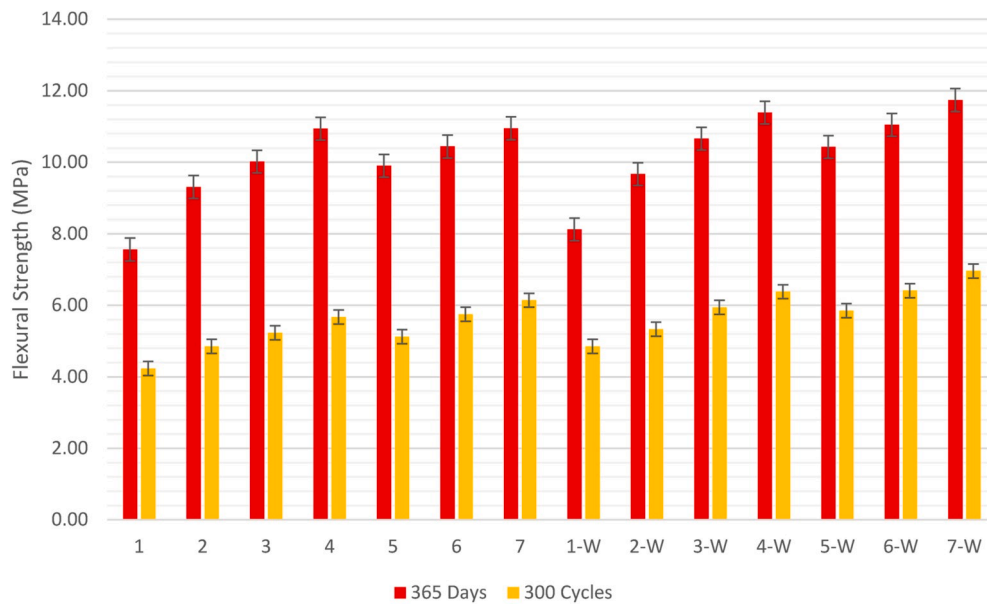


Fig. 3. Residual flexural strengths after the freeze-thaw test.

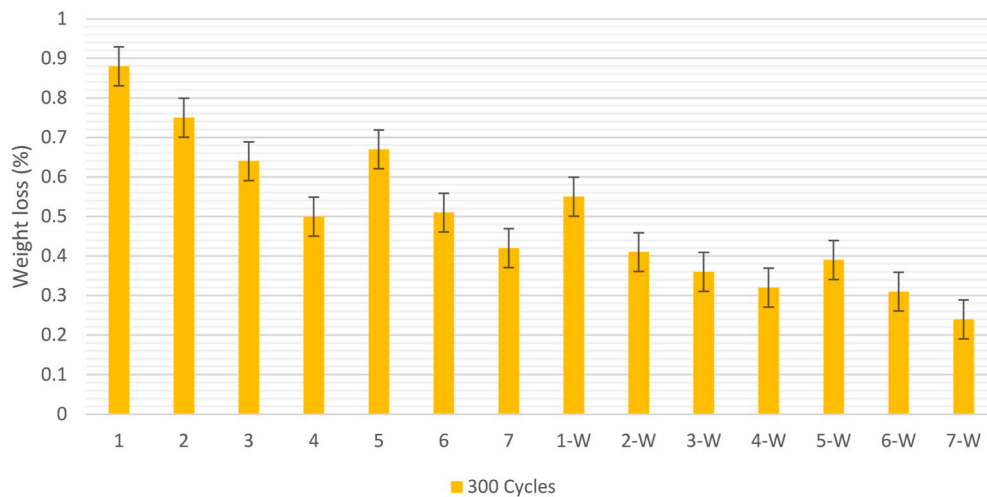


Fig. 4. Weight-loss rates after the freeze-thaw test.

making the interface defective. These microcracks connected to form a network. This caused minor damage to the structure's integrity and the decrease in mechanical properties. However, the microcracks formed slowly and were fixed in the geopolymer sample. Thus, high resistance was observed in geopolymer samples after 300 cycles. The obtained results are adaptable to the literature [35–38].

3.3. Hydrochloric acid effect

3.3.1. Strength results

The results of the samples experienced a drop over time (Figs. 6 and 7). The residual mechanical properties of the samples depend on the initial mechanical properties as well as the ratio of the total cross-sectional area damaged. In terms of compressive strength, the highest result was obtained in sample 6-W with 47.55 MPa after 2 months, 33.22 MPa after 4 months, and 23.56 MPa after 6 months. But the compressive strength results of sample 4 was 40.67 MPa after 2 months, 27.61 MPa after 4 months, and 17.7 MPa after 6 months. In terms of flexural strength, the highest result was obtained in sample 7-W with 8.06 MPa after 2 months, 6.73 MPa after 4 months, and 5.29 MPa after 6

months. In terms of flexural strength, the lowest result was obtained in sample 1 with 5.78 MPa after 2 months, 4.56 MPa after 4 months, and 3.12 MPa after 6 months. The addition of fibre was found to have a positive effect on flexural strength. Polyamide fibre improved the compressive strength when 1% was used. Also, wet-dry curing increased the results under acid solution compared to heat curing.

