

Effects on Geopolymer Mortars of The Blast-Furnace Slags Obtained from Different Regions

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Abstract – Geopolymer concrete/mortar production holds great promise for today's construction industry. In this study, microstructure and compressive and flexural strength analyses of blast furnace slags obtained from different enterprises were carried out. For this purpose, fly ash obtained from the Sivas/Kangal Thermal Power Plant and ground blast furnace slag types obtained from Bolu, and Iskenderun iron and steel industry were used as main binders in different proportions. As alkaline activators were prepared NaOH and Na₂SiO₃ solutions with three different molar ratios (8M, 10M, and 12M). The results show that the highest compressive strength was 84.7 MPa in the 95BFS1-5FA sample obtained with Bolu blast furnace slag compared to Iskenderun blast furnace slag. Besides, it was founded at 75 °C thermal curing (particularly the 8M and 95BFS5FA samples) had the highest values of flexural tensile strength (9.3 MPa), while those at underwater cured had the lowest values (6.1 MPa).

Keywords – Geopolymer Mortar, Thermal-Water Curing, Fly Ash, Blast Furnace Slag, Alkaline Activator.

I. INTRODUCTION

Geopolymers, a novel type of binding material, have been thoroughly examined by researchers as a potential replacement for conventional cement [1]. Geopolymers are a type of inorganic binder material that consists of amorphous, semi-crystalline, and three-dimensional geopolymeric structures. They could be produced at low temperatures and harden quickly [2]. In a study conducted by Phoongernkham et al., the effects of sodium hydroxide and sodium silicate activators on fly ash and blast furnace slag binders were investigated. Three different solutions using fly ash, granulated blast furnace slag, and their mixtures were prepared and tested. It was found that the 28-day compressive strength for different geopolymer samples was 42.8 MPa, 171.7 MPa, and 114.5 MPa [3]. Recently, the cement-based composite concept has been expanded to engineering-based geopolymer composites [4-8]. In another study, the low calcium F class fly ash was developed by fiber reinforced geopolymer composite and blast furnace slag. The slag was added to the fly ash at varying rates of 0%, 10%, 20% and 30% by mass. Slag mixed samples have higher initial cracking strength, higher tensile elasticity and higher ultimate tensile strength compared to samples without slag (100% fly ash-0% slag), while they have lower tensile strain capacity, lower toughness and lower stress. It is stated that it has an index. The results highlight that as more slag is used instead of fly ash, the sample becomes stronger but more brittle [5]. In another study using different geopolymer materials, standard sand, water, sodium silicate fly ash and press filter waste (PFA) were used. The 7, 28 and 56-daily compressive strengths of the samples prepared with a sand/binder ratio of 3.0 were determined

as 18.53 Mpa, 19.58 Mpa and 19.78 Mpa, respectively. It was observed that as the PFA ratio increased in the samples, there was a decrease in both unit mass values and compressive strength values [9].

The effect of different curing conditions on geopolymer structures is very important and needs to be investigated. It is emphasized that different curing conditions are very effective in terms of the mechanical and physical properties of geopolymer samples [10-13]. In addition, the mechanical properties of industrial waste-based geopolymers; It is emphasized in many studies that many features such as alkaline solution concentration, calcium content, cure temperature and age, Si/Al ratios and levels of influence from additives should be revealed [14]. Metallurgical slags, such as steel slag, copper slag, ferronickel slag, and lead-zinc slag, have varying reactivities due to their different chemical components. This diversity can affect the performance of geopolymer binders. Therefore, hybrid mortars can be used for interior and exterior coatings of buildings or to repair damaged coatings [15, 16].

