

The Effect of Silicate Modulus on the Properties of Polypropylene Fiber-reinforced Geopolymer Composite Material

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Abstract

This study investigated the effect of the silicate moduli on the properties of the geopolymer materials consisting of Class F fly ash (FA) and ground granulated blast furnace slag (GGBFS). For this purpose, geopolymer materials were prepared with alkaline solutions composed of 10 M NaOH and sodium silicate solutions (SSs) with two different silicate moduli (2Ms and 3Ms). The geopolymer mixtures were placed in molds (40 x 40 x 160 mm) and cured at 90 °C for 5 hours. After heat curing, physical, mechanical, XRD, and SEM-EDS analyses were performed on 7-day samples. According to the findings, polypropylene fiber reinforcement improved the flexural and compressive strength of the samples with high GGBFS content. Also, according to SEM-EDS analysis, it was concluded that the higher the CaO content, the lower the compressive strength, and the higher the Al₂O₃ content, the higher the compressive strength. As a result, the 2Ms SS improved the compressive strength of the samples with higher FA content, while the 3Ms SS improved the compressive strength of the samples with more GGBFS than FA. 1.5% polypropylene fiber-reinforced 3Ms SS samples with 25% FA had the highest compressive strength.

Keywords

silicate modulus, geopolymer, fiber, fly ash, GGBFS

1 Introduction

Geopolymers consist of liquid alkali activators and amorphous aluminosilicate materials in specific proportions [1]. Activators based on alkali or alkaline earth metal play an essential role in preparing geopolymers. They are generally in aqueous form and significantly increase the mixture pH, and therefore, initiate reactions rapidly. Sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) are the most popular activator solutions targeting strength and durability [2]. Alkali solutions interact with amorphous aluminosilicates and contain new binders, such as alkali metal or alkali-earth hydroxides, weak acid salts, strong acid salts, and siliceous salts. The most effective activators are NaOH and sodium silicate hydrate (water glass). Water glass-based solution improves mechanical properties the most [3].

Unlike hydration reactions, geopolymers generally require amorphous aluminosilicate precursor materials and alkaline reagents. They undergo various reactions, such as the dissolution of aluminosilicate species, the polymerization of dissolved minerals, gel precipitation, and the

final hardening of inorganic polymer-like structures [4]. The alkali solution dissolves the reactive part of the aluminosilicate powder and releases aluminium [AlO₄]⁵⁻ and silicon [SiO₄]⁴⁻ tetrahedral units into the solution. In the next few hours, neighboring ions bind via oxygen atoms and form new three-dimensional amorphous Si-O-Si or Si-O-Al bonds, resulting in water loss. Al³⁺ ions cause a negative charge balanced by cations from the raw material or the alkaline solution [5].

GGBFS is a mineral by-product used in high-performance concrete mixes [6, 7]. GGBFS improves the properties of concrete (workability, long-term strength, and durability) and significantly reduces the hydration of cement [6, 8]. It is, however, an erratic material because it has the chemical composition of glass fraction [7] and contains silica, calcium, and alumina [9]. Only 65% of GBFS can be reused (road constructions, buildings, and other industrial applications) [10]. Reinforcing Portland cement with GBFS may offer advantages in energy efficiency and reduce CO₂ emissions [6].

Fine-grained slags are activated by sodium, potassium hydroxide, carbonate, or silicate in suitable concentrations and silicate moduli. Activated slags are used in clinker-free concretes [11].

Fly ash (FA) is another industrial waste material used to produce geopolymer materials [12]. Fly ash, a supplementary cementitious material, is an abundant and ubiquitous byproduct of coal-burning power plants [12, 13]. According to ASTM C 618, Class F FA has low CaO (less than 10%) content, while Class C FA has high CaO content [13]. The reaction between cement and FA depends mainly on the chemical composition of the latter [12].

The pore structure of alkali-activated FA significantly differs from cement paste. The extended curing time under heat is good for the improvement of the pore structure of alkali-activated FA. The higher the silica and alkali content, the lower the total porosity [14]. However, it is stated that a more dense structure is formed in FA-based concretes and the resulting densities cause a decrease in porosity [15].

The main purpose of the fibers is to increase the fracture toughness by controlling the cracking of the matrix through the bridging action during the micro and macro cracking of the matrix [16]. Hence, fiber-reinforced composite materials play an important role in numerous industries and have numerous advantages over conventional materials. Fibers help improve mechanical properties, such as fracture toughness. Fibers reduce cracks and limit crack widths. They also improve the flexural strength of composites and the properties of geopolymers associated with energy absorption and deformation resistance. Short fibers improve the physical and mechanical properties of geopolymers. Inorganic (carbon or glass) fibers are the most common fibers added to geopolymer composites [17].

