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Research Article

Effect of cement and lime on strength and high-temperature resistance of class F and C fly ash-based geopolymer mortars

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ABSTRACT

Geopolymers have advantages such as good high-temperature, acid and sulfate resistance. Recently, researchers have been working on cement-geopolymer hybrid materials. According to these studies, it is possible to adjust the setting times, to gain strength at ambient temperature and to increase the strength with the use of cement. However, it is known that the structural stability of cement deteriorates at high temperatures, lowering its strength. In this study, the effect of slaked lime and cement inclusion on the strength and high-temperature resistance of Class F and Class C fly ash-based geopolymer mortars was investigated. For this purpose, fly ash was replaced with 10, 20 and 30% cement or 5, 10, 20 and 30% slaked lime. The lime and cement substitutions decreased the compressive strength by 8.9–24.4% in Class F fly ash-based geopolymer mortars. In Class C fly ash, however, the cement addition increased the compressive strength up to 46.6%, but the lime inclusion decreased the strength slightly. There was no significant change in the high-temperature resistance of cement or lime-included Class F fly ash geopolymer mortars exposed to 900°C. However, serious decrease was recorded in the high-temperature resistance of Class C fly ash geopolymers upon partial replacement of the fly ash with either cement or lime.

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1. INTRODUCTION

Portland cement concrete is the most used building material in the world. However, in addition to the high amount of carbon dioxide emitted, several other harmful wastes such as sulphur dioxide and dust are released to the atmosphere during cement production. Due to the rapid population growth, cement requirement is increasing day by day, and the damage to the environment is also increasing [1]. Cement production not only releases high amounts of CO_2, but also causes soil and water pollution, high energy and natural resources consumption [2]. According to the International Energy Agency [3], approximately 4.3 billion tons of cement was produced in the world in 2020. Approximately 1 ton of CO_2 is emitted per ton of cement produced [4]. These figures draw attention to the damages caused by the cement industry.

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Table 1. Chemical composition and physical properties of fly ashes and cement

Compound (% by weight)	Class F fly ash	Class C fly ash	Cement
CaO	4.93	15.94	63.95
SiO_2	53.45	47.07	20.14
Al_2O_3	20.63	11.56	4.7
Fe_2O_3	9.79	7.22	3.2
MgO	1.95	7.77	1.41
SO_3	0.19	2.78	3.04
Na_2O	1.12	1.59	0.48
K_2O	2.07	3.04	0.63
Loss on ignition	4.46	0.42	2.43
Physical properties			
Specific gravity	2.32	2.55	3.11
% retained on 45 µm sieve	15.5	32.5	2.1
Blaine specific surface (cm^2/g)	5253	2980	4044

Alternative binders are being developed and used in order to reduce the disadvantages of cement production. Substituting pozzolans with cement or using binders other than portland cement are among the alternatives [5]. Geopolymers are materials produced by dissolving and reacting aluminosilicates in an alkaline environment. It is possible to use natural aluminosilicates such as kaolin or metakaolin, as well as industrial waste materials such as fly ash and slag. By using industrial wastes in the production of geopolymer, a construction material that is both cheap and environmentally friendly is obtained [6]. In their study, Lloyd and Rangan [7] reported that the use of fly ash-based geopolymer concrete would be 10–30% cheaper than conventional concrete. Owing to their sustainable character and outstanding properties, the interest in geopolymers is increasing day by day [8].