The higher compressive strength found in binders with a high slag ratio in the mixture is due to the density of the C-S-H and A-S-H gel phases, as well as the microstructure. Microstructural analysis revealed that samples containing high slag had a more homogeneous and dense structure, with an amorphous structure and hydrates such as suolunite, thermonatrite, and laumontite, as well as sodium silicate hydrate with a denser crystal structure [17]. In a recent study, researchers compared bottom ash and fly ash with different activators as geopolymer binders. It was used NaOH as an alkaline activator in three different molarities (5M, 10M, and 15M) and the $\text{Na}_2\text{SiO}_3/\text{NaOH}$ mass ratio was 1.5. Upon examining the results, they found that the increase in viscosity with increasing molarity (for 15 M) made the matrix structure denser, which prevented the leaching of silica and alumina [18].

In this study, the binding properties of blast-furnace slags with different properties obtained from different plants and regions were investigated. In this direction, the effect of different physical and chemical properties on blastfurnace slag geopolymers with different binding functions in the presence of fly ash and activator was studied.

II. MATERIALS AND METHOD

This study aimed to determine the mechanical and physical properties of geopolymer mortar samples produced in different mineral contents. For this purpose, fly ash obtained from the Sivas/Kangal Thermal Power Plant and ground blast furnace slag types obtained from Bolu (BSF1), and Iskenderun (BSF2) iron and steel industry were tested as main binders in different proportions. Table 1 present some physical and chemical properties of these binders.

Table 1. Physical and chemical properties of ground blast furnace slag and fly ash

Chemical Properties	Obtained values		
	Bolu BFS	Isdemir BFS	Fly Ash
Component (%)			
SiO ₂	40.5	39.4	35.01
Al ₂ O ₃	12.8	10.6	14.2
Fe ₂ O ₃	1.11	1.5	5.42
CaO	35.5	33.8	25.75
MgO	5.81	7.1	3.12
K ₂ O	0.6	0.8	1.06
SO ₃	0.22	1.1	7.56
TiO ₂	0.71	0.63	0.84
Na ₂ O	0.7	0.5	1.21
Specific Gravity (g/cm ³)	2.91	2.90	2.72
Specific Surface (cm ² /g)	5384	4250	3320
Humidity	0.1		6.02
Insoluble residue	-	-	24.21

In this study, alkaline activators were prepared NaOH and Na₂SiO₃ solutions with different molar ratios of 8M, 10M, and 12M. The main binder consisted of different proportions as 75% by mass of blast furnace slag and 25% of fly ash (75BFS-25FA), 85% blast furnace slag and 15% fly ash (85BFS-15FA), 95% blast furnace slag and 5% fly ash (95BFS-5FA), and 100% blast furnace slag (100BFS). The ratio of alkaline activators (Na₂SiO₃/NaOH) was fixed at 2.5 for all experimental series. NaOH, in the form of solid grains, was dissolved in a beaker with predetermined amounts of water and left for a day (Fig. 1).