Civil engineering studies have extensively investigated the industrial waste materials used to manufacture geopolymers. Those studies have focused mainly on fly ash (FA) and ground granulated blast furnace slag (GGBFS) as construction or replacement materials [2]. So, in this study, FA and GGBFS based geopolymer mortars were produced. When the literature is examined, it is seen that the aluminosilicate source materials in the production of geopolymer are generally activated with a single type of sodium silicate solution. Considering that the solution that develops the best mechanical behavior is water glass-based [3], it is thought that the properties FA and GGBFS will reveal against changing silicate moduli are essential.

In addition, polypropylene fiber additions were made in the prepared geopolymer mixtures to improve the matrix's stability and strength properties, as stated in the literature.

The fact that sodium silicate solutions, which play an essential role in the activation of samples, have different silicate moduli is the main factor investigated in this study.

In this study, unlike in the literature, geopolymer materials consisting of polypropylene fiber-reinforced FA and GGBFS were prepared with alkaline solutions consisting of NaOH and sodium silicate (SS) solutions with two different silicate moduli (2Ms and 3Ms). In addition, polypropylene fiber was added to improve the matrix strength of the geopolymer materials. Also, it has evaluated the effects of the silicate moduli and polypropylene fiber on the properties of the geopolymer materials.

2 Materials and method

2.1 Materials

Fly ash and GGBFS were supplied from the Tunçbilek Thermal Power Plant (Kütahya, Turkey) and the Bolu Cement (Bolu, Turkey), respectively. XRF analysis of the materials used in the study is given in Table 1, and the XRD analysis is shown in Fig. 1 [18, 19]. Fly ash was Class F fly ash, according to ASTM C 618 [20]. Fly ash and GGBFS had a $\text{SiO}_2/\text{Al}_2\text{O}_3$ of 2.49 and 2.76, respectively (Table 1).

Alkali activators used in producing geopolymer composites are 10 M NaOH which is stated to give the best mechanical properties [1], and sodium silicate solutions (SSs). Two types of SS (2Ms and 3Ms) were used to produce the samples (Table 2). The NaOH/ Na_2SiO_3 ratio is 0.5 by weight in the alkaline solution. Twenty-four hours before, NaOH solutions were prepared and allowed to cool. Then, they were mixed with SSs before they were added to powder mixtures (FA + GGBFS). Table 2 shows their properties reported by the supplier company.

Table 1 XRF chemical analysis results for FA and GGBFS (%)

Oxide	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	SO ₃	CaO
FA	54.37	21.86	9.59	4.09	0.32	2.39	0.71	3.18
GGBFS	32.22	11.67	1.55	4.19	0.48	0.95	2.22	42.64

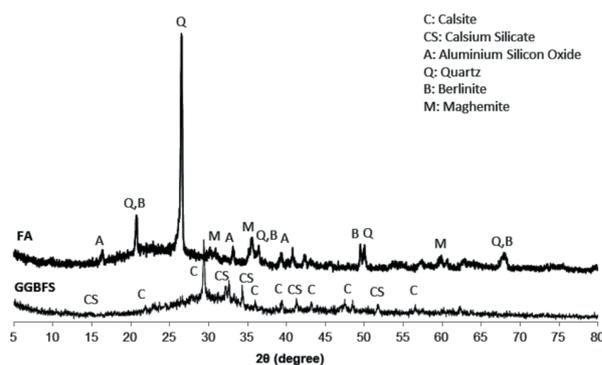


Fig. 1 XRD pattern of materials

Table 2 Properties of NaOH and SS

2Ms SS	3Ms SS	NaOH
Na ₂ O: 11.5–13.50 (%)	Na ₂ O: 7.4–9.4 (%)	M: 40 g/mol
SiO ₂ : 24.0–26.0 (%)	SiO ₂ : 24.6–28.0 (%)	NaOH ≥ 99,0 (%)
H ₂ O: 60.5–64.5 (%)	H ₂ O: 62.6–68.0 (%)	
Density: 1.38–1.42 (g/cm ³)	Density: 1.33–1.36 (g/cm ³)	

The polypropylene (PP) fibers were added to the mixtures at four different rates (0%, 0.5%, 1.0%, and 1.5% by weight) of the total binder for a homogeneous suspension, strong dispersion [21], and high strength. The polypropylene fibers were natural white, 6 mm in length, and 0.91 g/cm³ in density. They had circular-cross sections with diameters of 18–20 μm, a tensile strength of 450–700 MPa, and a modulus of elasticity of 3000–3500 MPa [22].