The presence of high amount of calcium in fly ash used in the production of geopolymer may result in C-S-H formation besides the geopolymer gel formation. The presence of C-S-H in the geopolymer structure seems to have a positive effect on the mechanical properties of geopolymers. It was also claimed that calcium can act as a balance-cation in the geopolymer structure [9]. Yip et al. [10] showed that in geopolymers produced with calcium-containing base materials, formation of C-S-H in addition to the geopolymer gel is possible. For this reason, researchers conducted studies on cement/geopolymer hybrid materials. Phoo-ngernkham et al. [9] investigated the effect of partial replacement of high-lime fly ash with cement on the compressive strength of geopolymer paste. For this purpose, 5, 10 and 15% of fly ash was replaced with cement. The cement substitution was considered to increase the compressive strength. The fact became more pronounced with the rising cement substitution level. Temuujin et al. [11] investigated the effect of CaO and $Ca(OH)_2$ replacement on the mechanical properties of fly ash-based geopolymer pastes. The rate of substitution ratios was 1, 2 and 3% by weight for CaO and 1.3, 2.6 and 3.9% for $Ca(OH)_2$. Ambient curing and 70°C oven curing were applied. It was reported that the effect of both CaO and $Ca(OH)_2$ on the geopolymer strength was significantly affected by the curing temperature and lime substitution level. Cao et al. [12] investigated the effect of calcium aluminate cement substitution on fly ash-based geopolymer concrete. 5, 10 and 20 wt.% of fly ash was replaced with cement and, activators produced with different NaOH concentrations were used. The positive effect of cement addition on the strength was emphasized and the optimum replacement ratio of cement reported as 10%.

Geopolymers have a lot of advantages such as good mechanical properties and excellent durability. It is reported that the high-temperature resistance of geopolymers is better than that of the portland cement [13]. The C-S-H and CH, the hydration products of ordinary portland cement, are not stable at high temperatures. The geopolymerization products, on the other hand, are more resistant to high temperatures [14]. However, C-S-H, which is likely to form in the structure of geopolymers containing high amounts of calcium, such as high-calcium fly ash or blast furnace slag, can reduce the high temperature resistance of the material.

In this study, the effects of calcium substitution from different sources (cement and slaked lime) on some properties of geopolymer mortars were investigated. It is well known that the increase in calcium content can cause the formation of additional phases such as C-S-H, C-A-S-H in the geopolymer matrix. To examine the effects of these possible new phases on compressive strength and high temperature resistance, cement and slaked lime, which are popularly used as building materials, were used. Either 10, 20 and 30 wt.% of the fly ash was replaced with cement or 5, 10, 20 and 30 wt.% of the fly ash was substituted by lime.

2. MATERIALS AND METHOD

2.1. Materials

Two types of fly ashes (F and C) were used as aluminosilicate sources in the study. The Class F fly ash was supplied from Izdemir Enerji/Izmir and the Class C fly ash was supplied from Cayirhan Thermal Power Plant/Ankara. An ordinary CEM I 42.5 R type cement and slaked lime were used as replacement materials. The chemical composition and some physical properties of fly ashes and cement are shown in the Table 1.

SEM images of fly ashes are shown in Figure 1. These images were obtained using by "Thermo Scientific Apreo S" device with the range of 5 and 7.5 kV. SEM images revealed that the Class F fly ash particles were more rounded than those of the Class C fly ash. Moreover, the particles of Class F fly ash were found to be more uniform in size than that of Class C fly ash particles.