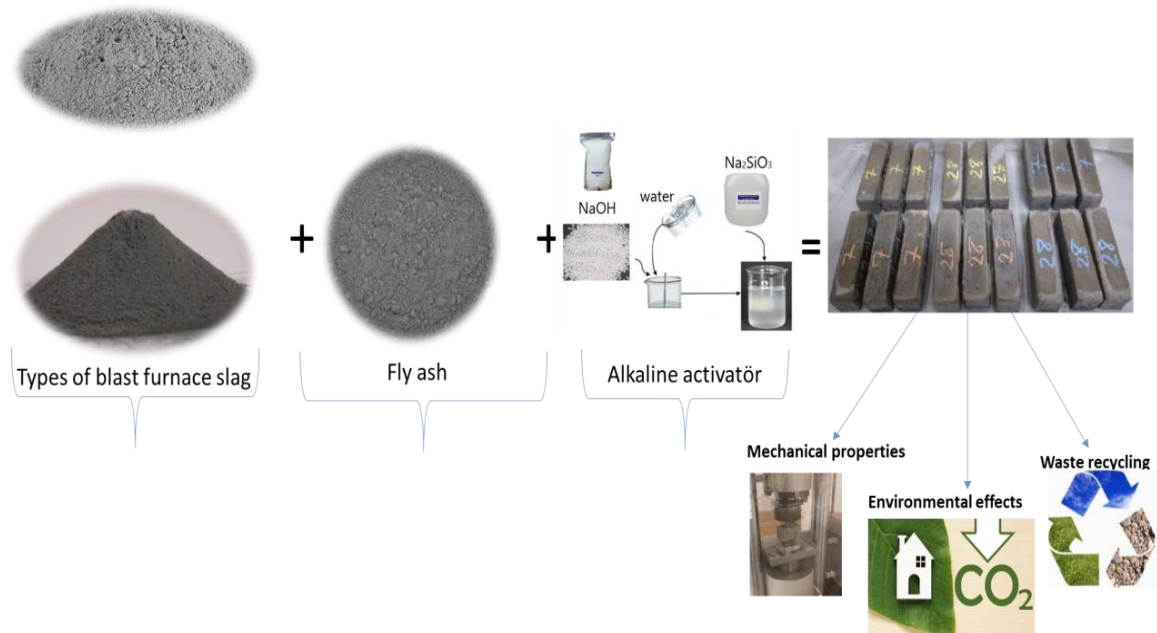


Figure 1. Geopolymer production process

The liquid/binder ratio in all geopolymer mortar samples is constant. The prepared mortars were poured in the triple moulds which dimensions are 40x40x160 mm. The first set of samples was subjected to an thermal curing at 75°C for 24 hours. The other samples were water-cured at 23±2°C. It was given the properties of this activators in Table 2 and Table 3.

Table 2. Sodium silicate physical and chemical properties [19]

Sodium silicate	Water	Solubility	Ph	Density (20 °C)	Boiling point (°C)
%35-40	%60-65	%100	11-12.4	1.3-1.5 g/cm ³	102

Table 3. Physical and chemical properties of sodium hydroxide [19]

NaOH (%)	99
Na ₂ CO ₃ (%)	0.3
SO ₄ (%)	≤0.01
Cl (%)	≤0.01
Al (%)	≤0.002
Fe (%)	≤0.002
pH	14
Density (g/cm ³)	2.13
Boiling point (°C)	1388

The samples was kept in water at $23\pm 2^{\circ}\text{C}$ and in the thermal curing at 75°C for 24 hours. The flexural tensile strength and compressive strength were applied to according to the test methods specified in the TSE EN 196-1 standard. In the experimental setup shown in Fig. 2, the flexural tensile strength test was carried out under 50 N/sec and the compressive strength test was carried out under 2400 N/sec loading. In the flexural tests the midpoint deflections at the moment of fracture were determined using a comparator.



Figure 2. Flexural tensile strength (left) and compressive strength (right) tests

III. RESULTS AND DISCUSSION

Figure 3 and Figure 4 show the flexural and compressive strengths of geopolymer specimens containing two different main binders in the presence of alkali activators at different molar ratios. However, considering both flexural and compressive strength results, higher strength of Bolu furnace slag, which is named as BFS1, was obtained. In addition, the highest compressive and flexural strengths were obtained in the samples where 8M solution was used in both furnace slags.