Loss of strength was observed when the geopolymer samples were exposed to hydrochloric acid because the acid disintegrates the oxy-aluminum bridge (Si-O-Al) in the gel. Si-O-Al bonds combine in the gel to strengthen the bonds forming the matrix [39–41]. Depolymerisation in geopolymeric products and zeolites are other factors that can cause loss of strength. The reason why geopolymer products are not easily degraded under the influence of acid is based on the strong alumina-silicate bonds that form during geopolymerisation. In this respect, the geopolymer products are more effective against chemical attacks than Portland cement. Likewise, the stability maintained in the geopolymer materials results from crystalline phase formation of the aluminosilicate. In acidic environments, more crystalline phases are formed and stability is increased [42]. Another factor that plays an active role against acid in geopolymer composite materials is the

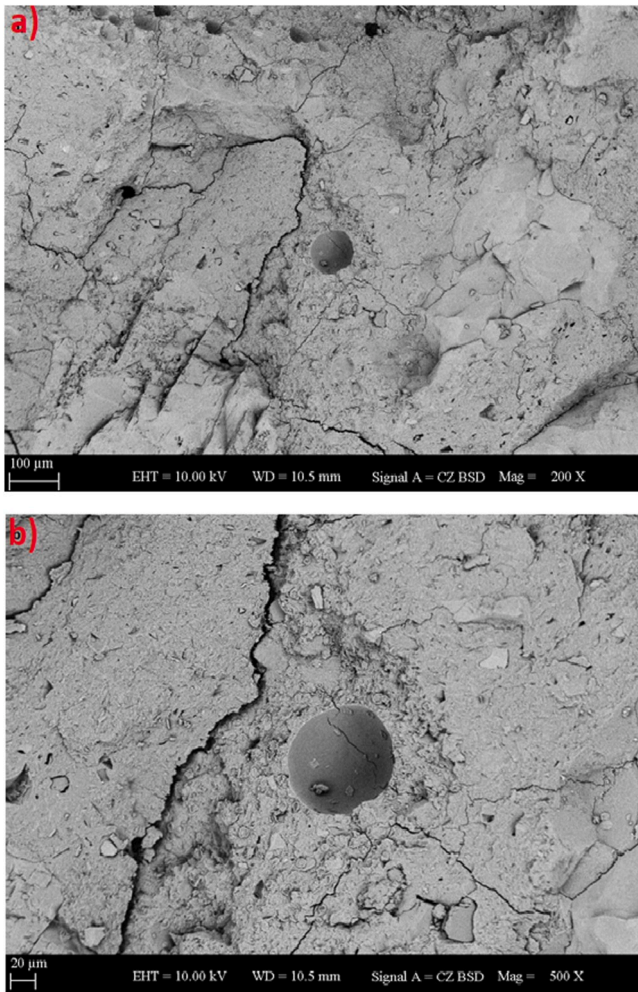


Fig. 5. SEM micrographs for sample 7-W after the freeze-thaw test.

morphology of the sample. The pore size becomes important here. The lower the porosity, the higher the total pore surface area and the threshold pore diameter of the composite, limiting acid diffusion in the microstructure as compared to Portland cement.

The layer formed by this effect reduces the rate of deterioration [43], and the compact structure of the sample prevents acid from entering. The compact structure is strengthened as the voids ratio decreases due to the wet-dry effect. By intensifying the interfacial transition region, the aggregate-paste interface, which has less permeability and porosity, formed and the passage of aggressive fluids into the structure was restricted. Less diffusion of harmful ions is achieved at an appropriate water:solid ratio and a compact geopolymer microstructure forms [44, 45]. Another factor that provides superiority to the acid effect in geopolymer samples compared to Portland cement is its low Ca content. The low calcium and lime content provides superior performance in geopolymer samples. In Portland cement, harmful ions increase the formation of gypsum and ettringite by entering the matrix under the influence of acid. Calcium oxide triggers this condition, and strength loss occurs due to expansion, cracking, and spreading [39,46].