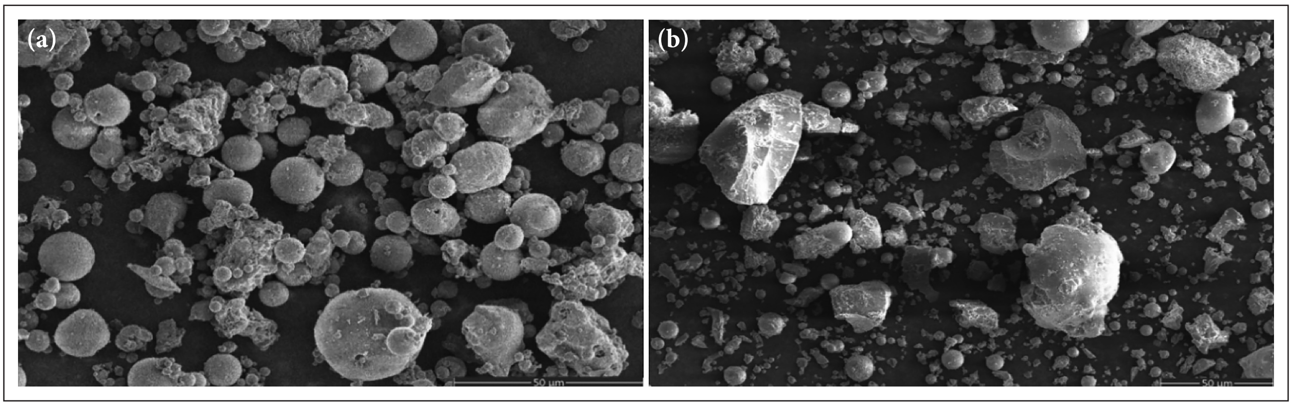

Figure 1. SEM images of fly ashes (a: Class F, b: Class C)

Sodium silicate solution, containing 11.32% Na_2O, 28.22% SiO_2 and 60.45% H_2O by weight, and sodium hydroxide in the form of pellets with 98% purity were used as activators. Sodium hydroxide pellets were dissolved in a given amount of sodium silicate solution to obtain the desired Ms ratio (total SiO_2 to Na_2O ratio by weight in activator). The obtained solution was used after 24 hours of rest.

Tap water and crushed limestone sand was used in the preparation of mortar mixes. The gradation of the sand is shown in the Figure 2.

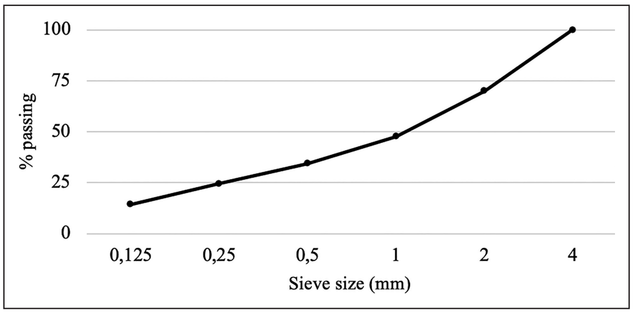

Figure 2. Gradation of sand.

2.2. Method

The experimental part of the study consisted of 2 stages.

At the first stage, the proportions of mixtures to obtain the highest compressive strength for each aluminosilicate were determined. For this purpose, 9 mortar mixtures were prepared with each aluminosilicate by using activators designed with 3 different Ms ratios and 3 different Na_2O percentages (total Na_2O ratio of the activator by weight of aluminosilicate). The compressive strengths as well as flow diameters of the mortar mixtures were determined. The flow diameters of the mortars were determined according to the ASTM 1437-20 [15] standard. In order for the mortars to have similar flow diameters, trail mixtures were prepared using different amounts of water. The target flow diameter for the mixtures was chosen as 18.5±1 cm. For the target flow value, Class C fly ash-based mortars water demand was higher than that of the Class F fly ash-bearing mixtures. The fact seems to be arisen from the morphological characteristics (angular shape) of the Class C fly ash particles which cause greater internal friction in the mixture reducing its consistency.

The ingredients were placed in the Hobart mixer bowl and mixed for 45 seconds at low speed. After scraping the materials adhering to the container, the mixer was operated for another 45 seconds at the same speed and the flow diameters were determined. The mortars were placed in the 50 mm cube molds in 2 layers and each layer was compacted by 25 drops in a jolting table. Immediately after preparing, the specimens were placed in an oven and cured at 60°C for 1, 3 and 5 days. At this stage, the mixture giving the highest compressive strength for each fly ash was chosen as the reference mixture. The proportions of the mixtures produced at this stage are shown in the Table 2. Mixtures were designated as shown in Figure 3. The compressive strength tests were carried out in a 2000 kN capacity concrete press. The loading rate was set as 0.9 kN/s. The reported strength values are the average of 3 samples.

