

# Effect of ceramic waste powder content and sodium hydroxide molarity on the residual mechanical strength of alkali-activated mortars

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## HIGHLIGHTS

- Ceramic waste powder can be used for alkali-activated material production.
- The highest performance was achieved by adding ceramic waste powder at 15%.
- The performance of 16 M of sodium hydroxide is higher than 8 M of sodium hydroxide.
- The alkali-activated materials resisted the durability tests.

## ARTICLE INFO

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## ABSTRACT

In the study, the effects of various ceramic waste powder (CWP) additions (5, 10, and 15%) and sodium hydroxide (SH) molarity (8 M and 16 M) on the residual mechanical properties of different ambient-cured alkali-activated mortar (AAM) samples were investigated under the elevated temperatures (300, 600, and 900 °C) and freeze-thaw (100 cycles) attacks. The fresh (flowability, initial and final setting time, water absorption, and void ratio) and hardened (compressive and flexural strength, ultrasonic pulse velocity, and weight loss) state performances were evaluated. Also, XRD and SEM analyses were carried out. The findings pointed out that flowability enhanced up to 15% of CWP replacements. The initial and final setting time was reduced with higher molarity, while improved with further CWP incorporations. The water absorption and void ratio decreased with higher CWP incorporations and SH molarity. Moreover, mechanical strengths increased with time, which were found to be more with a higher CWP replacement ratio and SH molarity. After elevated temperature, the average compressive strength losses were about 50% at 600 °C and 85% at 900 °C, while average flexural strength losses were about 70% at 600 °C and 85% at 900 °C. Meanwhile, similar residual compressive strengths were obtained at elevated temperatures, irrespective of the SH molarity and CWP incorporations. However, both CWP incorporation and high SH molarity slightly enhanced the residual flexural strengths. After freeze-thaw attacks, an average of 23% compressive strength loss and 29% flexural strength loss was obtained, and almost similar mechanical strength losses were observed with varying SH molarity and CWP replacement ratio.

## 1. Introduction

Ordinary Portland Cement (OPC) is a popular binder material that has been utilized widely in construction applications. During OPC production, a high amount of energy is required for the calcination process. However, energy prices in the world increases dramatically after the Covid-19 related pandemics and the Russian-Ukraine war. In the upcoming years, energy demand is going to increase due to the growing population and technological developments such as electric-powered

vehicles, smart transportation systems, cryptocurrency mining, and blockchain application technology. Unfortunately, the cement industry is responsible for CO<sub>2</sub> emissions (1.5 billion tons annually) and about 36% of the overall consumption of energy [1]. One way to prevent the possible upcoming energy wars should be restrictions on OPC production and its utilization in the construction sector. Also, OPC is regarded as the basic reason for environmental pollution due to the requirement of massive exploitations of raw materials, consumption of fossil fuels, and high carbon dioxide emissions during the production stage [2].

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**Table 1**

Chemical composition and physical properties of FA, GGBFS, and DSF 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 | Loss on Ignition | Specific Gravity | Blaine Fineness(m <sup>2</sup> /kg) |
|-----------|-------|------------------|--------------------------------|--------------------------------|------|-----------------|------------------|-------------------|------------------|------------------|-------------------------------------|
| FA (%)    | 2.10  | 54.76            | 25.26                          | 6.28                           | 2.08 | 0.20            | 4.04             | 0.38              | 3.30             | 2.13             | 387                                 |
| GGBFS (%) | 37.25 | 38.37            | 11.89                          | 1.05                           | 8.13 | 0.38            | 1.28             | 0.28              | –                | 2.93             | 432                                 |
| CWP (%)   | 4.65  | 65.44            | 15.45                          | 7.11                           | 1.28 | 0.06            | 2.15             | 0.80              | 3.06             | 2.62             | 545                                 |

During an organization of the World Cement Association, ICC encouraged both governments and industries to decrease OPC emissions [3]. Therefore, researchers spend a great effort to find out sustainable constructional materials instead of OPC.

Recently, alkali-activated materials have emerged as cementless construction materials and can be utilized in structural applications [4–7]. Ongoing studies revealed that alkali-activated materials (AAM) have superior flexural and tensile strengths, compressive strength, bond strength, impact strength [8–13], seawater, sulfate, and sulfuric acid resistances [14–17] for structural utilization. Also, these investigations showed that alkali-activated materials gain strength without heat curing, which is beneficial for on-site structural applications as well as prefabricated constructional elements. The production of alkali-activated materials doesn't only significantly decrease carbon dioxide emissions but also decreases the cost and is environmentally sustainable, of which their raw materials are generally composed of by-product materials such as ground granulated blast furnace slag (GGBFS) and F-type fly ash (FA) [18]. The GGBFS material includes calcium, silica, and alumina contents, and FA contains silica and alumina depending on the type of burnt coal. Their chemical compositions are beneficial for alkali activation, and their disposal in landfills is not in line with sustainable development goals [19]. Thus, these by-product waste materials are required to be used in construction sectors with higher amounts to decrease environmental degradation for sustainable design targets. Due to the low calcium oxide content, FA material needs elevated temperature content, which is impossible in-situ applications. To eliminate these drawbacks, 50% calcium-rich GGBFS material is included in the mixes of the study to form N-(C)-A-S-H types of geopolymeric product formation, which is beneficial for structural utilization at ambient conditions.

Besides being non-recyclable, ceramic wastes are bulky and create ecological problems for landfill disposal. Thus, for the protection of natural resources and environmental degradation, it is essential to find out novel products incorporating ceramic wastes for the utilization of them in constructional applications [20]. Recently, ceramic waste powder (CWP) addition in alkali-activated materials takes great attention, since its utilization solves the ceramic industry waste problem, and also more sustainable alkali-activated materials were obtained by utilizing higher ceramic waste powder replacement ratios [21]. CWP material is rich in alumina and silica, enabling additional pozzolanic reactivity [22–24]; thus, it can be regarded as a sustainable base material in alkali-activated concrete production. However, the majority of the literature investigations have mainly concentrated on utilizing CWP material as a partial replacement for natural aggregates due to the low availability of natural aggregates [25]. There are few investigations available for the cement replacement of CWP material. In a study, Vejmelková et al. replaced OPC with CWP material with different replacement ratios (0–60% by mass), and they reported that after 20% CWP replacements, compressive strength reduced significantly and the produced concrete lost its high-performance character [26]. In another investigation, two various CWP materials were utilized as OPC replacements, and it was concluded that both CWP materials have pozzolanic activity, enhancing concrete mechanical strength [27]. Furthermore, it was found that the CWP replacement level of 10%–20% by weight of OPC was suitable for optimizing the workability retention and strength of the mixture [28]. Therefore, CWP replacement ratios were selected as 5%, 10%, and 15% in this study.

In the literature, Shoaie et al. [21] pointed out that the flexural

strength and compressive strength of CWP-based alkali-activated materials were strongly influenced by the curing temperature and alkali solution/binder ratio. They stated that an alkali solution/binder ratio of 0.6 and a curing temperature of 90 °C yielded the optimum performance. Hwang et al. [29] stated that CWP-based alkali-activated pastes activated by a combination of sodium silicate and sodium hydroxide cured in an ambient environment did not set after 24 h of casting. Sun et al. [30] confirmed that the compressive strength of CWP-based alkali-activated materials depends on the initial reacting system, and alkali activators are important for the alkali activation process. The GGBFS-based alkali-activated materials showed superior mechanical strength performance due to the high CaO content [4,8], and high CaO content can be an initial reacting system. However, there are limitations to the utilization of GGBFS-based alkali-activated materials, such as fast initial and final setting time, leading to problems for structural utilization in ready-mixed and pumped concretes [31]. On the other hand, FA extends the setting time; however, elevated temperature curing is required for the FA-based alkali-activated materials, hindering structural utilization alone [8]. Therefore, ternary mixes that GGBFS blended with FA can be the best option for the structural utilization of AAMs. Huseien et al. [23] investigated the effects of higher CWP replacements on the elevated temperature resistance for slag/fly ash-based alkali-activated materials. They pointed out that higher CWP replacements enhanced the elevated temperature resistance of alkali-activated materials up to 900 °C.

Although there are studies investigating the CWP replacements in OPC concrete, there is very limited investigations have been carried out to research the mechanical performance and durability of ceramic waste powder (CWP) incorporated alkali-activated materials at ambient curing conditions. The literature findings revealed that the influence of CWP on mechanical strength and durability was variable depending on the mixture and CWP replacement ratios. Also, the elevated temperature resistance and freeze-thaw resistance of different CWP-incorporated alkali-activated materials with varying SH molarities (8 M and 16 M) have not been studied yet. Therefore, in the scope of the research, the influence of CWP contents (5%, 10%, and 15%) with varying SH molarities (8 M and 16 M) on the residual mechanical strength of alkali-activated materials was comprehensively investigated. For this purpose, flexural strength, compressive strength, weight loss, and ultrasonic pulse velocity tests were executed after elevated temperature and freeze-thaw attacks.

