The influence of blast furnace slag content on the mechanical and durability properties of raw perlite-based geopolymer mortars

DOI:10.36909/jer.11225

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ABSTRACT

In this work, 21 different raw perlite (RP) -based geopolymer mortars (RPGMs) were manufactured. Blast furnace slag (BFS) was replaced by RP in 7 different proportions with respect to the CaO/SiO_2 oxide ratio of RP and BFS mixture in the RPGMs. Sodium hydroxide (NaOH= 5, 10, and 15 M) was used as alkaline medium for geopolymer synthesis in the RPGMs. The ultrasound pulse velocity (U_{pv}), flexural strength (f_{fs}) and compressive strength (f_{cs}), water absorption, acid and sulfate durability of the RPGMs are investigated. The test results revealed that the BFS improved the mechanical properties of RPGMs for the low and medium alkaline medium of 5M and 10M, respectively. On the other hand, BFS had negative effect on the mechanical properties of RPGMs produced at a high alkaline medium of 15M. Moreover, the BFS improved the acid and sulfate durability of the RPGMs.

Key words: perlite; blast furnace slag; geopolymer; mechanical properties; durability.

INTRODUCTION

Geopolymers are inorganic polymers synthesized by polymerization of aluminosilicate precursors with alkaline solutions (Ranjbar et al., 2016). The solutions dissolve and release the tetrahedral units of [AlO_4] and [SiO_4], and these units were linked to the polymeric precursors by sharing oxygen atoms to form the amorphous geopolymers (Timakul et al., 2016). Any materials which are rich in amorphous Al and Si can be a potential source for geopolymer precursors (Temuujin et al., 2013; Kaya, 2021; Yurt & Emiroğlu, 2020). Based on the reaction mechanisms of the precursors, two types of bond systems can be classified (Gao et al., 2017). One is the (Si + Ca) system, the major reaction product is a C-A-S-H type gel as the main reaction product with a low Ca/Si ratio and a high Al incorporation (Brough & Atkinson, 2002). The other is the (Si + Al) system, including primarily N-A-S-H type gels within three-dimensional networks (Li et al., 2010). Due to their differences in gel characteristics and reaction mechanisms, both systems exhibit dissimilar behaviors (Gao et al., 2017). These systems can be synthesized together to achieve higher strength, better durability properties, and a promising future for the application of the geopolymers (Aydın, 2013 & Thaarrini & Ramasamy, 2016 & Praveen Kumar et al., 2019 & Çelikten et al., 2019).

Perlite is an igneous rock formed after the cooling of volcanic eruptions, which colors vary from gray to black (de Oliveira et al., 2019). Due to its high amorphous Si and Al content, raw perlite in powder form (RP) can be considered as a precursor material for low-Ca geopolymer synthesis (Çelikten & Isikdag, 2020). However, there are few studies performed on the potential geopolymer production with RP (Çelikten & Isikdag, 2020 & Taxiarchou et al., 2013 & Erdogan, 2015). Besides, these previous works focused on the mechanical properties of RP-based geopolymers and also, RP was used as the sole precursor for geopolymer production. On the other hand, the durability properties of RP-based

geopolymers and the properties of geopolymers synthesized from binary or ternary mixtures of RP and high-Ca precursors still need to be investigated.

As stated by the current studies, there is little information in the literature on RP-based geopolymers. Moreover, contrasting with previous studies that mainly concentrate on the mechanical properties of RP-based geopolymer mortars (RPGMs), the current study evaluates their durability performance in the acid or sulfate environment. In this work, RPGMs were prepared to investigate the mechanical and durability properties of the products. The blast furnace slag (BFS) was substituted by RP in seven different proportions in accordance with the total CaO/SiO_2 oxides available in the RP and BFS. The NaOH was employed for the production of RPGMs as the sole alkaline activator with three different molarities. This way, the changes in the properties of the RPGMs by the molarity of NaOH, were examined, and the role of BFS in these changes was investigated.