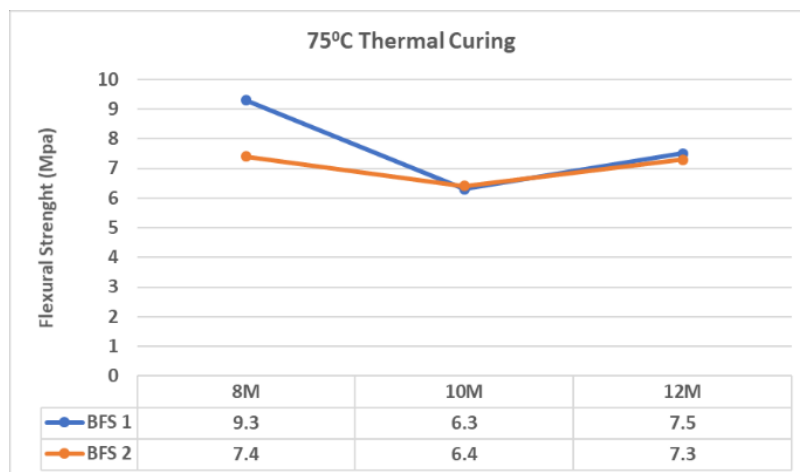


Figure 3. The mechanical properties of two different blast furnace slag

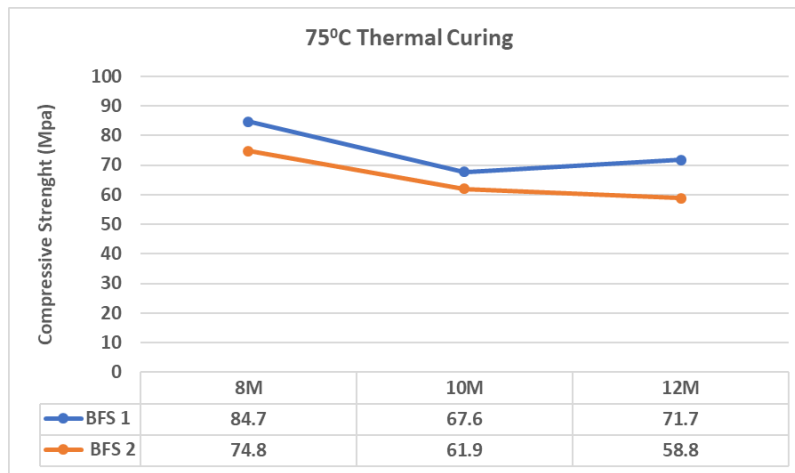


Figure 4. The mechanical properties of two different blast furnace slag

Fig. 5 and Fig. 6 shows that there are similar results for flexural tensile strength of the samples subjected to different curing conditions. The flexural tensile strength values obtained for samples containing activators at different molar ratios (8M, 10M and 12M) are shown in Fig. 5 and Fig.6. In general, looking at the flexural strength results, it could be said that the thermal cure applied to the samples kept in the thermal curing environment is more effective compared to the samples kept in water curing conditions. By increasing the reorganization of the gel with activator solubility, it is possible to reduce defects in the microstructure. The selection of activators with appropriate concentrations in molarity is very effective on the tensile strength in flexural. The results showed that the geopolymer samples at 75 °C thermal curing (particularly the 8M and 95BFS5FA samples) had the highest values of flexural tensile strength (9.3 MPa), while those at underwater cured had the lowest values (6.1 MPa). These findings suggest that temperature is a crucial factor, particularly for geopolymer samples, and that it could result in energy losses during the production phase. However, by determining the optimal temperature conditions for curing, these losses can be minimized. When comparing the curing conditions, it was observed that the 10M-95BFS5FA sample had a lower (6.3 MPa) loss of flexural tensile strength as compared to the other series. This result suggests that the impact of molarity could have both positive and negative effects on performance under thermal curing conditions.