## 2. Experimental study

### 2.1. Materials and mix design

For the production of alkali-activated mortar (AAM) specimens, low-calcium fly ash (F-type) and ground granulated blast furnace slag (GGBFS) were used as binder materials. The ceramic waste powder (CWP) was replaced by a mass of F-type fly ash with 5%, 10%, and 15%, while GGBFS content was kept constant at 50% for all mixture designs. The natural sand and crushed limestone with a grain size lower than 4 mm were used as fine aggregates. A combination of sodium silicate (SS) and sodium hydroxide (SH) was used as an alkali activator, and SH molarity was varied to 8 M and 16 M. The SS includes 14.7% sodium oxide, 29.7% silicon dioxide, and 55.5% water by mass. For the workability enhancement, a polycarboxylate ether-based superplasticizer was used in the AAM mixes. The mixes without ceramic waste powder

**Table 2**

Mix ingredients of alkali-activated mortars (g).

| Materials | Control-8M | Control-16 M | CWP5-8M | CWP5-16 M | CWP10-8 M | CWP10-16 M | CWP15-8 M | CWP15- 16 M |
|-----------|------------|--------------|---------|-----------|-----------|------------|-----------|-------------|
| SS        | 145        | 145          | 145     | 145       | 145       | 145        | 145       | 145         |
| SH        | 55         | 55           | 55      | 55        | 55        | 55         | 55        | 55          |
| FA        | 200        | 200          | 180     | 180       | 160       | 160        | 140       | 140         |
| GGBFS     | 200        | 200          | 200     | 200       | 200       | 200        | 200       | 200         |
| CWP       | 0          | 0            | 20      | 20        | 40        | 40         | 60        | 60          |
| Sand      | 900        | 900          | 900     | 900       | 900       | 900        | 900       | 900         |
| SP        | 15         | 15           | 15      | 15        | 15        | 15         | 15        | 15          |

**Table 3**

Fresh state test results of ceramic waste powder incorporated AAM mixes.

| Materials             | Control-8M | Control-16 M | CWP5-8M | CWP5-16 M | CWP10-8 M | CWP10-16 M | CWP15-8 M | CWP15- 16 M |
|-----------------------|------------|--------------|---------|-----------|-----------|------------|-----------|-------------|
| Flow-D1 (cm)          | 13.50      | 14.00        | 14.50   | 14.35     | 15.15     | 13.85      | 15.50     | 14.15       |
| Flow-D2 (cm)          | 13.70      | 14.15        | 14.35   | 14.05     | 15.25     | 13.65      | 15.65     | 14.25       |
| Flow-D3 (cm)          | 13.45      | 14.20        | 14.15   | 14.15     | 15.45     | 13.45      | 15.95     | 14.30       |
| Flow-D4 (cm)          | 13.75      | 14.15        | 14.25   | 14.25     | 15.50     | 13.55      | 15.45     | 14.35       |
| AVERAGE (cm)          | 13.60      | 14.13        | 14.31   | 14.20     | 15.33     | 13.63      | 15.63     | 14.26       |
| Initial Setting (min) | 30         | 28           | 43      | 39        | 56        | 34         | 67        | 45          |
| Final Setting (min)   | 75         | 70           | 88      | 72        | 98        | 78         | 107       | 92          |

**Table 4**

Physical properties of ceramic waste powder incorporated AAM samples.

| Specimens    | Void ratio (%) | Weight per unit of volume (g/cm <sup>3</sup> ) | Water absorption percent by weight (%) |
|--------------|----------------|------------------------------------------------|----------------------------------------|
| Control-8M   | 21.87          | 2.31                                           | 9.48                                   |
| CWP5-8M      | 21.02          | 2.35                                           | 8.76                                   |
| CWP10-8 M    | 20.23          | 2.38                                           | 8.47                                   |
| CWP15-8 M    | 19.44          | 2.44                                           | 8.02                                   |
| Control-16 M | 19.67          | 2.43                                           | 8.15                                   |
| CWP5-16 M    | 18.73          | 2.48                                           | 7.56                                   |
| CWP10-16 M   | 18.22          | 2.50                                           | 7.31                                   |
| CWP15-16 M   | 17.68          | 2.52                                           | 6.98                                   |

are called control specimens, and control-8M and control-16 M show the control specimens having 8 M and 16 M SH alkali activators. For the ceramic waste powder incorporated mixes, the specimens are called CWP5, CWP10, and CWP15, implying the 5%, 10%, and 15% ceramic waste powder incorporated AAM mixes, respectively. Table 1 presents the physical properties and chemical compositions of ground granulated blast furnace slag (GGBFS), ceramic waste powder (CWP), and F-type fly ash (FA) materials. It should be mentioned that CWP material is replaced only by FA, and GGBFS content is kept constant for all mixes.

## 2.2. Sample preparations

Table 2 illustrates the mix contents of the different AAM mixes, which were chosen after the preliminary experiments. During manufacturing, FA, GGBFS, and CWP (if available) binders were mixed in a container by the drill. After, a combination of SS/SH solution and SP was added and mixed for 2 min. Later, sand was included in the mixture and mixed for 3 min. After the completion of casting and vibration procedures, AAM samples were sealed in plastic bags to prevent the alkali activator solution evaporation. These AAM specimens were kept in an ambient environment and demolded 24 h later, then they were put in the ambient environment until the testing days. Due to the high calcium oxide content in GGBFS material, slag-incorporated AAM specimens can gain strength without oven-curing or water-curing [8]; therefore, AAM samples were cured in an ambient environment for the representative of structural utilization. The effects of various ceramic waste powder dosages on the mechanical and durability properties of

AAM specimens cured in an ambient environment were investigated in detail herein.

## 2.3. Test procedure

The initial setting time, final setting time, and flow spread of the AAM mixtures were found by fresh state tests. The cube samples having 50 mm sizes were produced for compressive strength tests by ASTM C 109 standard [32]. Also, density and water absorption values were calculated on AAM cube samples. The 40x40 × 160 mm AAM samples were used for the determination of ultrasonic pulse velocity and flexural strength by ASTM C 348 standard [33]. The high-temperature influence on the AAM samples was also studied after 56 days. The AAM samples were kept in an electric furnace in which the temperature level increased at a rate of 1 °C/min up to the desired temperature. After obtaining the maximum temperature, the AAM samples were left at the maximum temperature level for an additional 2 h. Later, the samples were left to cool down to room temperature. The application of the heating procedure is the standard fire time vs temperature, and heating-cooling cycles are close to the RILEM recommendation [34]. Meanwhile, after the 56 days of ambient curing periods, the AAM samples were subjected to 100 freeze-thaw cycles. For each cycle, the duration of both the freezing and thawing stages was 12 h. The temperature levels of the freeze-thaw cycles were in the range of −20 °C and +20 °C. The AAM specimens were subjected to freezing-thawing cycles and elevated temperatures (300 °C, 600 °C, and 900 °C), and the variations in flexural strengths, compressive strengths, weights, and ultrasonic pulse velocities were comprehensively evaluated. Also, XRD and SEM analyses were carried out for the microstructural changes.

## 3. Results and discussions

### 3.1. Fresh state tests

Table 3 shows the fresh-state test results of the AAM mixes. The findings pointed out that the flowability of the mixes reduced with an increase in molarity. The 8 M CWP incorporated AAM mixes showed higher flowability than the ones having 16 M AAM mixes, while control specimens having 16 M SH showed higher flowability than the control specimens having 8 M SH. The flowability enhanced from 13.60 cm to 14.13 cm for control AAM mixes, while it reduced from 14.31 cm to 14.20 cm for 5% CWP including mixes, from 15.33 cm to 13.63 cm for 10% CWP incorporated mixes, from 15.63 cm to 14.26 cm for 15% CWP

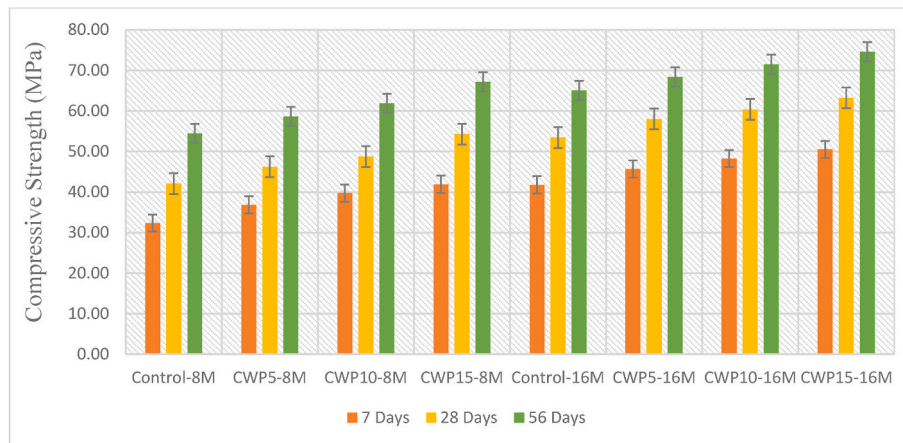


Fig. 1. Compressive strengths of the CWP incorporated AAM specimens.