EXPERIMENTAL STUDY

Raw perlite in powder form (RP) from Genper Mining Industry Trade Co., Kütahya Province, Western Turkey, was employed as the main precursor material for the RP-based geopolymer mortars (RPGMs). Blast furnace slag (BFS) was provided from Karçimsa Cement Industry located in Karabük Province, Northern Turkey. The BFS was used in the RPGMs as partial replacement precursor material by the RP. The chemical compositions of the precursor materials (RP and BFS) are given in Table 1. The specific gravities of RP and BFS were 2.54 and 2.86, respectively. Besides, the blaine specific surface areas of RP and BFS were about 3850 and 4000 cm^2/g, respectively. The NaOH was in solid form with >96% purity but was used in the RPGMs as the sole alkaline activator after dissolving in water to obtain the desired molarities of 5, 10, and 15 Molar (M). Standard sand identified by CEN

(Committee of European Norms) in the EN 196-1 was used for the production of the RPGMs. The grading of the sand is given in Table 2.

Table 1 Chemical compositions of precursors.

Oxide (%)	SiO_2	CaO	Al_2O_3	Fe_2O_3	SO_3	Na_2O	K_2O	MgO	Loss of Ignition
Blast furnace slag	32.47	32.45	9.94	1.25	0.82	0.31	0.85	9.31	0.46
Perlite	71.36	0.96	13.08	0.78	0.12	3.21	5.42	0.12	2.12

Table 2 The Grading of Sand.

Sieve Size (mm)	2.0	1.6	1.0	0.50	0.16	0.08
Cumulative Percentage	0	7	32	68	87	99

A total of 21 RPGM mixtures were prepared by varying the RP, BFS, and NaOH contents. The water and sand contents were kept constant to investigate the effect of the activator and precursor (RP and BFS) contents. The proportion of water to total precursor content (RP+BFS) ratio was 1:2 and the proportion of sand to precursor content was 3:1. The molarity variations of NaOH in the mixtures were 5, 10, and 15M. The BFS was replaced by RP according to the total CaO/SiO_2 oxide ratio of these precursors. The CaO/SiO_2 ratio of the precursors was designed as the increasing rate from 0.01 (RP as sole precursor) to 0.30. The mixture proportions of RPGMs for the three-cell mortar mould are shown in Table 3. As seen on the table, the RPGM mixtures were coded as the molarity of NaOH and the CaO/SiO_2 ratio of the precursors, respectively. Besides, the mixing procedures of RPGMs involved the following steps. The NaOH solution and RP+BFS mixture were put in the mixing bowl of Hobart mixer and mixed at 140 rpm (revolutions per minute) for 30 seconds. After the first 30 seconds, the sand was poured into the bowl in 30 seconds during mixing. Then, the mixture was mixed at 280 rpm for 30 seconds. After that, the mixture was held waiting in the bowl at a non-operating state for 90 seconds. Then, the mixture was mixed at 280 rpm for 60 seconds. Finally, the mixture was cast into 4×4×16 cm molds after waiting 15 seconds in compliance with the EN 196-1 standard. Immediately after casting, the RPGMs

were subjected to heat-curing in an oven at 100 ºC without being covered for 24 hours. Twenty-four 4×4×16 cm mortar specimens were obtained from each mixture to use the mechanical and durability tests. Three RPGM specimens were employed for each test and the final test results were obtained from averaging of three values obtained from the three specimens.

Table 3 Mix proportions of the mortars

Serial Code	Mixture Code	RP (g)	BFS (g)	NaOH (g)	Sand (g)	Water (g)	CaO/SiO_2 Ratio of Precursors
5M	5M-0.01	450	0	45	1350	225	0.01
	5M-0.05	414	36	45	1350	225	0.05
	5M-0.10	369	81	45	1350	225	0.10
	5M-0.15	333	117	45	1350	225	0.15
	5M-0.20	297	153	45	1350	225	0.20
	5M-0.25	266	184	45	1350	225	0.25
	5M-0.30	239	211	45	1350	225	0.30
10M	10M-0.01	450	0	90	1350	225	0.01
	10M-0.05	414	36	90	1350	225	0.05
	10M-0.10	369	81	90	1350	225	0.10
	10M-0.15	333	117	90	1350	225	0.15
	10M-0.20	297	153	90	1350	225	0.20
	10M-0.25	266	184	90	1350	225	0.25
	10M-0.30	239	211	90	1350	225	0.30
15M	15M-0.01	450	0	135	1350	225	0.01
	15M-0.05	414	36	135	1350	225	0.05
	15M-0.10	369	81	135	1350	225	0.10
	15M-0.15	333	117	135	1350	225	0.15
	15M-0.20	297	153	135	1350	225	0.20
	15M-0.25	266	184	135	1350	225	0.25
	15M-0.30	239	211	135	1350	225	0.30