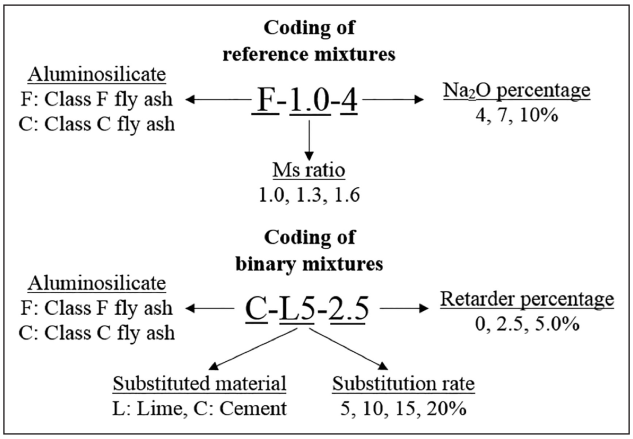

Figure 3. Designating of geopolymer mortar mixtures.

At the second stage, definite amounts of fly ashes were replaced with cement or slaked lime. The effects of substituted materials on the compressive strength and high-tem-

Table 2. Mix proportions and flow diameters of mixture

Code	Activator (g)	Water (g)	Flow diameter (cm)	Code	Activator (g)	Water (g)	Flow diameter (cm)
F-1.0-4	103.7	150.4	18.0	C-1.0-4	103.7	234.3	17.7
F-1.0-7	181.5	68.5	17.8	C-1.0-7	181.5	181.5	18.5
F-1.0-10	259.3	87.1	18.0	C-1.0-10	259.3	136.8	18.0
F-1.3-4	125.8	133.4	19.0	C-1.3-4	125.8	243.8	18.2
F-1.3-7	219.7	69.6	18.0	C-1.3-7	219.7	61.5	18.0
F-1.3-10	313.8	28.4	19.0	C-1.3-10	313.8	27.7	18.0
F-1.6-4	147.4	108.3	19.2	C-1.6-4	147.4	214.0	18.8
F-1.6-7	257.9	29.6	18.5	C-1.6-7	257.9	99.7	17.8
F-1.6-10	368.3	0	20.0	C-1.6-10	368.3	52.8	22.0

Sand: 1620 g, fly ash: 600 g for all mortar mixtures.

Table 3. Proportions and flow diameters of mixtures containing cement and lime

	Material (g)							Flow diameter (cm)
	Sand	Fly ash	Cement	Lime	Activator	Retarder	Water	
F-1.3-10 (R)	607.5	225.0	–	–	117.5	–	10.7	19
F-C10	607.5	202.5	22.5	–	117.5	–	12.4	
F-C20	607.5	180.0	45.0	–	117.5	–	21.8	
F-C30	607.5	157.5	67.5	–	117.5	–	20.6	19±0.3
F-L10	607.5	202.5	–	22.5	117.5	–	18.9	
F-L20	607.5	180.0	–	45.0	117.5	–	21.5	
F-L30	607.5	157.5	–	67.5	117.5	–	35.2	
C-1.3-10 (R)	607.5	225.0	–	–	117.5	–	10.4	18
C-C10	607.5	202.5	22.5	–	117.5	–	34.3	
C-C20	607.5	180.0	45.0	–	117.5	–	33.1	
C-C30	607.5	157.5	67.5	–	117.5	–	34.0	
C-L5	607.5	213.8	–	11.2	117.5	–	15.4	
C-L5-2.5	607.5	213.8	–	11.2	117.5	5.2	38.3	
C-L5-5	607.5	213.8	–	11.2	117.5	10.6	34.9	18±0.3
C-L10	607.5	202.5	–	22.2	117.5	–	32.0	
C-L10-2.5	607.5	202.5	–	22.2	117.5	5.0	39.1	
C-L10-5	607.5	202.5	–	22.2	117.5	10.1	40.3	
C-L20	607.5	180.0	–	45	117.5	–	40.5	
C-L20-2.5	607.5	180.0	–	45	117.5	4.5	53.0	
C-L20-5	607.5	180.0	–	45	117.5	8.9	52.9	

R: Reference mixture.

perature resistance (300, 600, 900°C) of geopolymer mortars were determined. At this stage, it was aimed to keep the flow diameters of the mixtures as close as possible to that of the reference mixture. For this purpose, differences between the flow diameters of cement- and lime-bearing mixtures and that of the control mixtures were kept within the range of ±0.3 cm. There was a rapid loss of consistency in some of the test mixtures. Thus, a retarder admixture was added to these mixtures. Trial and error were applied to determine the retarder admixture requirement of the mixtures. The proportions of the mixtures containing cement and lime are shown in Table 3.