Table 5

Flexural strength, compressive strength, and UPV results of the AAM specimens.

|              | Compressive strength (MPa) |        |        | Flexural strength (MPa) |        |        | UPV (m/s) |        |        |
|--------------|----------------------------|--------|--------|-------------------------|--------|--------|-----------|--------|--------|
|              | 7-day                      | 28-day | 56-day | 7-day                   | 28-day | 56-day | 7-day     | 28-day | 56-day |
| Control-8M   | 32.35                      | 42.11  | 54.48  | 5.85                    | 7.06   | 8.41   | 3132      | 3315   | 3523   |
| CWP5-8M      | 36.82                      | 46.22  | 58.62  | 6.48                    | 7.91   | 9.09   | 3225      | 3398   | 3604   |
| CWP10-8 M    | 39.76                      | 48.75  | 61.89  | 6.79                    | 8.37   | 9.41   | 3284      | 3485   | 3637   |
| CWP15-8 M    | 41.91                      | 54.27  | 67.15  | 7.21                    | 8.98   | 10.24  | 3361      | 3562   | 3692   |
| Control-16 M | 41.74                      | 53.43  | 65.06  | 7.08                    | 8.54   | 9.83   | 3327      | 3539   | 3663   |
| CWP5-16 M    | 45.67                      | 58.02  | 68.35  | 7.55                    | 9.23   | 10.79  | 3422      | 3613   | 3736   |
| CWP10-16 M   | 48.23                      | 60.37  | 71.48  | 7.93                    | 9.69   | 11.23  | 3475      | 3659   | 3782   |
| CWP15-16 M   | 50.49                      | 63.21  | 74.59  | 8.26                    | 10.04  | 11.87  | 3531      | 3702   | 3843   |

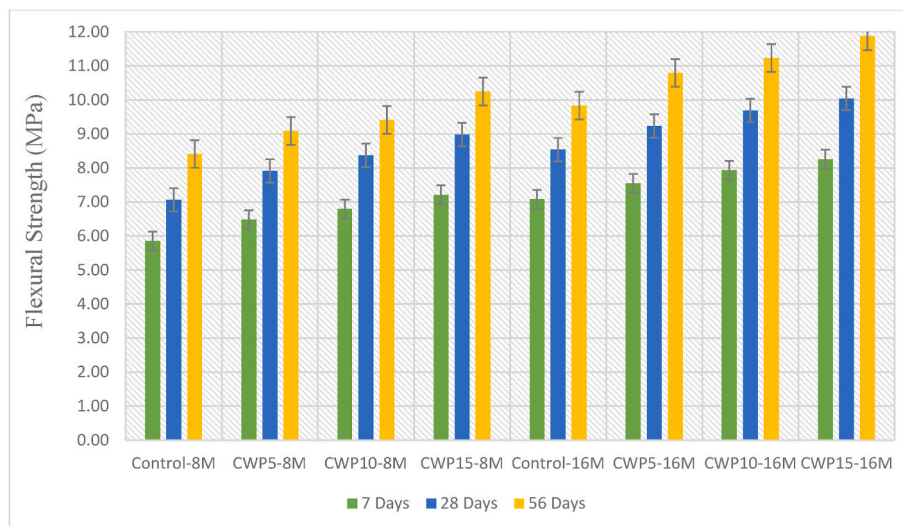


Fig. 2. Flexural strengths of the CWP incorporated AAM specimens.

containing mixes, with the increment in the molarity from 8 M to 16 M. Also, flowability improved with the CWP replacement ratio, and the highest flowability was obtained on the specimens having 15% CWP. In addition, the initial and final setting times of the AAM mixes were found, and the findings pointed out that both the initial setting time and final setting time were reduced with the increasing molarity. The specimens having 16 M SH showed lower initial and final setting values than the specimens having 8 M SH. Also, the initial and final setting time of the AAM mixes improved with higher CWP additions so that the mixes incorporating 15% CWP showed the highest initial and final setting

time. This can be attributed to lower calcium oxide content, resulting in a delay in the C-N-A-S-H type gel formation.

Table 4 shows the physical properties of ceramic waste powder (CWP) containing AAM specimens. The findings referred that the void ratio of the samples reduced with an increasing CWP replacement ratio and molarity. The AAM samples incorporating 15% CWP showed the lowest void ratio, and it was further reduced with higher molarity. This can be attributed to the fineness of CWP, enhancing the packing density and interfacial transition zone between matrix and aggregates. Also, water absorption of the CWP-containing AAM samples was reduced with



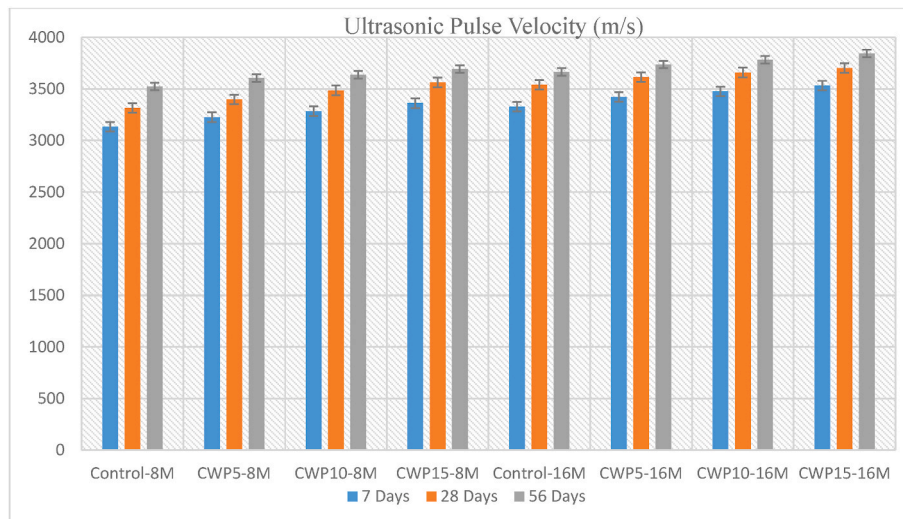


Fig. 3. Ultrasonic pulse velocity results of the CWP incorporated AAM specimens.

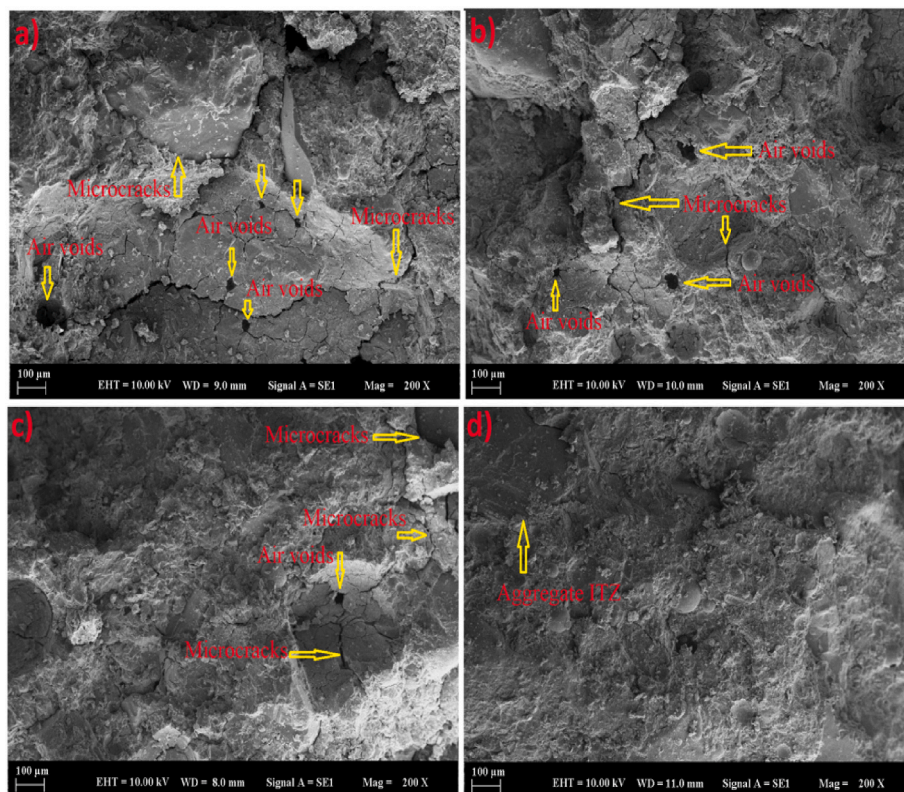


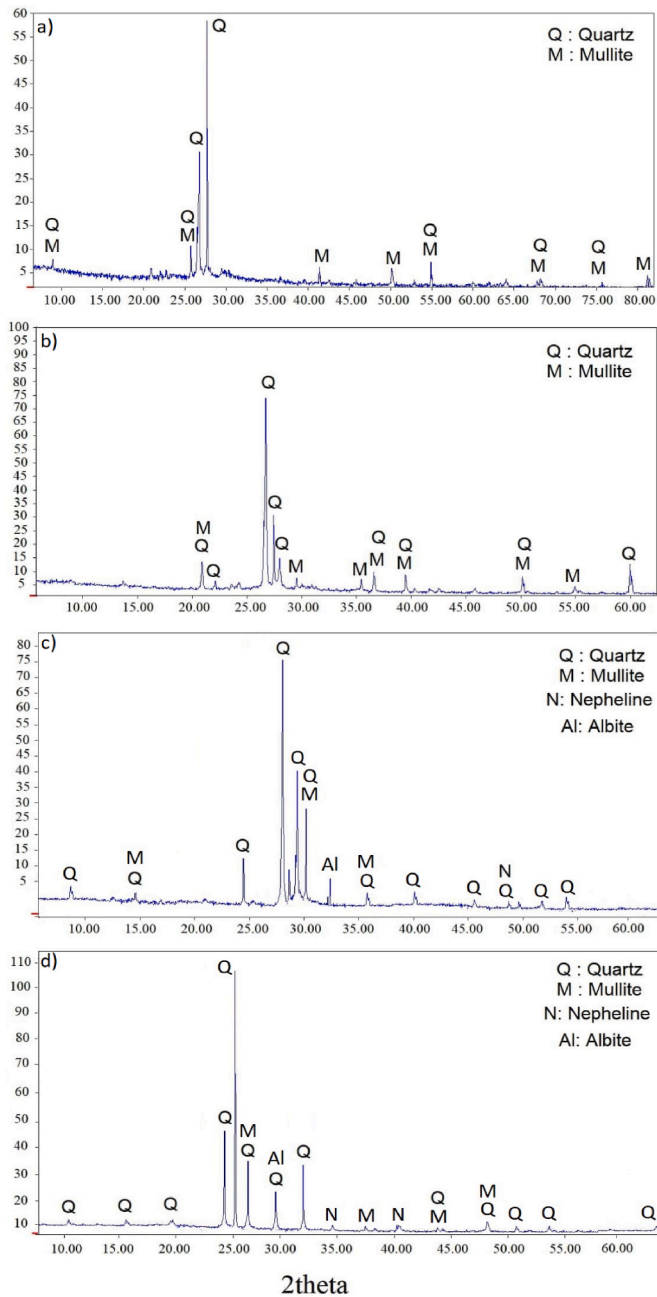
Fig. 4. SEM images of the samples: a) Control-8M, b) Control-16 M, c) CWP15-8 M, and d) CWP15-16 M.

an increasing CWP utilization ratio. In addition, the AAM samples containing 16 M SH showed a lower water uptake than the samples with 16 M SH. Considering physical properties, both 16 M SH molarity and 15% CWP replacements improved the physical properties of the AAM samples.