The ultrasonic pulse velocity (U_{pv}), flexural strength (f_{fs}), compressive strength (f_{cs}), and water absorption tests were performed on the RPGMs after 24 hours from the heat-curing process. The U_{pv} tests were conducted according to ASTM C597-16. The accuracy of U_{pv} test was 0.10 s. The f_{cs} and f_{fs} tests were executed with a universal testing machine in compliance with TS EN 1015-11. The f_{fs} tests were conducted on the RPGMs in center-point

(i.e. three-point) loading conditions. The semi-prisms were broken after the f_{fs} tests were used for the f_{cs} tests. The f_{cs} tests were conducted by placing 4×4 cm thin steel plates on the both top and bottom of broken specimens. The water absorption test was performed on the RPGMs according to ASTM C 642-06 standard. Besides, six specimens for each RPGM mixture were immersed in HCl solution (pH=2), 5% Na_2SO_4, and 5% $MgSO_4$ solutions for 90 and 180 days, separately. The f_{cs} test was conducted on these specimens after immersion in the solutions. Finally, the residual f_{cs} values of the RPGMs were calculated with the following equation:

$$\text{Residual } f_{cs}\ (\%) = [(f_{csi} - f_{csf}) / f_{csi}] \times 100 \quad (1)$$

Where, f_{csi}/f_{csf} are the f_{cs} of the RPGM specimens unexposed/exposed to acid or sulfate, solutions, respectively.

RESULTS AND DISCUSSION

Figure 1 and Table 4 reports the U_{pv} results of the RPGMs. The U_{pv} of RPGMs was affected by the amount of BFS and molarity of NaOH, significantly. The U_{pv} values of RPGMs made with 5M, 10M and 15M NaOH solution were in the range of 2.2–3.5 km/sec., 2.6–3.4 km/sec. and 2.6–2.9 km/sec., respectively. It was clear that an increase in NaOH content led to improved U_{pv} for RPGMs made with only RP as a precursor (CaO/SiO_2 ratio is 0.01). The observed improvement in the U_{pv} of these RPGMs by including higher NaOH can be attributed to the development a more compact microstructure and better activation of RP at high alkali concentration as reported in previous studies (Çelikten & Isikdag, 2020 & Erdogan, 2015). The BFS caused to increase the U_{pv} of RPGMs. Moreover, the BFS content became more effective as the decreasing NaOH concentration. The U_{pv} of RPGMs was enhanced up to 59%, 30%, and 7.5% by increasing CaO/SiO_2ratio of the precursors (RP and BFS) from 0.01 to 0.3 at 5M, 10M, and 15M alkaline medium, respectively. It was

previously reported that U_{pv} of mortars decreased with porosity (Mendes et al., 2020 & Lafhaj & Goueygou, 2009). Also, it was indicated that the geopolymers made with precursors having low CaO/SiO_2 ratio have more porous microstructure than the geopolymers produced with a high CaO/SiO_2 ratio (Luna Galiano et al., 2016). These results were compatible with the decrease in the U_{pv} of RPGMs as increasing BFS or CaO/SiO_2 ratio.

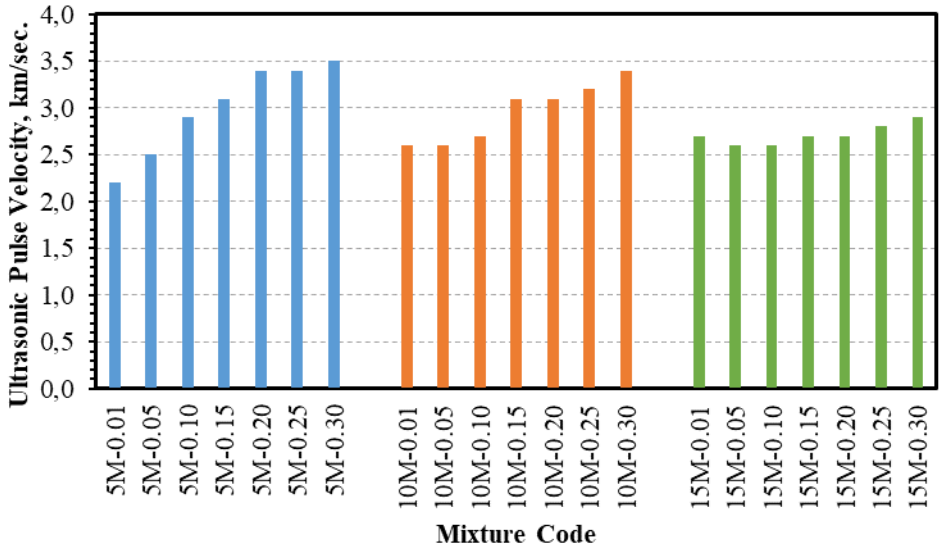

Figure 1 Ultrasonic pulse velocity results of RPGMs.