Payakaniti et al. [13] investigated the high temperature resistance of Class C fly ash based geopolimer pastes and determined that the compressive strength starts to decrease after temperatures of 200°C, the rate of decrease was more considerable in the range of 400–600°C, and the rate of strength loss decreased after 600°C. Researchers also

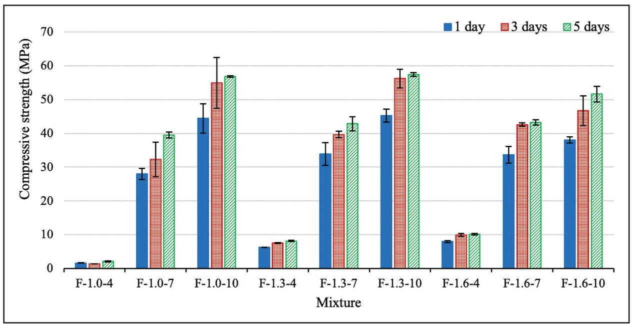

Figure 4. Compressive strength of class F fly ash-based geopolymer mortars.

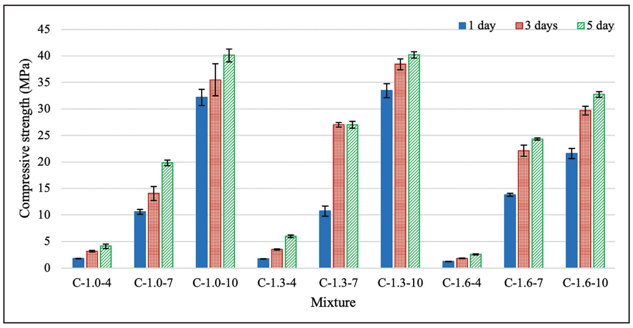

Figure 5. Compressive strength of class C fly ash-based geopolymer mortars.

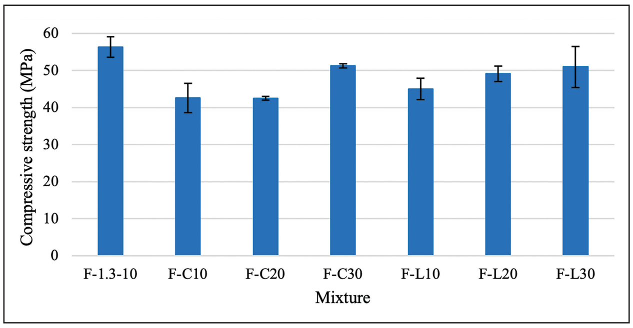

Figure 6. Compressive strength of Class F fly ash-based geopolymer mortars including cement and lime.

reported that new crystalline phases appeared in the matrix 800°C and beyond that these crystalline phases had an positive or negative effect on the strength depending on the numbers of these new crystals. Lahoti et al. [16] stated that the first major shrinkage in geopolymers under the influence of high temperature starts at 150–300°C, and the second major shrinkage starts around 600°C in geopolymers produced with activators containing sodium ions. Regarding these findings and probability of presence of components such as C-S-H and CH in the matrix, it was decided to conduct high temperature resistance tests at 300, 600 and 900°C in the present study. The experiments were carried out in a muffle furnace. The temperature was increased at a rate of 20°C/min and the exposure time at the target temperature was set as 3 hours. At the end of this time, the samples were left to cool in the closed furnace. The compressive strength tests were applied immediately when the specimens cooled to the room temperature.