2.2 Methods

The geopolymer composite mixtures (Table 3) were homogeneously prepared in an automatic programmable cement mixer according to TS EN 196-1 [23]. Samples were prepared in forty different mixtures. The definitions of the samples were made as to the weight per cent of the fly ash contained in them.

The mixtures were placed in molds (40 × 40 × 160 mm) using a vibrating table. The samples were then cured in a lab-type oven at 90 °C for 5 hours and removed from the molds after 24 hours. The samples were kept in a water tank for 24 hours to determine their physical properties. In order to determine the physical properties of the relevant samples, suspended weights, surface dry water-saturated weights, and oven-dry weights were determined. Then, water absorption by weight, apparent porosity and bulk density were determined according to TS EN 772-4 [24] and TS EN 771-1 [25] standards. Flexural and compressive strength were determined according to TS EN 196-1 [23] standard. 7-day samples (three from each mixture) were used in all tests. Samples were selected for SEM-EDS and XRD analyses according to the findings obtained from the samples. XRD (with Shimadzu XRD-6000 diffractometer with a Ni filter and CuKα radiation) and areal SEM-EDS (SEM device LEO 1430 VP model) analyses were conducted at the Technology Application and Research Center of Afyon Kocatepe University (Afyonkarahisar-Turkey).

3 Results and discussion

Water absorption affects the service life of material under normal conditions. The higher the water absorption, the

Table 3 Materials and mix proportions

No	FA (%)	FA (kg)	GGBFS (kg)	PP fiber (%)	NaOH (kg)	SS (kg)	Ms SS (Modulus)
1	100	1.00	-	-	0.15	0.30	2
2	75	0.75	0.25	-	0.15	0.30	2
3	50	0.50	0.50	-	0.15	0.30	2
4	25	0.25	0.75	-	0.15	0.30	2
5	0	0	1.00	-	0.15	0.30	2
6	100	1.00	-	-	0.15	0.30	3
7	75	0.75	0.25	-	0.15	0.30	3
8	50	0.50	0.50	-	0.15	0.30	3
9	25	0.25	0.75	-	0.15	0.30	3
10	0	0	1.00	-	0.15	0.30	3
11	100	1.00	-	0.5	0.15	0.30	2
12	75	0.75	0.25	0.5	0.15	0.30	2
13	50	0.50	0.50	0.5	0.15	0.30	2
14	25	0.25	0.75	0.5	0.15	0.30	2
15	0	0	1.00	0.5	0.15	0.30	2
16	100	1.00	-	0.5	0.15	0.30	3
17	75	0.75	0.25	0.5	0.15	0.30	3
18	50	0.50	0.50	0.5	0.15	0.30	3
19	25	0.25	0.75	0.5	0.15	0.30	3
20	0	0	1.00	0.5	0.15	0.30	3
21	100	1.00	-	1.0	0.15	0.30	2
22	75	0.75	0.25	1.0	0.15	0.30	2
23	50	0.50	0.50	1.0	0.15	0.30	2
24	25	0.25	0.75	1.0	0.15	0.30	2
25	0	0	1.00	1.0	0.15	0.30	2
26	100	1.00	-	1.0	0.15	0.30	3
27	75	0.75	0.25	1.0	0.15	0.30	3
28	50	0.50	0.50	1.0	0.15	0.30	3
29	25	0.25	0.75	1.0	0.15	0.30	3
30	0	0	1.00	1.0	0.15	0.30	3
31	100	1.00	-	1.5	0.15	0.30	2
32	75	0.75	0.25	1.5	0.15	0.30	2
33	50	0.50	0.50	1.5	0.15	0.30	2
34	25	0.25	0.75	1.5	0.15	0.30	2
35	0	0	1.00	1.5	0.15	0.30	2
36	100	1.00	-	1.5	0.15	0.30	3
37	75	0.75	0.25	1.5	0.15	0.30	3
38	50	0.50	0.50	1.5	0.15	0.30	3
39	25	0.25	0.75	1.5	0.15	0.30	3
40	0	0	1.00	1.5	0.15	0.30	3

more the density and mechanical properties (flexural and compressive strength) are affected [26].

The reference geopolymers had an apparent porosity of 12.3% to 27.8% (Fig. 2) and water absorption by weight of 7.1% to 21.9% (Fig. 3). The higher the GGBFS content, the lower the apparent porosity and water absorption by weight in the samples with 75% and 50% FA. At the same time, the apparent porosity and water absorption by weight slightly increased in the samples with 25% and 0% FA in the matrix. However, the samples with 50% FA had the lowest porosity and water absorption by weight of the reference samples. At this point, the Si/Al ratio of FA (2.197) is lower than GGBFS (2.441). Therefore, FA (with a low Si/Al ratio) used at a higher rate creates larger pores in the body. For this reason, it is thought that the porosity values of the samples increased [27].