The f_{fs} results in the various RPGMs are plotted in Figure 2 and Table 4 with varying CaO/SiO_2 ratios and NaOH concentrations. From the results, it could be observed that both alkali content of NaOH solution and replacement level of BFS by RP had an important influence on the f_{fs} development of the RPGMs. The f_{fs} values of RPGMs in the series of 5M, 10M, and 15M NaOH solution were in the intervals of 2.5-3.4 MPa, 4.7-5.1 MPa, and 3.0-5.9 MPa, respectively. The f_{fs} of RPGMs enhanced as increasing molarity of NaOH from 5M to 10M, significantly. The enhancement was more distinct for the RPGMs having CaO/SiO_2ratios of 0.01 and 0.05. On the other hand, f_{fs} values of the RPGMs made with CaO/SiO_2 ratio of above 0.1 decreased as increasing the molarity of NaOH from 10M to 15M. The f_{fs} values of the 15M-0.25 and 15M-0.30 RPGMs were almost 38% lower than the

10M-0.25 and 10M-0.30 RPGMs. These results indicate that the activation of BFS was negatively influenced by the higher NaOH concentration than 10M. It can be said that the RP showed better activation in a high alkaline medium with respect to the BFS. Besides, the highest f_{fs} of 5.9 MPa was achieved for the 15M-0.01 mortars in all the RPGMs.

Table 4 Test results of the RPGMs.

Mix code	U_{pv} (km/sec.)	f_{fs} (MPa)	f_{cs} (MPa)	Water Abs. (%)	f_{cs} after 90 days HCI immersion (MPa)	f_{cs} after 180 days HCI immersion (MPa)	f_{cs} after 90 days Na_2SO_4 immersion (MPa)	f_{cs} after 180 days Na_2SO_4 immersion (MPa)	f_{cs} after 90 days $MgSO_4$ immersion (MPa)	f_{cs} after 180 days $MgSO_4$ immersion (MPa)
5M-0.01	2.2	2.5	11.6	11.3	4.0	3.6	4.2	3.7	4.1	3.4
5M-0.05	2.5	2.6	12.2	11.0	4.3	3.8	4.5	3.9	4.3	3.9
5M-0.10	2.9	3.3	15.3	10.4	5.8	4.7	6.1	5.0	5.7	4.8
5M-0.15	3.1	3.4	17.4	9.6	8.0	7.1	8.8	7.4	7.7	6.9
5M-0.20	3.4	3.4	18.8	8.8	9.3	8.4	9.5	8.7	9.2	8.4
5M-0.25	3.4	3.2	20.1	8.1	10.4	9.1	10.5	9.4	10.3	9.4
5M-0.30	3.5	3.4	22.5	7.6	11.7	10.3	12.2	10.6	11.5	10.1
10M-0.01	2.6	5.1	20.8	10.9	5.1	4.2	5.4	4.5	4.9	4.1
10M-0.05	2.6	4.8	21.3	10.7	5.0	4.1	5.8	4.8	5.1	4.3
10M-0.10	2.7	4.7	21.9	10.3	7.3	6.1	7.9	6.6	7.1	5.9
10M-0.15	3.1	4.9	22.5	9.3	7.5	6.3	10.5	8.8	8.7	7.3
10M-0.20	3.1	4.7	22.6	8.9	9.7	8.1	11.2	9.4	9.6	8.0
10M-0.25	3.2	5.0	24.3	8.3	10.3	8.6	12.6	10.5	10.9	9.1
10M-0.30	3.4	4.8	24.8	7.8	11.0	9.2	13.0	10.8	11.1	9.3
15M-0.01	2.7	5.9	23.6	10.2	5.3	4.1	5.6	4.3	5.2	4.1
15M-0.05	2.6	5.2	23.4	10.0	5.2	4.0	5.5	4.2	5.1	4.2
15M-0.10	2.6	4.7	23.0	9.8	6.4	5.3	6.7	5.6	6.3	5.3
15M-0.15	2.7	4.4	21.2	9.7	6.5	5.7	6.9	6.0	6.6	5.8
15M-0.20	2.7	3.9	20.8	9.4	6.8	6.0	7.1	6.3	7.0	5.8
15M-0.25	2.8	3.1	18.9	9.0	7.1	6.2	7.5	6.5	7.0	5.9
15M-0.30	2.9	3.0	18.3	8.7	7.6	6.1	7.9	6.4	7.7	6.1