### 3.2. Strength and UPV test outcomes

Fig. 1 illustrates the compressive strength test results at the ages of 7 days, 28 days, and 56 days. The findings pointed out that ceramic waste powder (CWP) addition improved the compressive strength results, and the highest compressive strength improvement was obtained in the 15%

CWP replacement ratios. In addition to this, the increase in the SH molarity enhanced the compressive strength performance. The specimens having 16 M SH molarity exhibited the best compressive strength performance for all CWP replacement ratios, which can be attributed to the additional silica and alumina content from CWP material, enhancing C–N–A–S–H type gel products, making the microstructure denser. The high dissolution rates of  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  in geopolymer samples containing CWP were an important factor. However, the increase in  $\text{Na}^+$  production due to CWP and high molarity increased the formation of C–N–A–S–H type gels by acting as a charge balancer in  $[\text{AlO}_4]$ . Thus, both the increase in the Ca ratio thanks to the slag and the increase in the  $\text{Si}^{4+}$  and  $\text{Al}^{3+}$  ratio thanks to the CWP strengthened the compact structure of the



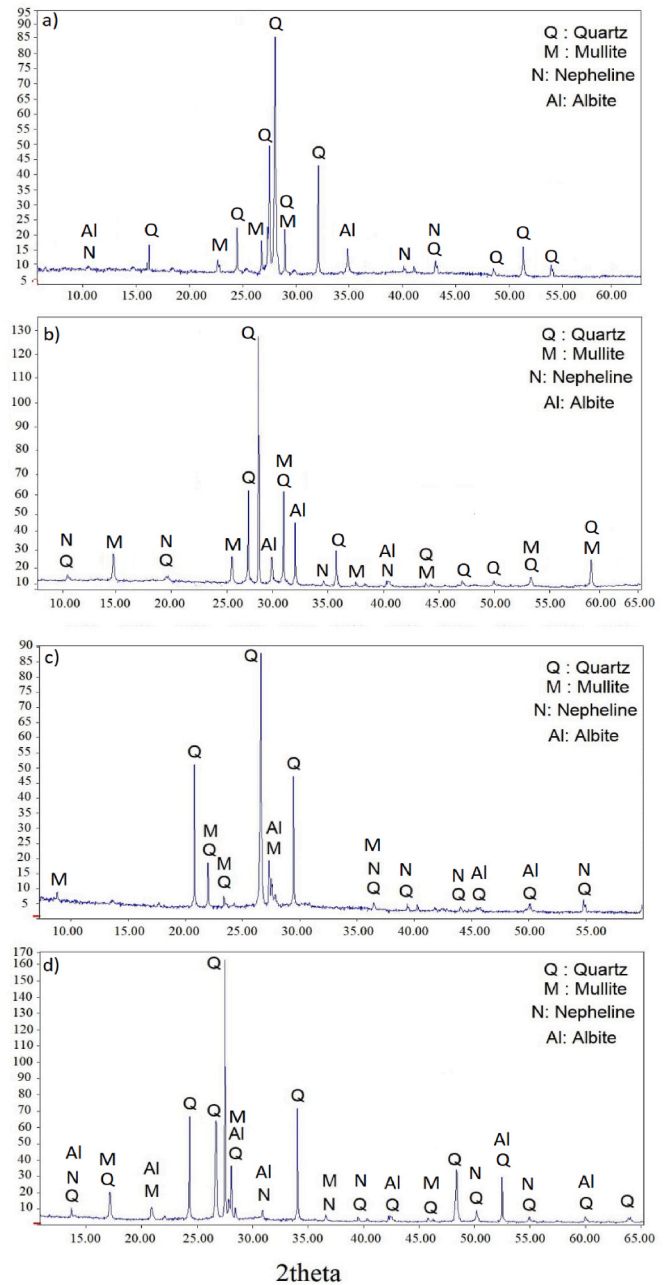
**Fig. 5.** XRD patterns of the samples: a) Control-8M, b) Control-16 M, c) CWP5-8M, and d) CWP5-16 M.

geopolymer by strengthening the C–N–A–S–H type gel formation with the specified Equation (1) and Equation (2).



A similar finding that the substitution of fly ash by ceramic waste powder up to 20% showed a slight enhancement in the early and later compressive strength was also reported [35].

The compressive strength of the AAM specimens improved with time due to the continuous alkali activation reactions. It should be noted that AAM specimens were left in an ambient environment and heat curing was not applied to the specimens considering possible applications of them for structural utilization. Table 5 summarizes the compressive strength development. The results revealed that compressive strength



**Fig. 6.** XRD patterns of the samples: a) CWP10-8 M, b) CWP10-16 M, c) CWP15-8 M, and d) CWP15-16 M.

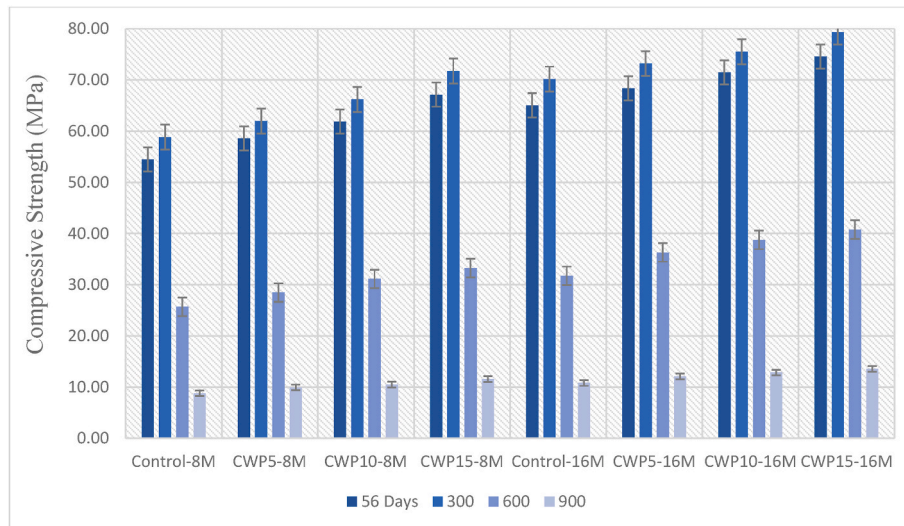
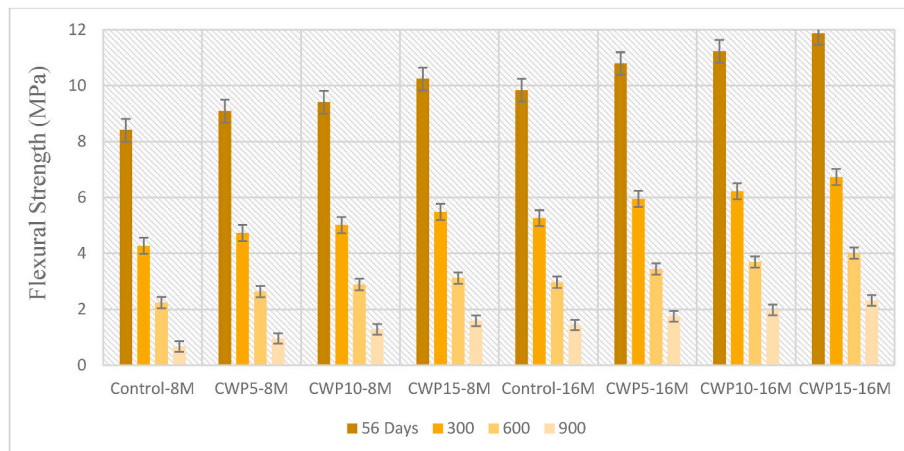
increment was observed for all specimens up to the 56-day. The average compressive strength increments were found to be about 27% from 7-day to 28-day and 23% from 28-day to 56-day. Also, the compressive strengths of the specimens were found in the range of 40 MPa–60 MPa, satisfying the allowable compressive strength limits used in constructional elements based on the regulations/standards. The CWP replacements from 0 to 15% increased the compressive strength values up to 10 MPa. In Fig. 1, the compressive strength enhancements can easily be seen with an increase in the CWP replacement ratio and SH molarity.