3.3.2. Weight loss results

The weight loss results after 2, 4, and 6 months are given in Fig. 8. The main reason for the loss under acid effect is the increase in voids with the formation of microcracks [47]. As time elapsed, the results decreased. While the samples were under the action of acid, the hydrated products of calcium reacted with the acid to create calcium sulfates and calcium sulfoaluminates. Under this effect, a weak layer that was soluble in the solution formed, resulting in weight loss [48,49]. The stability of the structure due to its compact structure played a role in limiting the weight loss [50]. Under the effect of 10% acid, Song et al. [51] found weight loss to be 3% for the geopolymer sample after 56 days, while 41% weight loss for ordinary Portland Cement was detected after 28 days. This result supports the stable and resistant structure observed in these studies. Also, Na-rich gel neutralises acid through acid-base reactions and improves performance.

In terms of weight loss, the lowest results were obtained in sample 7-W, while the highest results were obtained in sample 1. Sample 7-W had a weight loss of 0.29% after 2 months, 0.93% after 4 months, and 1.44% after 6 months. Sample 1 had a weight loss of 0.96% after 2 months,

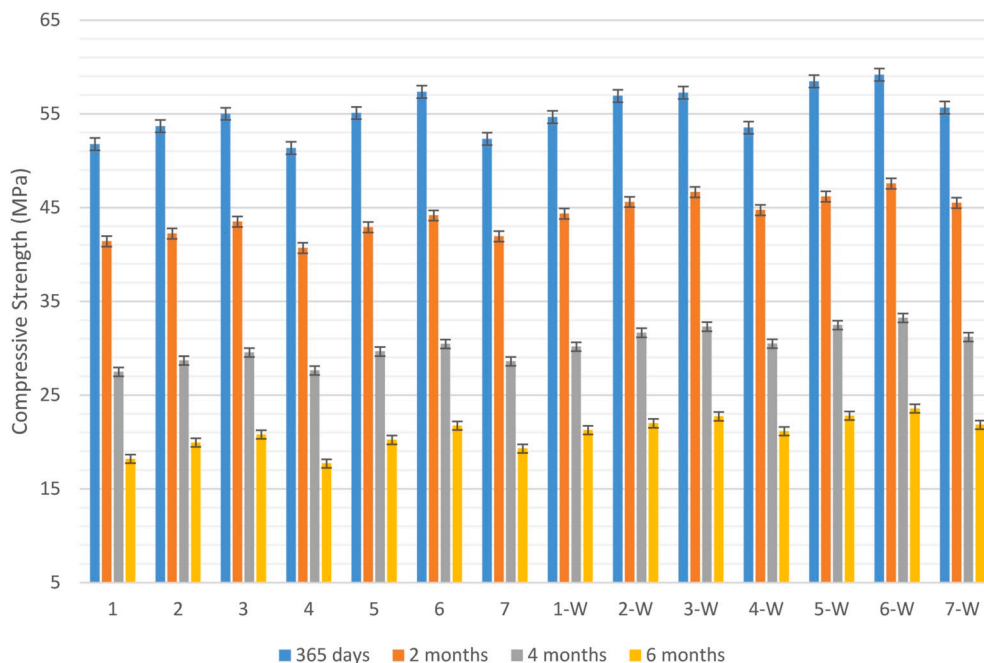


Fig. 6. Residual compressive strengths after hydrochloric acid effect.