The 3Ms SS slightly increased the apparent porosity and water absorption by weight of the reference samples and the samples with 0.5% and 1.0% fiber reinforcement. Of 1.5% fiber-reinforced geopolymers, the samples with GGBFS of $\geq 50\%$ had high apparent porosity and water absorption by weight due to the 2Ms SS. The moduli did not significantly affect the apparent porosity and water absorption by weight of the reference geopolymers with 0% FA (Figs. 2 and 3).

The impact of PP fiber on apparent porosity and water absorption by weight varied from geopolymer to geopolymer. In general, when the fiber ratio increases, the open pore ratios are expected to increase, which leads to a decrease in strength at certain rates [28]. The two types of SS had almost the same effect on the apparent porosity of the samples with 50% FA. The 2Ms SS slightly increased the porosity and thus the water absorption of the 1.5% fiber-reinforced samples with increasing amounts of GGBFS in the matrix. The fiber-reinforced geopolymer mortars had an apparent porosity of 12.2% to 28.3% and water absorption by weight of 7.2% to 21.8%. Compared to the reference samples, the PP fiber had no significant effect on porosity. However, the samples with $\leq 50\%$ FA had a slightly higher water absorption by weight than the reference samples.

Density is directly related to porosity [29]. For this reason, it is expected that the porosity will decrease in the samples with increasing density. Also, GBFS is a crucial industrial by-product [10]. The lower the FA content in the mixture, the higher the bulk density in the geopolymer reference samples. The 2Ms SS geopolymers had slightly higher bulk density. The reference samples had a bulk density of 1270 kg/m³ to 1930 kg/m³ (Fig. 4).

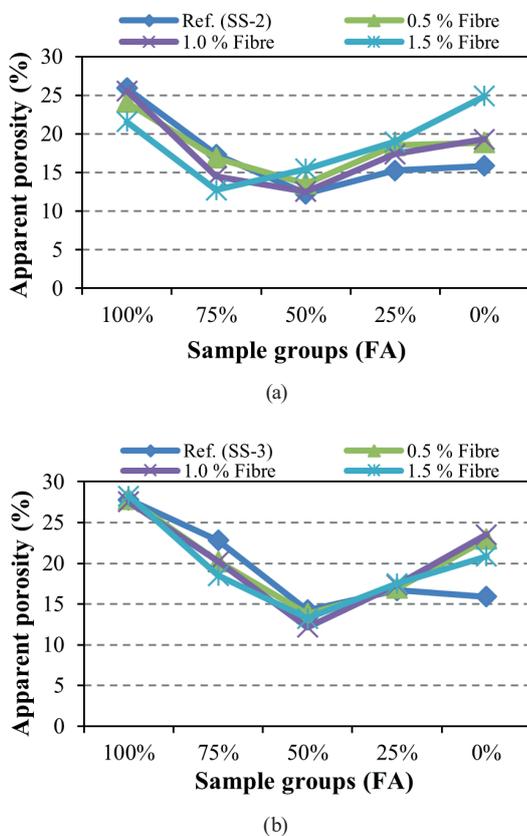


Fig. 2 Apparent porosity of geopolymer materials; a) geopolymer materials with 2Ms SS, b) geopolymer materials with 3Ms SS

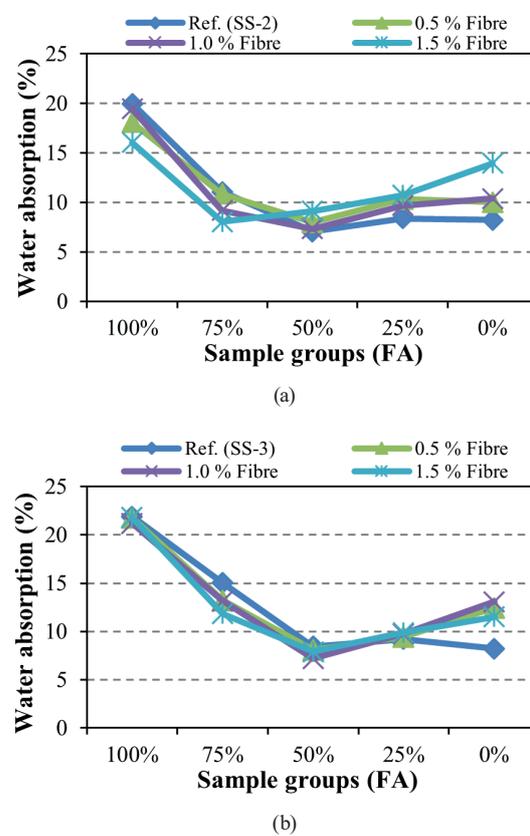


Fig. 3 Water absorption by weight of geopolymer materials; a) geopolymer materials with 2Ms SS, b) geopolymer materials with 3Ms SS