Fig. 2 shows the flexural strength results of the AAM samples at the ages of 7, 28, and 56 days. Based on the obtained results, the flexural strength of the specimens was improved with the CWP additions up to 15% replacement ratios. Also, the flexural strength of the specimens was enhanced with the increased SH molarity. The samples having 15% CWP and 16 M SH molarity performed superior flexural strength, while the

**Table 6**

Residual compressive strength, flexural strength, and UPV results (%) after elevated temperature.

|              | Compressive strength (%) |        |        | Flexural strength (%) |        |        | UPV (%) |        |        |
|--------------|--------------------------|--------|--------|-----------------------|--------|--------|---------|--------|--------|
|              | 300 °C                   | 600 °C | 900 °C | 300 °C                | 600 °C | 900 °C | 300 °C  | 600 °C | 900 °C |
| Control-8M   | 108.02                   | 47.12  | 16.12  | 50.77                 | 26.63  | 7.97   | 96.08   | 52.28  | 27.50  |
| CWP5-8M      | 105.71                   | 48.55  | 16.92  | 52.04                 | 28.93  | 10.56  | 95.53   | 53.75  | 28.41  |
| CWP10-8 M    | 107.01                   | 50.32  | 16.95  | 53.24                 | 30.71  | 13.60  | 95.82   | 54.55  | 29.75  |
| CWP15-8 M    | 106.84                   | 49.53  | 17.20  | 53.52                 | 30.47  | 15.53  | 95.37   | 55.55  | 30.77  |
| Control-16 M | 107.87                   | 48.80  | 16.62  | 53.51                 | 30.21  | 14.65  | 95.80   | 55.53  | 30.49  |
| CWP5 -16 M   | 107.11                   | 53.14  | 17.67  | 55.14                 | 31.88  | 16.22  | 94.97   | 56.80  | 31.66  |
| CWP10-16 M   | 105.67                   | 54.22  | 17.96  | 55.39                 | 32.86  | 17.63  | 94.84   | 57.06  | 32.02  |
| CWP15 -16 M  | 106.41                   | 54.69  | 18.18  | 56.70                 | 33.78  | 19.55  | 94.04   | 57.35  | 32.68  |

**Fig. 7.** Residual compressive strengths of the AAM specimens after being exposed to elevated temperatures.**Fig. 8.** Residual flexural strengths of the AAM specimens after being exposed to elevated temperatures.

samples without CWP and 8 M SH molarity exhibited the poorest flexural strength. The favorable contribution of CWP material on flexural strength increased with an increase in SH molarity. Similar to compressive strength enhancement, the flexural strength of the specimens was enhanced with time. The flexural strength of the specimens was around 7 MPa at 7-day, 8.5 MPa at 28-day, and 10 MPa at 56-day as given in Table 5. The flexural strength enhancement can be attributed to the ongoing geopolymerization reactions due to the further C-N-S-A-H type gel formations.

Fig. 3 points out the ultrasonic pulse velocity (UPV) values of the

samples at the ages of 7, 28, and 56 days. The findings revealed that CWP inclusions enhanced UPV values, and the maximum UPV values were obtained on the specimens having 15% CWP material, which may be due to the dense microstructure caused by CWP incorporation, resulting in a delay in the wave propagation [36–38]. Similarly, an increase in SH molarity from 8 M to 16 M enhanced the UPV values. This may be attributed to the further C-N-S-A-H type gels with increasing sodium hydroxide molarity, leading to a dense microstructure and resulting in a delay in UPV values. Furthermore, an increase in the age of the specimens improved the UPV results, and the highest UPV values



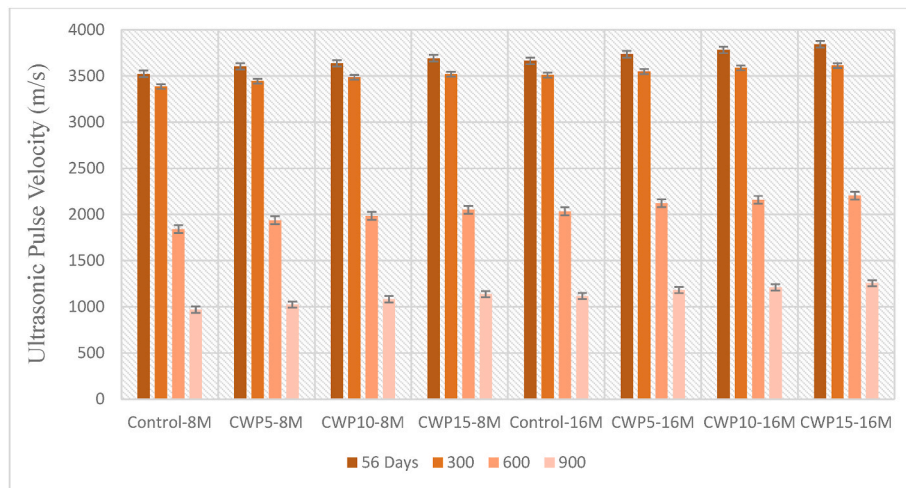


Fig. 9. Residual UPV results of the AAM specimens after being exposed to elevated temperatures.

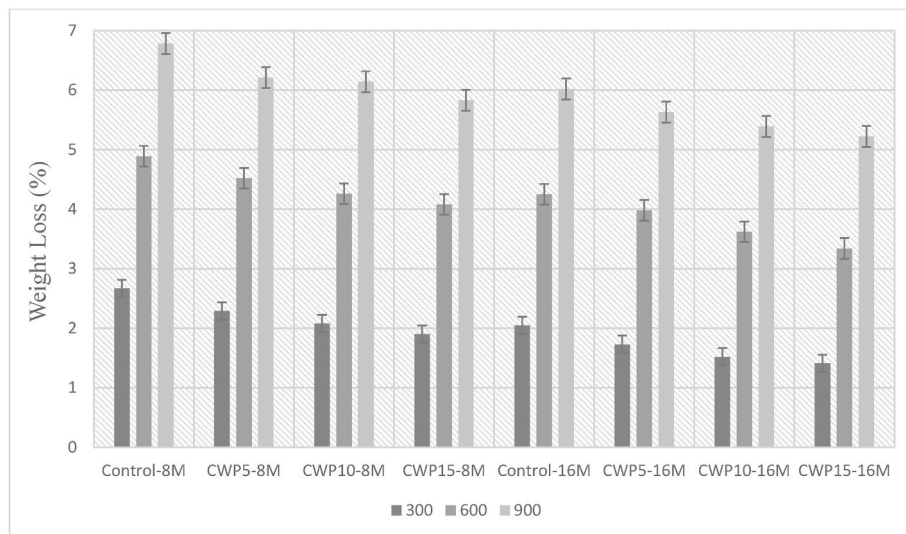


Fig. 10. Weight loss results of the samples at elevated temperatures.

were obtained on the specimens at the age of 56 days. This can be explained by the further geopolymerization reactions with time, resulting in a dense microstructure by the formation of a C-N-A-S-H-type gel. The average UPV values of the specimens were found to be around 3500 m/s, which is in line with the previous investigation [39].

### 3.3. SEM and XRD results

SEM analysis was carried out to examine the changes in the microstructure of the geopolymer mortar samples. While examining the potential characterization changes in the sample microstructure with the molarity effect and the substitute material (ceramic powder) ratio, the degree of the aggregate-binding interfacial transition zone (ITZ) was also examined. SEM micrographs were examined for samples Control-8M, Control-16 M, CWP15-8 M, and CWP15-16 M and are given in Fig. 4. By examining the SEM micrographs, it was determined that the strength results showed parallelism with the analysis results. It was observed that the matrix became denser in terms of silica together with the ceramic powder. While the addition of ceramic powder increased the SiO<sub>2</sub> content, it also increased the C-S-H gel formation with the amount of CaO in the activator and slag content. However, increasing the degree of reaction with alkali activators formed more C-N-A-S-H type gel

products. While low molarity increased the air voids, high molarity was effective by improving the activation process. While the high molarity allowed the structure to be concentrated, the ceramic powder also improved the microstructure. In addition, the fineness of the ceramic powder provided a filling function between the binder and the aggregate, thus strengthening the interface transition zone [20–23,39].

XRD analyzes are given in Figs. 5 and 6 for samples Control-8M, Control-16 M, CWP5-8M, CWP5-16 M, CWP10-8 M, CWP10-16 M, CWP15-8 M, and CWP15-16 M. The high ratio of silica and alumina in the binder materials played a role in the formation of geopolymerization and also created an amorphous phase. According to the diffraction images, it was observed that there were crystal quartz peaks. It was observed that mullite peaks formed lower-intensity peaks. While wide bands in the range of 25–30° 2θ were observed in all samples, this proved that classical amorphous geopolymer gel phases were formed. In addition, it has been observed that the amorphous phases in the main binder materials contributed to the geopolymerization reactively. Geopolymerization has also been found to promote the reaction by contributing to the reorganization of the system structure. With the substitution of ceramic powder, silicon oxide was concentrated in the sample diffraction image, and with the increasing substitution rate, nepheline and albite crystal phases began to be seen. Here, when looking



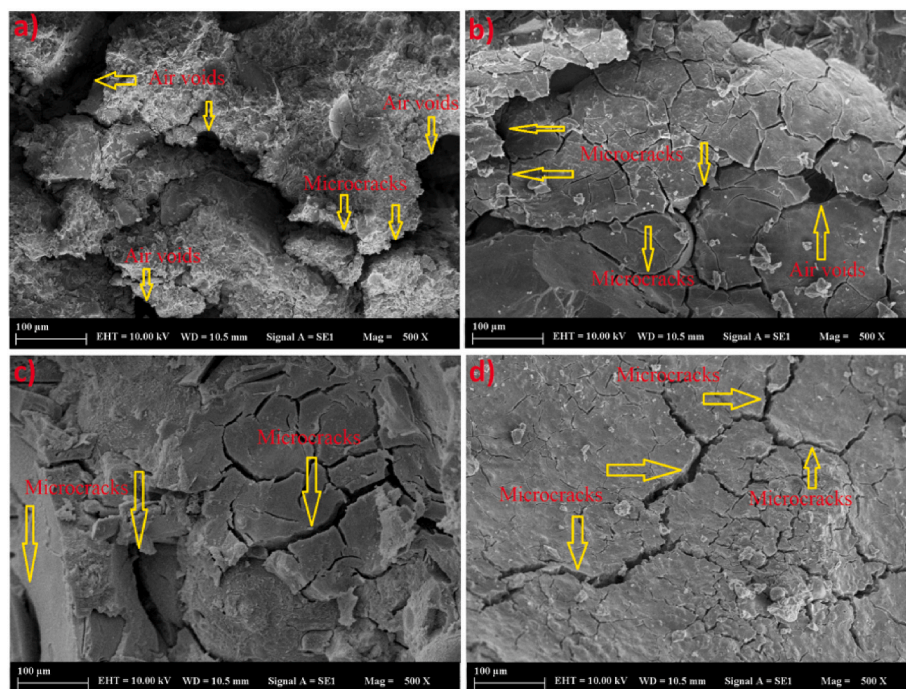


Fig. 11. SEM images of the samples: a) Control-8M, b) Control-16 M, c) CWA15-8 M, and d) CWA15-16 M after 900 °C.