3. RESULTS AND DISCUSSION

3.1. Determination of Reference Mixtures

The compressive strength of Class F and C fly ash-based geopolymer mortars are shown in Figure 4 and Figure 5, respectively. With the increase in the curing time, the compressive strength of both Class C and Class F fly ash-based mortars increased. At the same Ms ratio and Na_2O percentage Class F fly ash mortars were found to be superior to the Class C fly ash mixtures. It is thought that the high water requirement of Class C fly ash-based mortars for a given flow is the cause of their low strength. The highest compressive strengths at all Ms ratios and Na_2O percentages were obtained with 5 days of curing. However, the increase in compressive strength was limited after 3 days. Chithambaram et al. [17] investigated the compressive strength of fly ash-based geopolymer mortars containing sodium silicate-sodium hydroxide activator in different concentrations at different curing times (3, 7, 28 days) and reported that the compressive strength increased with the increase in curing time, but the rate of gaining strength decreased in time. Bellum et al. [18] examined the 7, 14, 28, 60 and 91- day compressive strength of slag-bearing fly ash-based geopolymers and stated that the increase in curing time had a positive effect on the compressive strength.

When the strengths of the mortars prepared with the same Ms ratio and cured for the same time are examined, it is seen that the compressive strengths of the geopolymers produced with both fly ashes increased significantly with the increase in the Na_2O percentage. The highest compressive strength achievable in mortars produced with activator having 4% Na_2O content was approximately 10 and 6 MPa for Class F and C fly ash mixtures, respectively. However, with the increase of Na_2O ratio, compressive strengths beyond 55 MPa in Class F fly ash geopolymers, and around 40 MPa in Class C fly ash mixtures were observed. Cho et al. [19] investigated the effect of sodium hydroxide concentration on geopolymers by using sodium hydroxide solution with 4, 6, 8 and 10 M concentrations and found that increasing the sodium oxide concentration improved the compressive strength. The researchers stated that this was due to the increased solubility of aluminosilicate with increasing alkalinity of the mixture.

Both Class F and Class C fly ash mortar mixtures with 1.3 Ms ratio and 10% Na_2O content subjected to 5 days curing showed the highest compressive strength. However, since the compressive strengths of the mixtures cured for 3 and 5 days were very close to each other, thus, 3 days was decided to be sufficient for the curing of the mixtures.

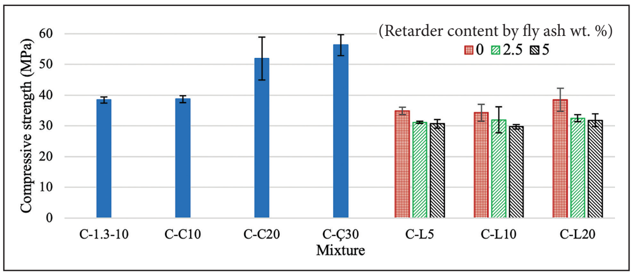

Figure 7. Compressive strength of Class C fly ash-based geopolymer mortars including cement, lime and retarder.

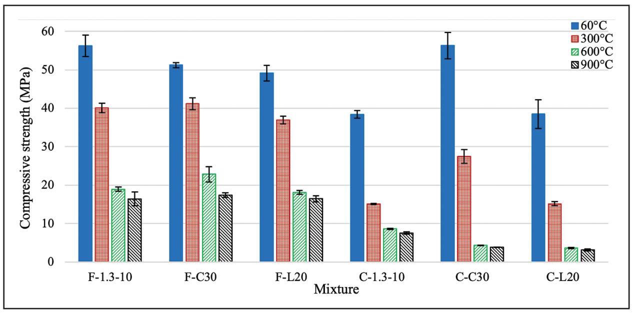

Figure 8. Compressive strength of geopolymer mortars exposed to high temperatures.

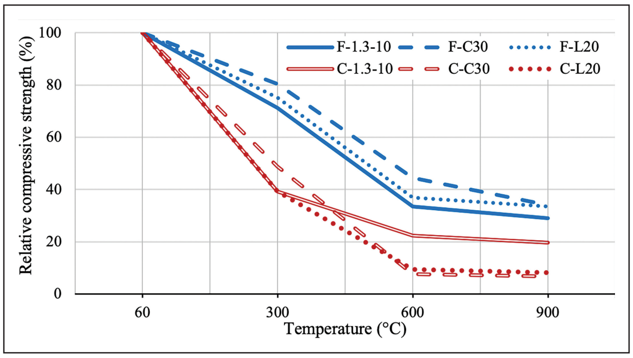

Figure 9. Relative compressive strength of geopolymer mortars exposed to high temperatures.