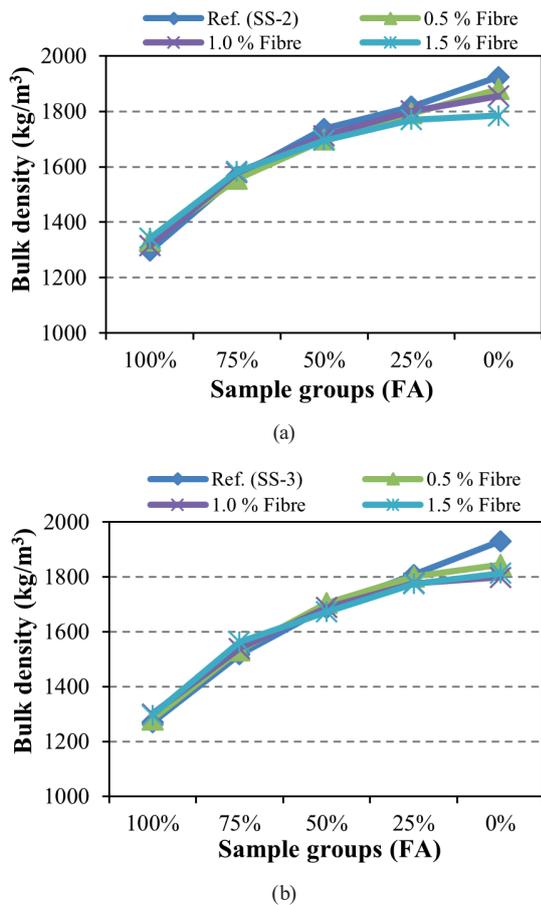


Fig. 4 Bulk density of geopolymer materials; a) geopolymer materials with 2Ms SS, b) geopolymer materials with 3Ms SS

The fiber-reinforced samples had a bulk density of 1280 kg/m³ to 1880 kg/m³. The PP fiber had a varying effect on the bulk density of the geopolymer materials. However, it is thought that the low specific gravity of PP fibers and the pores between the fiber clumps reduce the bulk density relatively in geopolymers with high FA content [21]. What is more, the SS modulus had no significant effect on the results. Bulk density increased with a decrease in FA and an increase in GGBFS. At this point, it is stated that the specific gravity of fly ash (Tunçbilek-Turkey) is 2.18-2.34 [30], and the specific gravity of GGBFS is 2.86 [31]. Therefore, the density of geopolymers increased with the increasing GGBFS contribution in the mixtures. It was determined that the density values were similar to the reference samples in the mixtures with high FA content. However, in parallel with the increase in the amount of GGBFS in the body, the density values increased (Fig. 4).

3Ms SS improved the flexural strength of the samples with high FA content. However, the flexural strength of samples became weaker due to the increased amount of GGBFS in the body. Of the fiber geopolymer composites, the samples

with 25% and 0% FA tended to have lower flexural strength. On the other hand, considering that the flexural strength increases, and the compressive strength decrease in fiber addition to conventional cement mortars [32], the flexural strengths of the samples were increased with the increase in fiber content (with 1.0% and 1.5%). The highest flexural strength values were obtained in the samples in which 50% of the FA and GGBFS materials were used (Fig. 5).

Curing temperature significantly affects flexural and compressive strength. However, increases in curing temperature lead to a continuous decrease in strength in slag mortars activated by NaOH. When sodium silicate solution and NaOH were used together, an increase in curing temperatures reduced compressive strength, but its effect on flexural strength was unclear [33]. In this study, in general, the 3Ms SS samples yielded better results. Moreover, the higher the slag and fiber content, the higher the flexural strength. Of the reference samples, geopolymers with 100% FA had the highest flexural strength values of 5.6 MPa (3Ms SS) and 4.8 MPa (2Ms SS). The fiber-reinforced geopolymers had a flexural strength of 2.4 MPa