Fig. 12. Photo of the samples: a) Control-8M, b) CWA5-8M, c) CWA10-8 M, d) CWA15-8 M e) Control-16 M, f) CWA5-16 M, g) CWA10-16 M, and h) CWA15-16 M after 900 °C.

at Figs. 5 and 6, the incidence and peak values of nepheline and albite increased with increasing ceramic powder ratio. In the most general sense, ceramics can be defined as inorganic compounds formed as a result of the combination of one or more metals with non-metallic elements and sintering. So, the appearance of nepheline and albite peaks was due to the high-temperature sintering of ceramics. The sintering process caused the amorphous phase to completely transform into the crystalline phase. Thus, the amorphous phase bump disappeared and the crystallographic composition changed. In addition, the increase in ceramic powder resulted in a higher density of quartz peaks. In addition, high molarity also enhanced the geopolymerization and increased the quartz peaks. In other words, alkali activators have contributed by increasing the reaction and improving the N-A-S-H gel formation [20–23,39].

### 3.4. Elevated temperature

Table 6 shows the residual strength and UPV values after being exposed to 300, 600, and 900 °C compared to the strengths of the unexposed specimens at 56 days. The findings revealed that deterioration was found on the samples depending on the exposed temperature level, only except for the compressive strengths at 300 °C. This can be attributed to the further geopolymerization reactions with increasing temperatures up to 300 °C. After elevated temperature, strengths and UPV results were comprehensively evaluated herein.

#### 3.4.1. Evaluation of strength results after elevated temperatures

The effects of elevated temperatures on the resulting performance of AAM samples were evaluated after being exposed to 300, 600, and 900 °C. In Figs. 7 and 8, the residual strengths of the specimens were compared with the unexposed specimens at the age of 56 days. At 300 °C, the compressive strength of the AAM specimens was enhanced by about 7% due to the ongoing geopolymerization reactions. The strength enhancements were found to be less on the CWP-incorporated specimens as compared to the control (without CWP) ones. This may be attributed to the lower fly ash amount at higher CWP incorporations, which requires elevated temperature curing for geopolymerization reactions [8]. Thus, control specimens (including higher FA) showed more compressive strength enhancement. The influence of SH molarity is not clear on the resulting compressive strength performance at 300 °C. Otherwise, residual flexural strength results were also evaluated on specimens at 300 °C. The findings revealed that the flexural strength of the specimens reduced significantly (about 46% reduction) after the elevated temperature of 300 °C. This reduction can be attributed to the fact that flexural strengths are more sensitive to microstructural defect development, cracking formation and propagation, and the growth of porous microstructure [40]. The residual flexural strength was found to be almost 52% for 8 M, and 55% for 16 M, indicating that SH molarity slightly enhanced the residual flexural strength at 300 °C.

Figs. 7 and 8 also show that both mechanical strengths decreased seriously after being exposed to 600 and 900 °C. This can be due to the free water evaporation and dehydration of the matrix, and thermal reaction mechanisms at 600 and 900 °C [41]. The average compressive

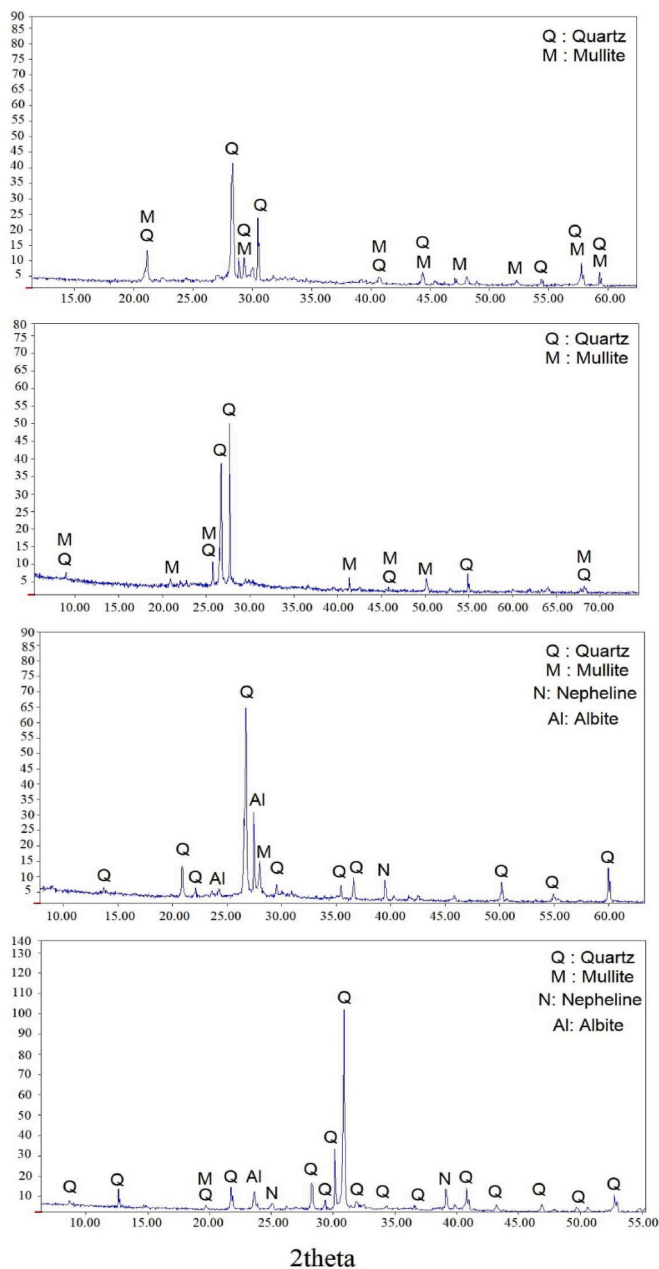


Fig. 13. XRD patterns of the samples: a) Control-8M, b) Control-16 M, c) CWA15-8 M, and d) CWA15-16 M after 900 °C.

strength reductions were about 50% at 600 °C and 85% at 900 °C, while average flexural strength reductions were about 70% at 600 °C and 85% at 900 °C. These higher strength losses can be due to the steam effect at higher temperatures. The free water turns into steam at elevated temperatures, applying tensile stresses to the matrix, and cracks form when the steam pressure surpasses the tensile strength capacity of the matrix. After the thermal crack formation, compactness and rigidity reduce, leading to the loss of mechanical strength.

The results also pointed out that similar residual compressive strengths were obtained at elevated temperatures, irrespective of the SH molarity and CWP incorporations. However, residual flexural strength results revealed that both CWP incorporation and high SH molarity slightly enhanced the residual flexural strength results, and this enhancement was found to be more with increasing elevated temperature. The 15% CWP incorporated specimens having 16 M SH molarity performed superior elevated temperature resistance. This may be

attributed to two effects; first, the filler effect of CWP material enables it to create a dense packing system that favors high performance. Second, a pozzolanic effect is more pronounced at later ages compared with an early age. However, a sufficient amount of calcium hydroxide should be available for the pozzolanic activity of CWP to take place [42].

### 3.4.2. Evaluation of UPV results after elevated temperature

The ultrasonic pulse velocity (UPV) results are influenced by the defects, voids, and pores in the microstructure. After the elevated temperature, porosity increases due to the water evaporations, resulting in lower UPV results. Fig. 9 shows the residual UPV values after 300 °C, 600 °C, and 900 °C. The findings demonstrated that all UPV results reduced seriously after 300 °C, which is consistent with the compressive strength results. The results also pointed out that the influence of CWP and high molarity has no impact on the resulting UPV values, almost similar UPV losses were obtained at 300, 600, and 900 °C. More UPV losses were obtained with an increase in the elevated temperatures.

### 3.4.3. Evaluation of weight loss results after elevated temperature

Fig. 10 demonstrates the weight losses of the samples after being exposed to 300 °C, 600 °C, and 900 °C. The findings revealed that more weight losses were obtained with higher temperatures. The weight losses arising from the elevated temperature slightly decreased with an increase in both the CWP replacement ratio and SH molarity. The lowest weight loss was obtained on the samples having 16 M SH molarity and 15% CWP incorporations. When the temperature rises, dehydration reactions occur and moisture takes away from the inner sides to the outer sides, resulting in internal damage and loss of weight [43]. In the initial stage of the heating, weight loss emerges rapidly due to the bound water as a result of the alkali activation process and water evaporation [44]. As the CWP content increases, the filler effect of CWP material enables it to create a dense packing system that enhances the interfacial transition zone (ITZ). Therefore, free water escape becomes difficult for high CWP inclusions and high SH molarity.