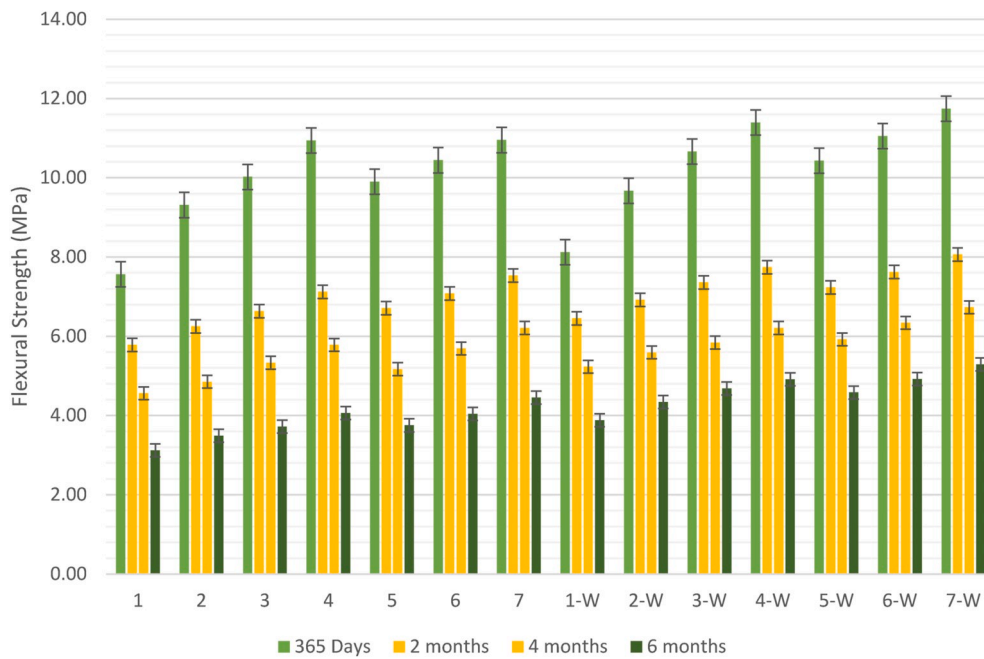


Fig. 7. Residual flexural strengths after hydrochloric acid effect.

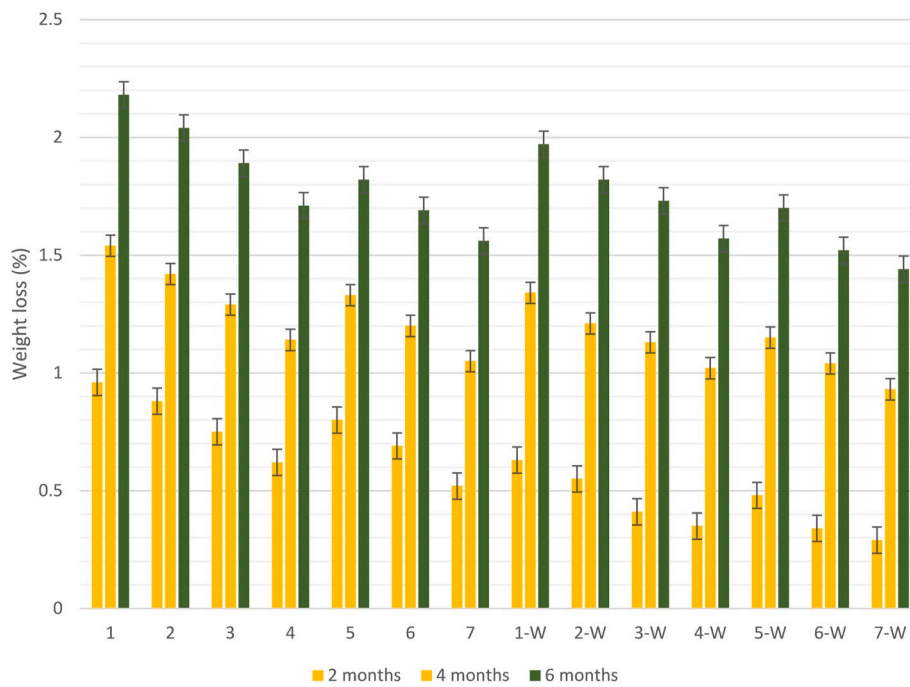


Fig. 8. Weight-loss rates after hydrochloric acid effect.

1.54% after 4 months, and 2.18% after 6 months. So the fibre effect had a positive effect on weight loss.

3.3.3. Visual inspection and SEM analyses

The visual appearance of the outer surface of geopolymer samples after 6 months is shown in Fig. 9. Deformation was observed on the surfaces of some samples following the acid test. The degree of damage corresponded to the high concentration of the solution. After the test, some softening was observed on the surface, but it was not easily scratched. Some erosion was observed at the corners and near the edges. Tiny white flaky material was observed on the surface. The formation of gypsum crystals which formed high volumetric expansions that

disintegrated the surface layers increased deterioration. However, the overall structure and stability of the samples were preserved. In other words, the physical changes that occurred were minimal [52].

Microstructural changes in sample 7-W after 6 months under the effect of hydrochloric acid are shown in Fig. 10. The dense microstructure of geopolymer samples is porous due to microcracks caused by the degradation of Si-O-Al bonds by the acid [43]. Bubbles and cracks are seen in the micrographs due to reactions with the acid. However, some needle or lather particles formed in the broken surfaces of pores. The cracks are caused by the shrinkage of the corrosion-resistant gel layer, thereby facilitating the passage of hydrochloric acid into the microscopic interstices. Subsequent loss of the needle-shaped particles can be



Fig. 9. Visual inspection after hydrochloric acid effect over 6 months.