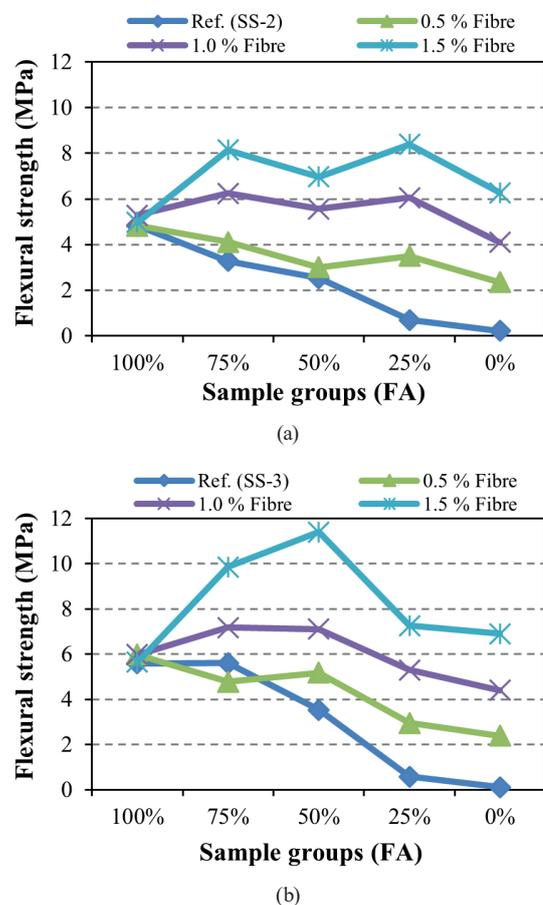


Fig. 5 Flexural strength of geopolymer materials; a) geopolymer materials with 2Ms SS, b) geopolymer materials with 3Ms SS

to 11.4 MPa (Fig. 5). It is stated that PP additions do not have a significant effect on the flexural strength in only fly ash-based geopolymers [21]. However, according to the data obtained from the prepared 100% FA samples, it was determined that PP fiber addition did not show a significant improvement in flexural strength.

The reference samples had a compressive strength of 15.3 MPa to 60.9 MPa (Fig. 6). The 3Ms SS samples with 100% GGBFS had higher compressive strength development, followed by the 3Ms SS samples with FA. The sodium silicate solution had a varying effect on the compressive strength of the fiber-reinforced geopolymer samples, depending on the FA content. The 3Ms SS samples with 25% FA had the highest compressive strength. Changes in $\text{SiO}_2/\text{Na}_2\text{O}$ ratio in solutions may change the polymerization degree of dissolved chemical species [3]. The higher the silicate ratio ($\text{SiO}_2/\text{Na}_2\text{O}$), the greater the bond between silicon atoms. However, the higher the polymerization degree [3]. Therefore, it is thought that higher strength values are obtained in the samples using high modulus sodium silicate solution.

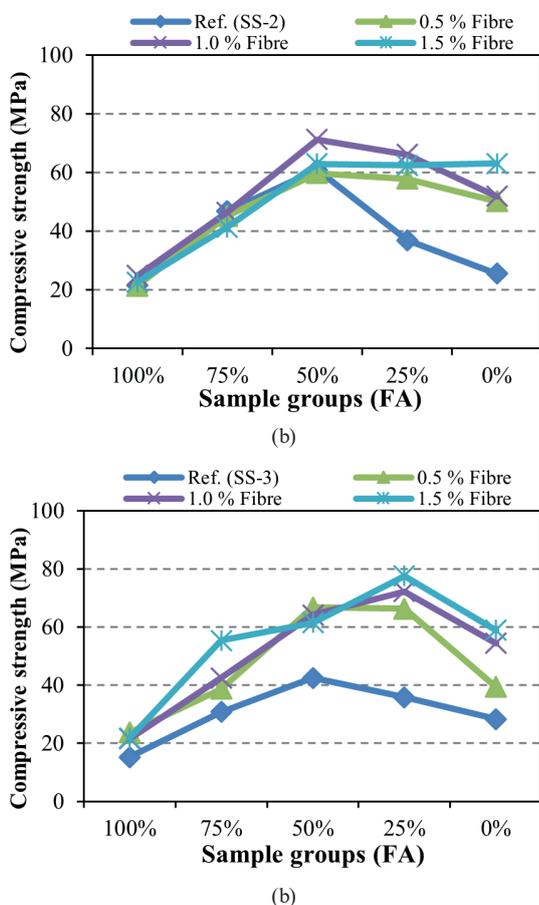


Fig. 6 Compressive strength of geopolymer materials; a) geopolymer materials with 2Ms SS, b) geopolymer materials with 3Ms SS

In this study, the fiber-reinforced geopolymers had a compressive strength of 21.4 MPa to 77.6 MPa (Fig. 6), which was higher than the strength values reported in earlier studies [34–36]. Therefore, the compressive strength improved with the increase in fiber rates. The strong interface between the fiber and the matrix is thought to be the reason for the increase in the strengths [37].