### 3.4.4. SEM and XRD results

After 900 °C was applied for the high-temperature test, SEM analysis was used for the microstructure changes. SEM micrographs were viewed for the samples Control-8M, Control-16 M, CWP15-8 M, and CWP15-16 M and are shown in Fig. 11. The high-temperature effect caused the formation of micro-cracks. Low molarity reduced the formation of N-A-S-H gel, which was indicative of geopolymerization formation, and caused progressive microcracks and increased air voids. While the geopolymer matrix structure deteriorated, weight losses and phase transformations increased this situation. Defects have increased and intensified with the effect of all the situations. Increasing defects also resulted in a more porous microstructure. The use of ceramic powder as a substitute material increased the degree of geopolymerization and increased the response to high temperatures. However, 16 M sodium hydroxide formed more N-A-S-H gel, resulting in a stronger matrix structure. Despite the parallel decreases in mechanical properties, the geopolymer specimens managed to maintain their stable positions. Although high temperatures caused a brittle structure by increasing crack formation in the structure, large pieces that were dispersed and broken have not been seen and the stable position has not been lost. The resulting conditions were also supported by visual inspection of all samples in Fig. 12 after 900 °C [20–23,39].

While the compositions of the reaction products formed in the geopolymer mortar samples with the effect of high temperature were determined by XRD analysis, the occurrences of the phase transformations in the sample structure were also examined in detail. After 900 °C application, XRD spectra of samples Control-8M, Control-16 M, CWP15-8 M, and CWP15-16 M were examined and shown in Fig. 13. The deteriorated mechanical properties created structural problems after 900 °C was applied for high-temperature testing. Atomic species such as Si and Al formed a rearranged and restructured microstructure, while



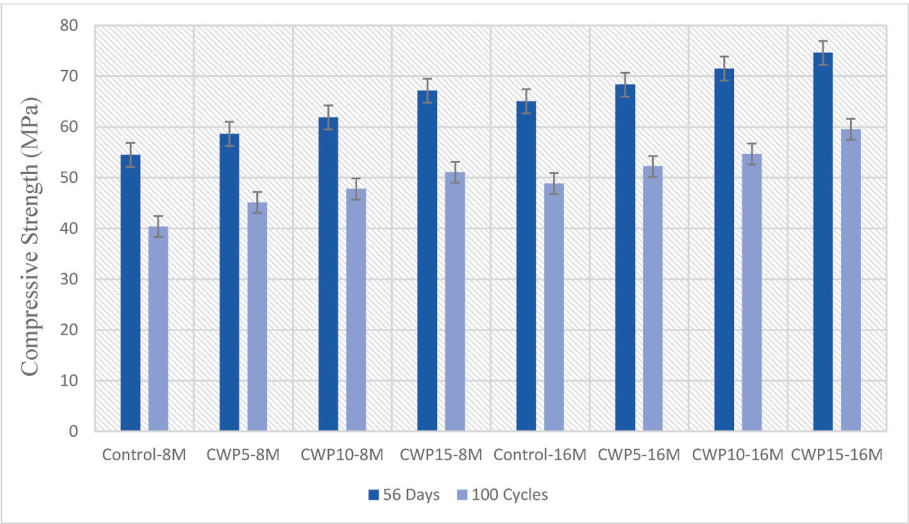


Fig. 14. Residual compressive strengths of the AAM specimens after being exposed to freeze-thaw attack.

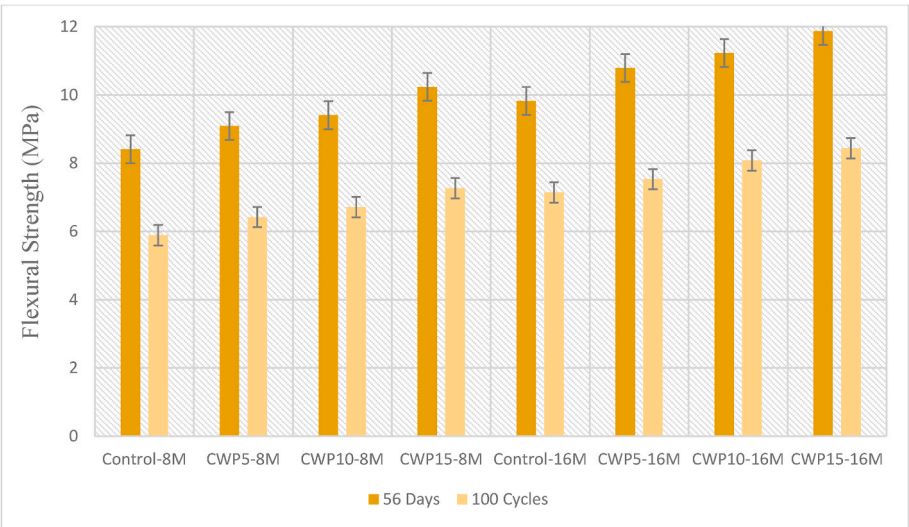


Fig. 15. Residual flexural strengths of the AAM specimens after being exposed to freeze-thaw attack.

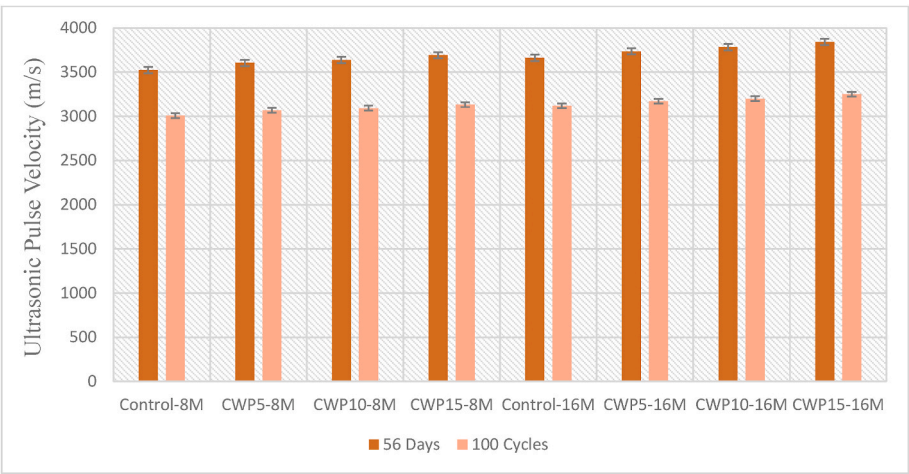


Fig. 16. Residual UPV results of the AAM specimens after being exposed to freeze-thaw attack.



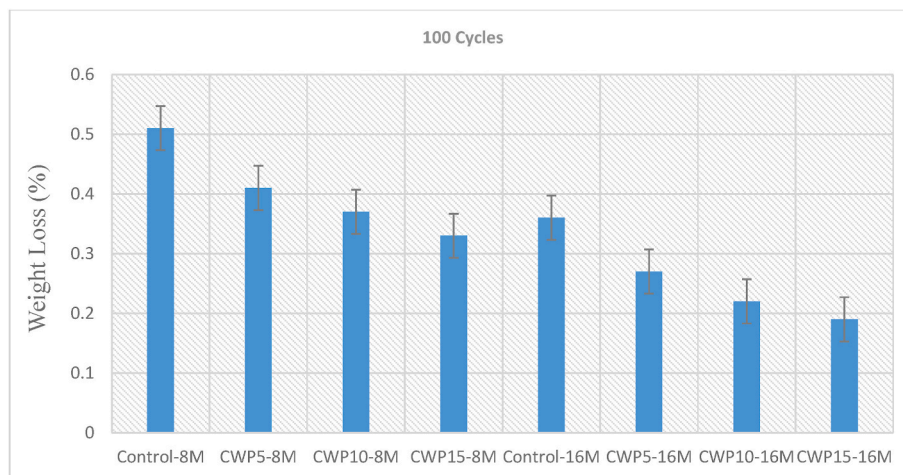


Fig. 17. Weight loss results of the AAM specimens after being exposed to freeze-thaw attack.