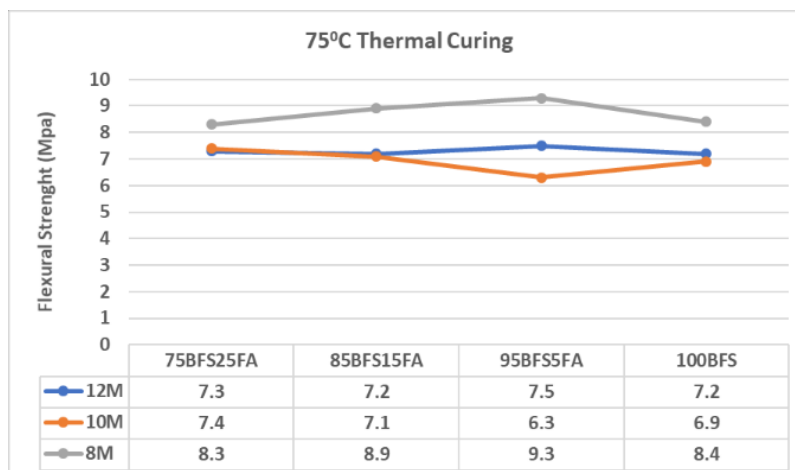


Figure 5. Flexural strength results at 8M,10M and 12M of geopolymer samples

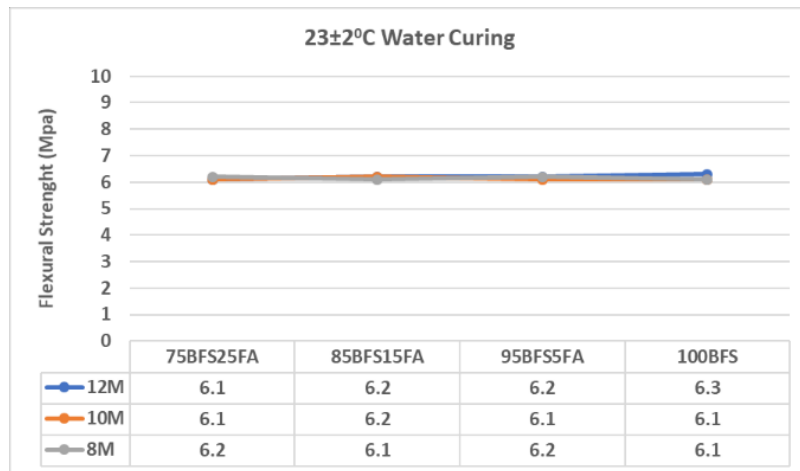


Figure 6. Flexural strength results at 8M,10M and 12M of geopolymer samples

Fig. 7 and Fig. 8 show the compressive strengths of geopolymer samples. Upon examination of the results, it is made that the lowest compressive strength was obtained by water-cured samples, while the highest strength value was achieved under thermal cure conditions. Upon comparing the overall results, it could be concluded that the highest strength (as 84.7 MPa) occurs in the 8M solution as an alkaline activator and the 95BFS1-5FA geopolymer sample. Upon examining the comparative percentages of samples subjected to distinct curing conditions, it was observed that there were more significant increases in samples utilizing 8M solutions. Conversely, an overall decline was noted as the fly ash percentage in the blend increased.