The decreases in FA content and the increases in GGBFS content up to 75% improved the compressive strength of the samples. It was reported that the higher the slag content, the higher the compressive strength in alkali-activated slag/FA mortars [38]. Under the literature and according to obtained data, GGBFS shows higher reactivity than FA. Because the self-cementing property of GGBFS is considered an effect of increasing strength values [39]. At this point, considering that no sand is used in geopolymer composites, the current compressive strength values are more meaningful. In addition, the 10M NaOH solution made a significant contribution to this result. However, it was found that the samples with 10M NaOH solutions had better mechanical properties than those with 8M NaOH solutions [3]. The samples had high compressive strength values, but research shows that FA and GGBFS-based mortars have lower compressive and flexural strength values than metakaolin-based geopolymer mortars [40].

The 2Ms SS resulted in better compressive strength in the geopolymers with high FA content, while the 3Ms SS resulted in better compressive strength in the geopolymers with more GGBFS than FA. At this point, increasing PP fibers caused significant increases in compressive strength. PP fiber improves the strength of geopolymer materials but causes discontinuities, reducing the compressive strength of conventional Portland cement mortars [32]. 1.5% PP fiber-reinforced 3Ms SS samples with 25% FA had the highest compressive strength. High strength in alkali-activated slag cement depends on the specific slag surface area, curing temperature, activator concentration, and alkali activator type [33]. Therefore, it is thought that the high GGBFS content and the type of alkali activator were the reason behind our compressive strength values.

XRD analyses were performed on the samples with 50% FA. Figs. 7 and 8 show the XRD patterns. The results showed that the samples contained similar minerals such as quartz, maghemite, aluminium-silicon oxide, and calcium phosphate. In addition, the 2Ms SS samples also contained Barium aluminium hydroxide. SEM-EDS analyses were performed on the XRD sample groups. In addition,

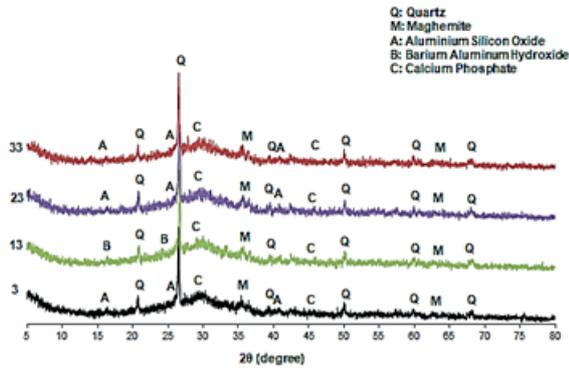


Fig. 7 XRD pattern of geopolymer materials (2Ms SS)

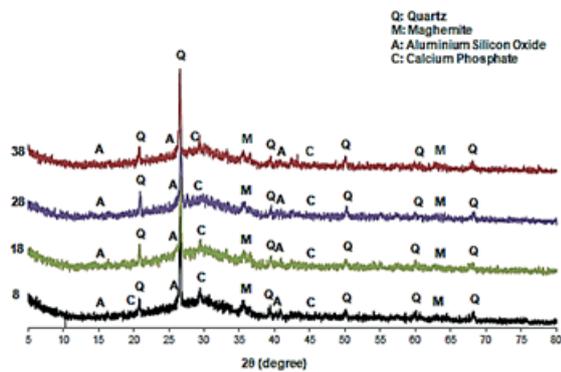


Fig. 8 XRD pattern of geopolymer materials (3Ms SS)

areal EDS analyses were performed on SEM images. The results showed that the samples mainly contained SiO_2 , CaO , Al_2O_3 , Na_2O , and Fe_2O_3 . The 2Ms SS samples had similar SiO_2 content, while 1.5% polypropylene fiber-reinforced samples mostly contained CaO (18.81%). However, the same samples had the lowest compressive strength. 1.0% polypropylene fiber-reinforced samples mostly contained Al_2O_3 (% 14.08) and Na_2O (% 11.55) (Fig. 9). They also had the highest compressive strength.

The 3Ms SS samples had similar SiO_2 content, while the reference samples contained CaO (19.85%) the most. The samples with the highest CaO content had the lowest compressive strength. 0.5% and 1.5% polypropylene fiber-reinforced samples contained Al_2O_3 (17.54%) and Na_2O (8.57%) the most, respectively. 0.5% polypropylene fiber-reinforced samples with the highest Al_2O_3 content had the highest compressive strength (Fig. 10).