3.2. Determination of Effect of Cement and Slaked Lime Replacement

The effect of cement and lime on compressive strength of Class F and C fly ash-based geopolymer mortars are shown in Figure 6 and Figure 7, respectively. Within the scope of the study, 10, 20 and 30% lime and cement were replaced with Class F fly ash and the effect of the replacement of these materials on the compressive strength was investigated. The replacement levels of Class C fly ash with cement were same as those of Class F fly ash. However, the substitutions levels of lime with Class C fly ash was somewhat different, i.e., 5, 10, 20 and 30%. Since it was determined that the lime substituted Class C fly ash geopolymers set very quickly (with in 3–4 minutes), mixtures with set retarder were also produced with two different admixture dosages (2.5 and 5.0% by weight of fly ash). In spite of presence of set retarder admixture, the Class C fly ash-based geopolymer containing 30% lime showed flash set during mixing. Therefore, the mixture containing 30% lime could not be produced. Similarly, workability deficiencies were observed in Class F fly ash mixtures containing 30% lime.

Vafaei and Allahverdi [20] studied the effect of partial substitution of natural pozzolan with calcium aluminate cement on the compressive strength of geopolymer mortars. It was reported that the compressive strengths increased with calcium aluminate cement replacement. The strengths increased gradually further with increasing the cement replacement level. The researcher stated that the increased amount of alumina in the geopolymer mixture is important for the formation of N-A-S-H gel, and extra CaO in cement may react with SiO_2 and Al_2O_3 to form C-A-S-H. Yip and van Deventer [21] investigated the formation of C-S-H in geopolymers produced using metakaolin and blast furnace slag. Researchers reported that when calcium-containing aluminosilicates are used in the production of geopolymers, C-S-H could form in the matrix in addition to the geopolymer gel. However, the Ca/Si ratio of this C-S-H was found to be lower than that formed upon portland cement hydration. The increase in strength of the geopolymer mixtures observed in this study was attributed to the C-S-H formation. In the present study, it was observed that the compressive strength of Class F fly ash mortars decreased by 8.9 to 24.4% with cement and lime replacement. However, calcium inclusion was expected to improve strength by formation of additional phases (like C-S-H or C-A-S-H) in the matrix. The fact seems to be due to the effect of the extra water requirement of the mixture for the same workability. A similar situation was observed in Class C fly ash upon substitution of small amount of lime. With lime replacement, a strength loss of up to 10.9% occurred in the compressive strength of these mixtures. The addition of retarder resulted in a further reduction in strength. The water present in the retarder is thought to be the reason for the further strength reduction. However, in spite of its high water content, the compressive strength of cement-bearing Class C fly ash mixtures improved at all inclusion ratios reaching 34.5% and 46.6% upon 20% and 30% cement addition. The improvement in compressive strength of these mixtures, in spite of their high water content, was probably due to the presence of new phases in the matrix. Chindaprasirt et al. [22] also reported that calcium hydroxide and cement substitution improved the compressive strength of alkali-activated fly ash pastes. According to the researchers, this was related to the formation of additional C-S-H and C-A-S-H phases in the matrix and the shortening of the both initial and final setting times. Temuujin et al. [11] stated that the compressive strength of fly ash-based geopolymer pastes cured at ambient temperature increased with the inclusion of CaO and $Ca(OH)_2$, but on the contrary, the compressive strengths decreased upon curing at 70°C. The researchers attributed the fact to the increased rate of evaporation of water at high temperature increasing the porosity.

3.3. Determination of the Resistance of Selected Mixtures to High-Temperature

High- temperature resistance is also one of the important durability issues for geopolymers. There are many studies on the high- temperature resistance of geopolymers in the literature. F-C30, C-C30, F-L20 and C-L20 mixtures were selected for high-temperature resistance tests. Except for F-L20 mixture, the others showed the highest strength in their own group. Owing to its very short setting time, F-L30 mixture having the highest strength in its group was not selected.