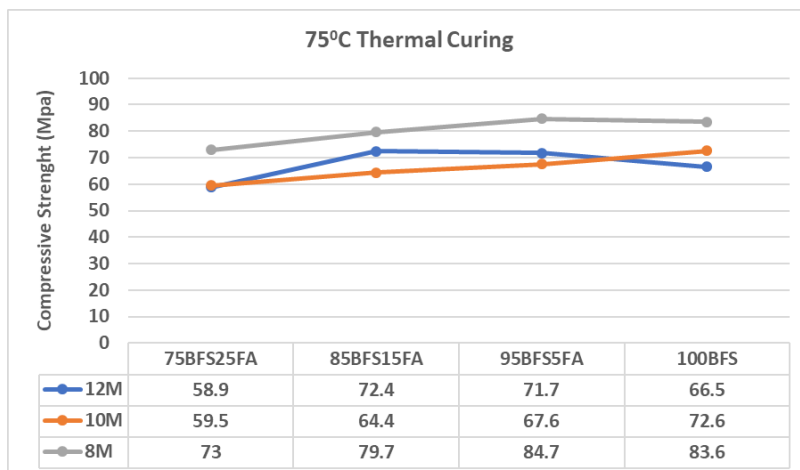


Figure 7. Compressive strength results at 8M,10M and 12M of geopolymer samples

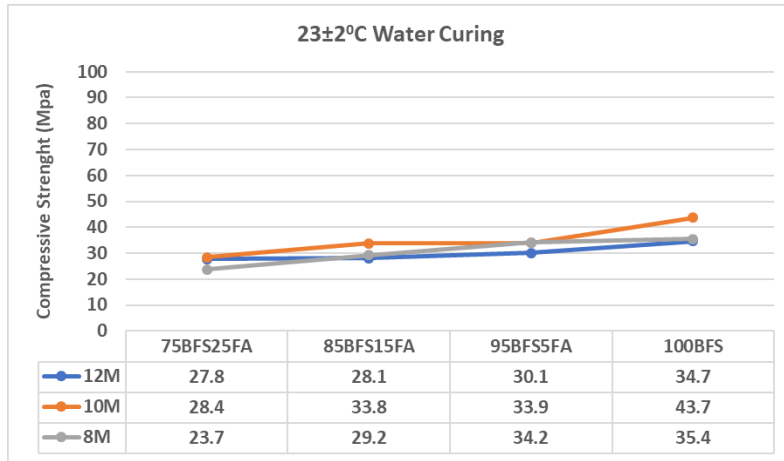


Figure 8. Compressive strength results at 8M,10M and 12M of geopolymer sample

The prevalent theory suggests that cracks and pores that may arise in microstructures due to elevated fly ash ratios in the geopolymer sample lead to the formation of C-(A)-S-H gels and a subsequent reduction in density [4,24]. On the other hand, as the molarity ratio decreases, there are generally increases in strength values. The NaOH in low molarity causes geopolymer mortars to have higher strength [25]. It has been observed that better results can be obtained, especially with fly ash-based geopolymer mortars that could be used at low rates, and that the blast furnace slag ratio and molarity are quite effective on compressive strength. When the physical properties of the blast furnace slag and fly ash used in the study were examined, it was stated that furnace slag contained higher amounts of silica and aluminum oxide than fly ash. It could be said that this situation may create more Si-O-Si and Al-O-Al bonds for the components containing slag at high amounts, which has positive effects on compressive strength [20].

In microstructural analysis, the internal structure of the geopolymer sample that gave the highest compressive strength was examined. The samples prepared for SEM/EDX analyses, approximately 1x1x1 cm in size, were kept at ambient conditions for 120-day, and then the combinations formed in the atomic structure were presented in comparison with the structure in powder form before the reaction. Fly ash and blast furnace slag are two materials commonly used in construction. Fly ash has many spherical, void, rough and smooth structures with smooth surfaces, while blast furnace slag has a flat geometry with no voids (Fig. 9-10). SEM analysis revealed that both Bolu and Iskenderun blast furnace slag have very similar geometric shapes and dimensions.