According to the areal EDS analysis, the higher the CaO content, the lower the compressive strength, while the higher the Al_2O_3 content, the higher the compressive strength. Given that the samples had similar SiO_2 content, it is thought that the increase in the geopolymerization products increased the compressive strength values.

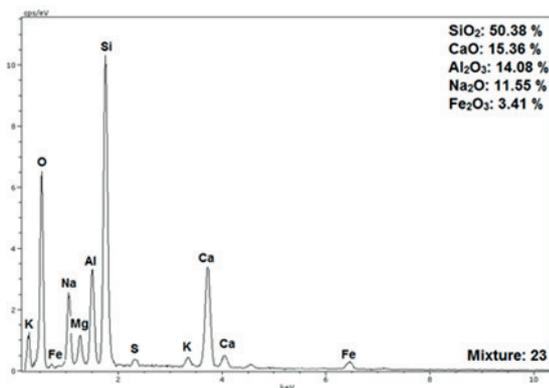
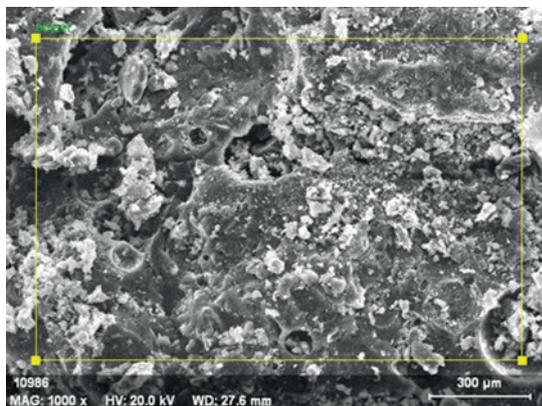


Fig. 9 SEM-EDS analysis of geopolymer materials with 2Ms SS (1.0% polypropylene fiber-reinforced samples)

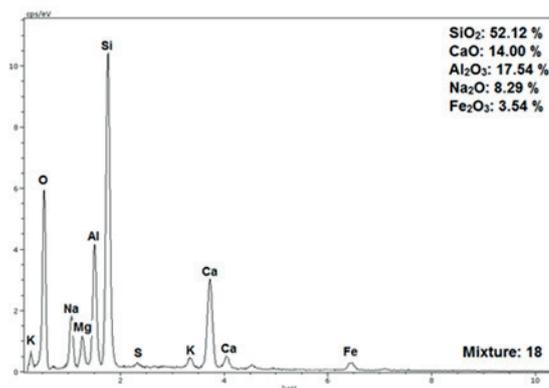
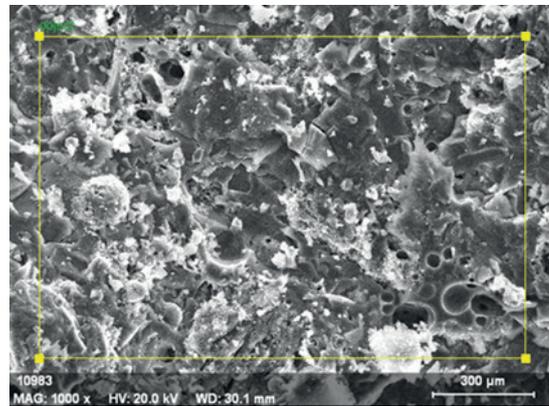


Fig. 10 SEM-EDS analysis of geopolymer materials with 3Ms SS (0.5% polypropylene fiber-reinforced samples)

4 Conclusions

In this study, geopolymer materials consisting of PP fiber-reinforced FA and GGBFS were prepared with alkaline solutions consisting of NaOH and sodium silicate solutions (SSs) with two different silicate moduli (2Ms and 3Ms). According to obtained data:

High modulus (3Ms) sodium silicate solutions used in the reference samples slightly increase the apparent porosity and water absorption. The 2Ms SS geopolymers had slightly higher bulk density.

The sodium silicate solution had a varying effect on the compressive strength of the fiber-reinforced geopolymer samples, depending on the FA content. The 3Ms SS samples had the highest compressive strength. The fibrous geopolymer composite samples were compared with the reference (non-fiber) samples, a significant increase was observed in the flexural and compressive strengths according to fiber content.

The ideal results were obtained when the mixtures prepared with 50% FA and 50% GGBFS were activated with 3Ms SS. As a result, the 2Ms SS improved the compressive strength of the samples with higher FA content, while the 3Ms SS improved the compressive strength of the samples with more GGBFS than FA. Thus, it was concluded that they could be used in structures.

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