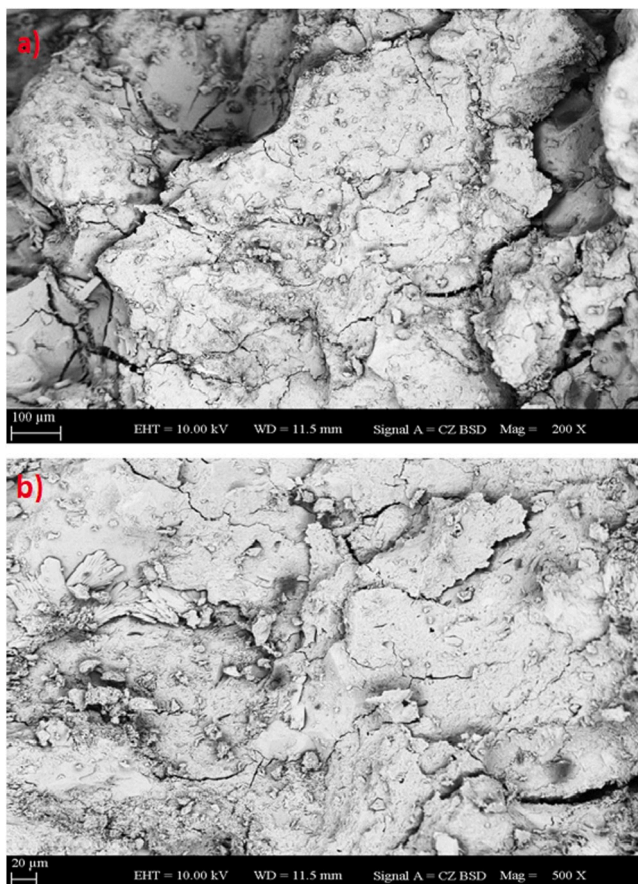


Fig. 10. SEM micrographs of sample 7-W after hydrochloric acid effect over 6 months.

explained by dissolution in the acidic solution. New crystals were observed in the microstructures of the samples that belong to the crystal structure of gypsum [53,54]. Gypsum formed the whitish layer in the sample after the acid effect, and the degree of degradation around the sample is attributed to the formation of a different gypsum phase; thus, the amount of calcium present in the geopolymer structure increased gypsum formation. Gypsum crystals accelerated crack formation and loss of strength loss. The behaviour of the acid solution in the geopolymer sample was related to pore structure and connectivity. It was found that permeability affects durability and acid resistance increased with the presence of Na-rich gel. Pimraksa et al. [55] showed that the degree of polymerisation was affected by the Si:Al ratio, and the low reduction in Si:Al in the samples was due to the effect of acid.

4. Conclusions

The results of this study can be summarised as follows:

- Since polyamide fibres have higher tensile strength than polyolefin fibres, they produced higher results in mechanical properties. The highest results were obtained in polyamide fibres of 1% by volume in all samples, but if the volume was increased, the fibres negatively affected the results. The wet-dry curing method was used in addition to heat curing. Particles that did not react with the heat curing did continue geopolymerisation, resulting in higher results with the wet-drying curing.
- A low rate of loss of engineering properties was observed after 300 freeze-thaw cycles. The major reason is that the compact structure of the matrix is resistant to the effects of freezing and thawing and maintains a good degree of adhesion; the matrix also experiences progress due to freeze-thaw cycles. Thus, high resistance to freeze-thaw cycles was observed in the geopolymer specimens after 300 cycles.
- Loss of strength was observed when the samples were exposed to hydrochloric acid. The acid disintegrated the oxy-aluminum bridge (Si-O-Al) causing strength loss in the geopolymer gel. Other factors active against acid are the morphology and low Ca content of geopolymer samples. In Portland cement, harmful ions increase the formation of gypsum and ettringite by entering the matrix under the influence of the acid. Calcium oxide triggers this condition. The results of all samples experienced a drop over time. The dense microstructure of geopolymer samples is porous due to microcracking caused by the degradation of Si-O-Al bonds through exposure to acid.
- All durability studies showed that despite the decrease in mechanical properties after 365 days post-production, the compact structure of the geopolymer composite withstood the durability tests performed due to the effect of temperature on curing conditions. With the experiments conducted this way, unlike laboratory conditions, knowledge of situations that may occur in the field was obtained.

CRedit authorship contribution statement

Yurdakul Aygörmez: Conceptualization, Funding acquisition, Formal analysis, Data curation, Writing - original draft, Writing - review & editing, Validation. **Orhan Canpolat:** Conceptualization, Funding acquisition, Formal analysis, Data curation, Writing - original draft, Writing - review & editing, Validation. **Mukhallad M. Al-mashhadani:** Conceptualization, Funding acquisition, Formal analysis, Data curation, Writing - original draft, Writing - review & editing, Validation.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.job.2020.101453>.

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