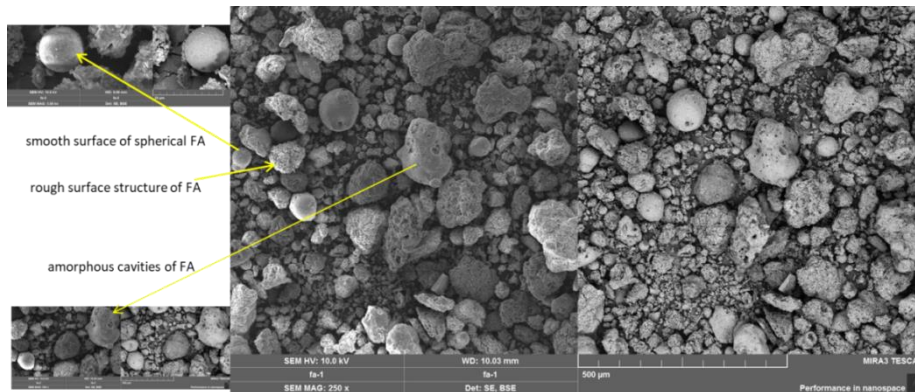


Figure 9. SEM/EDX images of the FA at 250x magnification

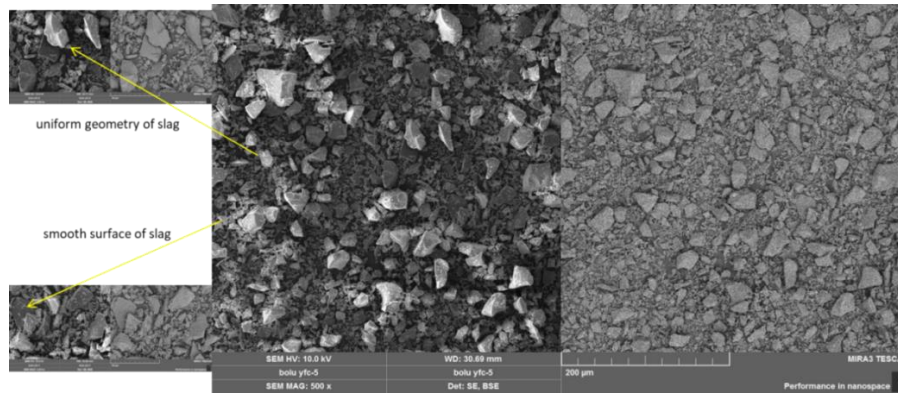


Figure 10. SEM/EDX images of the BFS at 250x magnification

Figure 11 and Figure 12 show the SEM/EDX images of geopolymer samples containing Bolu and Isdemir BFS, respectively. It could be said that unreacted slag particles, microcracks, C-S-H, and N-(C)-A-S-H gels show in the morphological images. When examining the geopolymer microstructure, it could be seen that fly ash particles are generally degraded, reacting with alkalis to form N-A-S-H gels. It could be changed in geopolymerization and formation of C-S-H gel when increasing the percentage of slag, which in turn increases the compressive strength [20]. When the surface morphology was examined, it was observed that the structure was rougher, but microcracks were less common in the sample called 95BFS1-5FA (Geopolymer mortar with Bolu BFS1) compared to the sample called 95BFS2-5FA (Geopolymer mortar with Isdemir BFS2). This situation, which is especially visible under 2000x magnification, is thought to be effective on compressive strength. When the EDX element distributions are examined, the atomic element distributions on the surfaces of the 95BFS1-5FA sample are 30.22% C, 55.70% O, 5.84% Si, 1.46% Al, 2.44% Na, 3.37% Ca and others. This distribution is as follows: 29.12% C, 58.60% O, 5.44% Si, 1.66% Al, 2.40% Na and 3.9% Ca and other elements for the 95BFS2-5FA sample.

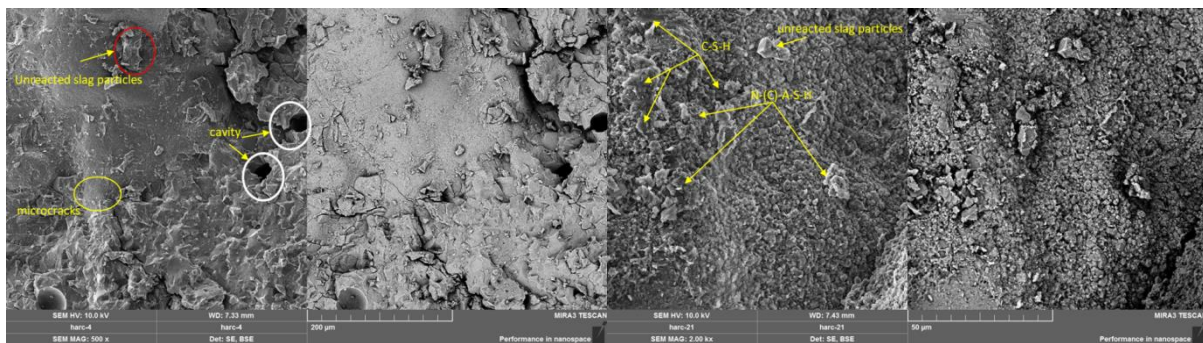


Figure 11. SEM/EDX images of geopolymer mortar (95BFS1-5FA) at different magnification