The compressive strengths of the selected geopolymer mortars before and after exposure to 300, 600, 900°C are shown in Figure 8, and the relative compressive strengths are shown in Figure 9. The compressive strength of both Class C and Class F fly ash-based mortars decreased gradually with the increase in temperature. The reduction in strength of Class F fly ash mixtures after exposing to 300°C was in the range of 19.6 to 28.8%. The corresponding range was 51.2 to 60.7% in Class C fly ash mortars. The higher amount of calcium in Class C fly ash-based mixtures seems to be the reason for this situation. With the increase in temperature, the strength losses also increased so that upon exposure to 900°C the residual strengths in the Class F and C fly ash-based mortars were merely in the range of 29.1 to 34.0% and 6.8 to 19.7%, respectively. The presence of 16–17 MPa residual strength in Class F fly ash mixtures even after exposure to 900°C is a good measure of the high-temperature resistance of these mixtures. Klima et al. [23] stated that the evaporation of the water present in the geopolymers at high temperatures caused both thermal shrinkage and vapour pressure damaging the structure. Payakaniti et al. [13] reported that the compressive strength losses at temperatures higher than 400°C in geopolymers may be caused by thermal stresses, and the changes in the crystal structure at 1200°C was found to be responsible for further reduction of the mechanical properties. A very important part of the strength loss experienced within the scope of the present study was observed at temperatures up to 600°C. It is thought that the evaporation of water coupled with thermal shrinkage/stress are the causes of the strength loss.

As it can be seen from Figure 9, the loss in strength of Class F fly ash mixtures either with or without inclusions upon exposure to extreme temperatures are very close to each other, irrespective of the temperature level. This indicates that either the presence or type of the substitute has no significant effect on residual strength of Class F fly ash geopolymer exposed to high temperature. However, the opposite is true for Class C fly ash-based mortars. The cement and lime substituted Class C mixtures showed considerably higher loss in strength than their control mixture particularly when exposed to 600 and 900°C. The fact probably is arisen from the increase in the amount of C-S-H in the matrix due to the increased calcium content, which also negatively affects the compressive strength after exposing to high temperatures.

4. CONCLUSION

For the materials used and test methods applied the following conclusions were drawn:

- Class C fly ash had a lower fineness than Class F one. In spite of this, for a given flow, Class C fly ash geopolymer mortar showed a higher water requirement than Class F fly ash counterpart. The fact seems to be arisen from the angular shape of the Class C fly ash particles which cause greater internal friction.
- For a given activator composition, activator content and same curing conditions, Class F fly ash-based geopolymer mortars showed higher compressive strength than that of the Class C fly ash-based mortars. The fact most probably was arisen from the morphology of Class C fly ash particles which increased the water demand of the geopolymer mortar.
- In both Class C and F fly ashes, the highest mortar compressive strength was obtained at 1.3 Ms ratio and 10% Na_2O content. The highest average compressive strengths attained after 5 days of curing at 60°C were recorded as 57.4 and 40.2 MPa for Class F and C fly ash mixtures, respectively.
- The cement or lime substitution reduced the compressive strength of Class F fly ash-based mortars between 8.9 and 24.4%. On the contrary, in Class C fly ash mixtures, the cement replacement had a positive effect on the compressive strength and the strength increased by 46.6% upon 30% cement inclusion, while lime substitution reduced the strength slightly.
- The high-temperature resistance of Class F fly ash-based mixture was higher than that of its Class C fly ash counterpart.
- The high-temperature resistance of Class F fly ash geopolymer mortars did not changed considerably upon lime or cement substitution. On the other hand, in Class C fly ash geopolymer mortars containing either 20% lime or 20% cement the residual compressive strengths were only 6.8 and 8.2% upon exposure to 900°C, respectively.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

FINANCIAL DISCLOSURE

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PEER-REVIEW

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