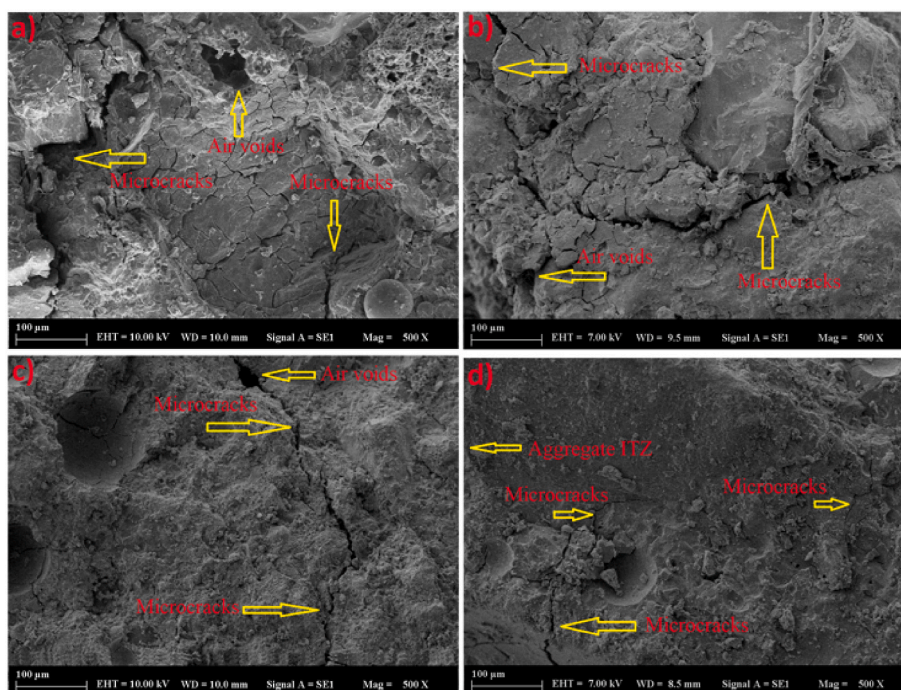


Fig. 18. SEM images of the samples: a) Control-8M, b) Control-16 M, c) CWA15-8 M, and d) CWA15-16 M after being exposed to freeze-thaw attack.

the high temperature shifted the amorphous humps to lower angles. Thermal incompatibility and crystallization caused gel separation and decreased the strength values. There were sufficient voids in the matrix structure for alkali ions, weakly bonded water molecules, and tetrahedral  $\text{AlO}_4$  and  $\text{SiO}_4$  units found in polymeric chains. The temperature created high kinetic energy in the water molecules in the voids. While the increased kinetic energy caused the internal pressure to rise, there were divergences in the form of steam. Internal forces and microstructural distortions as a result of displacements in XRD humps caused internal cracks and surface cracks. Although the negative conditions were reflected in the XRD spectra, it was determined that the basic structure was preserved for the classical geopolymer and the deterioration remained at a low rate. Geopolymer samples showed good resistance in terms of thermal stability with a zeolite-like structure. The mixing procedure, binder material type, aggregate type, activator type, unreacted alumina and silica, and other impurities were effective in the differentiation of characteristic peaks. The high silica content of the

ceramic powder and the high fineness supported the strengthening of the reaction. In addition, the increase in the ceramic powder ratio also formed nepheline and albite crystal phases. However, high molarity also increased the formation of N-A-S-H gel, resulting in the geopolymerization progress. These cases were seen at peaks [20–23,39].

### 3.5. Freeze-thaw results

During the freeze-thaw experiments, the AAM specimens were subjected to 100 freeze-thaw cycles, and the duration of freezing and thawing cycles that range from  $-20^\circ\text{C}$  to  $20^\circ\text{C}$  was 6 h. Figs. 14–16 illustrates the residual strengths and UPV results of the specimens after freeze-thaw attacks. The results of the freeze-thaw tests were compared with the unexposed specimens at 56 days. The results pointed out that a reduction was observed in both compressive and flexural strengths after freeze-thaw cycles. The residual compressive strengths due to the freeze-thaw attacks were 74% for Control-8M, 77% for both CWP5-8M and



Fig. 19. Photo of the samples: a) Control-8M, b) CWA5-8M, c) CWA10-8 M, d) CWA15-8 M e) Control-16 M, f) CWA5-16 M, g) CWA10-16 M, and h) CWA15-16 M after 100 cycles.

CWP10-8 M, 76% for CWP15-8 M, 75% for Control-16 M, 76% for CWP5-16 M and CWP10-16 M, and 80% for CWP15-16 M specimens with an average residual compressive strength of 77% as compared to the unexposed compressive strength of the AAM specimens at 56-day. The residual flexural strengths due to freeze-thaw attacks were 70%, 71%, 71%, 71%, 73%, 70%, 72%, and 71% for the Control-8M, CWP5-8M, CWP10-8 M, CWP15-8 M, Control-16 M, CWP5-16 M, CWP10-16 M, and CWP15-16 M specimens, respectively. The average residual flexural strength loss became 71% as compared to the unexposed flexural strength at 56-day. The mechanical strength results pointed out that almost similar mechanical strength losses were observed on the AAM specimens irrespective of SH molarity and CWP replacement ratio after freeze-thaw cycles. The mechanical strength reduction mechanisms due to the freeze-thaw cycles can be explained briefly. During freezing-thawing cycles, samples were left in water, enabling to fill of the pores with water, which is useful for C-N-S-A-H type of geopolymerization reaction products. After several freeze-thaw cycles, softening occurs first on the surface, then the disintegration of mortars starts from the surface and progresses into the inner parts (core region) of the specimens. The reason behind the disintegration process is the water expansion in the case of freezing. In the case of freezing, water volume rises by about 9%, and hydraulic pressure is emerged [45]. If the stress surpasses the tensile strength of the specimens, micro-cracks occur and deterioration initiates from the cracked regions. Then, depending on the number of freeze-thaw cycles, deterioration progresses, resulting in a loss of strength.

Fig. 16 shows the UPV results of the AAM specimens after the freeze-thaw attack. A similar UPV reduction was obtained on the specimens with and without CWP incorporation and high molarity. The UPV values of the AAM specimens were slightly reduced from 3500 m/s to 3000 m/s for all specimens, indicating a similar deterioration for all AAM samples after freezing-thawing cycles. Increasing the substitution of ceramic powder and higher molarity values decreased the loss in UPV values. This was related to the greater adhesion between the matrix and the aggregate. In addition, due to the fineness of the ceramic powder, it filled the voids and made the pores more impermeable. The increase in the degree of continuity of the pores was effective in this case. Similar results were obtained for weight losses after 100 cycles (Fig. 17), and overall weight losses remained low due to the compact structure of the geopolymer matrix [20–23,39].

SEM micrographs after 100 cycles were investigated for samples Control-8M, Control-16 M, CWP15-8 M, and CWP15-16 M. Fig. 18 shows the SEM micrographs examined. The presence of micro-cracks was detected by examining the micrographs. The interface between the gel and the fly ash created a defective region, facilitating the formation of micro-cracks. The micro-cracks caused by the formed defects were connected to each other and formed a network. The fact that the

geopolymer matrix had a compact structure reduced the effects of these conditions and reduced the damages. In this way, slow micro-crack formation was observed. Thus, there were low UPV and strength losses. While low molarity weakened the microstructure, it led to an increase in the freeze-thaw effect. The fineness of the ceramic powder increased the microstructure density by filling the voids. As a result of this situation, the durability has increased. In addition, the high silica content also contributed to the geopolymerization. These conditions increased the resistance of geopolymer composites to 100 cycles test. The results obtained were also compatible with the literature [39,45]. The appearance of the samples in Fig. 19 after 100 cycles also supported this situation.

#### 4. Conclusions

In the research, the influences of ceramic waste powder replacement (0%, 5%, 10%, and 15%) and SH molarity (8 M and 16 M) on the mechanical and durability performance of fly ash/slag-based alkali-activated mortar (AAM) specimens were investigated. The following findings were obtained:

- When the physical properties were examined, it was observed that the flowability property and setting time (initial and final) decreased with increasing sodium hydroxide molarity, while they increased with the CWP substitution ratio. So, the superior flowability and the highest initial and final setting times were found on the specimens having 15% CWP and 8 M SH molarity. However, it was observed that the void ratio and water absorption decreased due to the formation of a dense and compact structure with both an increasing CWP replacement ratio and increased SH molarity.
- The compressive and flexural strengths and ultrasonic pulse velocity (UPV) values improved with higher CWP inclusions and SH molarity. Due to the ongoing geopolymerization, compressive strength enhancement was found to be more with time. The average compressive strength increments were found to be about 27% from 7-day to 28-day and 23% from 28-day to 56-day. The CWP replacements from 0 to 15% increased the compressive strength values up to 10 MPa. The strength enhancement became more with a higher CWP replacement ratio and SH molarity.
- After exposure to 300 °C, compressive strength results improved by 7% due to shrinkage and greater geopolymerization, as opposed to flexural strength. Here, it was seen that the increase rates were higher in the samples without CWP. This was due to the higher ratio of fly ash, which required higher temperatures for curing, in these samples. Flexural strength, on the other hand, decreased because it was more susceptible to microstructural defect development, crack formation and propagation, and the growth of porous microstructure. The effect of SH molarity was not clear on the resulting compressive strength performance at 300 °C.
- After exposure to 600 °C and 900 °C, all properties decreased due to free water evaporation, dehydration of the matrix, thermal reactions, and vapor pressure. Average compressive strength losses were approximately 50% at 600 °C and 85% at 900 °C, while average flexural strength losses were approximately 70% at 600 °C and 85% at 900 °C. However, both the filling effect and the pozzolanic effect of CWP, as well as the high SH molarity, increased the geopolymerization, increasing the residual mechanical properties at increasing high temperatures.
- After 100 cycles of freeze-thaw attacks, an average of 23% compressive strength loss and 29% flexural strength loss was obtained on the AAM specimens. Almost similar mechanical strength losses were observed on the AAM specimens irrespective of SH molarity and CWP replacement ratio.



## CRediT authorship contribution statement

**Ferhat Erol:** acquisition of data, Formal analysis, Writing – original draft, Writing – review & editing. **Mukhallad M. Al-mashhadani:** Conception and design of study, acquisition of data, Formal analysis, interpretation of data, Writing – original draft, Writing – review & editing. **Yurdakul Aygörmmez:** Conception and design of study, acquisition of data, Formal analysis, interpretation of data, Writing – original draft, Writing – review & editing, critically for important intellectual content. **Anıl Niş:** Conception and design of study, acquisition of data, Formal analysis, interpretation of data, Writing – original draft, Writing – review & editing, Approval of the version of the manuscript to be published.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

No data was used for the research described in the article.

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