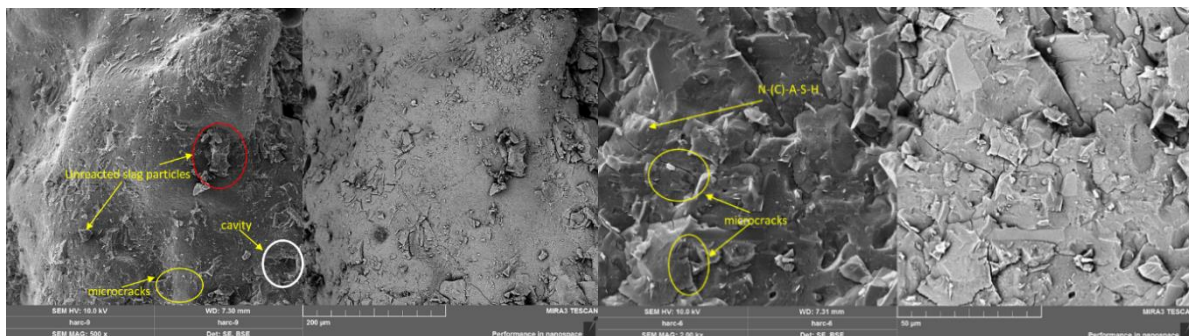


Figure 12. SEM/EDX images of geopolymer mortar (95BFS2-5FA) at different magnifications

The geopolymer properties of blast furnace slag obtained from two different plants are examined comparatively. To observe the effect of different blast furnace slag, parameters such as fly ash ratio in the mixture, $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio, and curing conditions were kept constant. In the previous stage, a new mixture was created using the mixture ratios that achieved the highest compressive strength. The 95BFS1-5FA and 95BFS2-5FA belong to BFS-containing mixtures belonging to Bolu (BSF1) and Isdemir (BSF2) plants, which contain 95% blast furnace slag + 5% fly ash. The total ratio of Silica+Aluminum+Iron oxide in Bolu BFS slag is 54.41, while this value for Isdemir slag is 51.4. It was believed that these differences in the pozzolanic activity index values could affect the binding functions (Table 1). Additionally, when it was calculated the basicity coefficient, was found to be 0.77 for Bolu BFS slag and 0.81 for Isdemir slag. Examining the specific surface areas, it thought that the surface shape factor increased the homogeneity and binding ability of the mixture. The increase in the Si/Al ratio creates strong Si-O-Si bonds, causing increases in compressive strength [21]. Both blast furnace slag have higher calcium oxide content compared to fly ash. Additionally, Bolu BFS has a slightly higher calcium oxide content compared to Isdemir BFS. In particular, the ability of calcium oxide to react with water to form calcium hydroxide accelerates the geopolymerization process, and geopolymers containing a high percentage of furnace slag generally reach higher compressive strength values [22,23].

IV. CONCLUSION

The results of the mechanical and microstructural analyses of the compressive and flexural strengths of the geopolymer specimens with different main binders are listed as follows. These are;

- Both flexural and compressive strength results were obtained, and a higher strength of Bolu furnace slag, which is named BFS1, was obtained.
- The results showed that the geopolymer samples at 75 °C thermal curing (particularly the 8M and 95BFS5FA samples) had the highest flexural tensile strength values (9.3 MPa), while those at underwater cure had the lowest values (6.1 MPa).
- Upon comparing the overall results, it could be concluded that the highest strength (84.7 MPa) occurs in the 8M solution as an alkaline activator and the 95BFS1-5FA geopolymer sample.
- When the surface morphology was examined, it was observed that the structure was rougher, but microcracks were less common in the sample called 95BFS1-5FA (Geopolymer mortar with Bolu BFS1) compared to the sample called 95BFS2-5FA (Geopolymer mortar with Isdemir BFS2).
- Bolu BFS has a slightly higher calcium oxide content compared to Isdemir BFS. It could be said that especially the ability of calcium oxide to react with water to form calcium hydroxide accelerates the geopolymerisation process and higher compressive strength values are generally achieved with geopolymers containing a high percentage of furnace slag.
- In future studies, the effect rates of different parameters on durability and strength properties could be examined by performing optimisation studies.

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