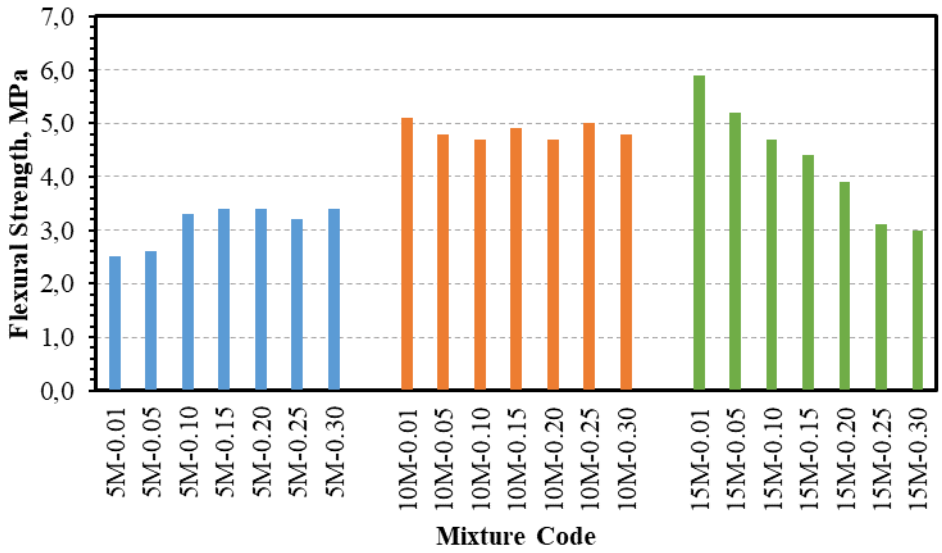

Figure 2 Flexural strength results of RPGMs.

Figure 3 and Table 4 show the f_{cs} test results of the RPGMs. The highest f_{cs} of 24.8 MPa was recorded for 10M-0.30 RPGMs. The f_{cs} values of the RPGMs made by using RP as the sole precursor (CaO/SiO_2=0.01) improved by increasing NaOH content. The f_{cs} increase was 79% and 13% in these RPGMs from 5M to 10M and from 10M to 15M, respectively. With increasing concentration of NaOH in the RPGMs, the solubilities of amorphous Si and Al in RP also increased as stated in previous works for fly ash (Çelikten & Isikdag, 2020) and calcined clay (El Hafid et al., 2017) as another low Ca precursors. The f_{cs} values of the RPGMs significantly increased by increasing the CaO/SiO_2 ratio or the BFS content for the 5M NaOH medium. On the other hand, the f_{cs} values of RPGMs decreased as increasing the BFS content at 15M alkaline medium. These results indicate that the 15M NaOH content negatively influenced the dissolution of Si and Al species from the BFS. The lower dissolution of these species can be attributed to the excessive OH^- (Part et al., 2015 & Cho et al., 2017) or excessive Na^+ (Sun et al., 2018) adsorbed on the surface of fly ash particles as stated for fly ash-based geopolymers in the previous works. The optimum alkaline concentration varies from one precursor to another due to their different solubility and degree of amorphousness. For example, in previous works, 12M NaOH was reported as appropriate

molarity for ceramic sanitaryware waste-based geopolymers (Atabey & Ozturk, 2020) and 16M for expanded perlite-based geopolymer mortars (Çelikten & Isikdag, 2020). In the present work, BFS exhibits better activation than the RP in a low alkaline medium. Additionally, the f_{fs}/ f_{cs} ratios of the RPGMs were gradually decreased as increasing BFS content. The ratio was 0.21, 0.24 and 0.25 for the 5M-0.01, 10M-0.01 and, 15M-0.01 RPGMs, respectively. On the other hand, the 5M-0.30, 10M-0.30 and, 15M-0.30 RPGMs had the f_{fs}/ f_{cs} ratios of 0.15, 0.19, and 0.16, respectively. The reason for decrease in the ratio with BFS inclusion can be attributed to the sensitivity of BFS to the heat-curing process.

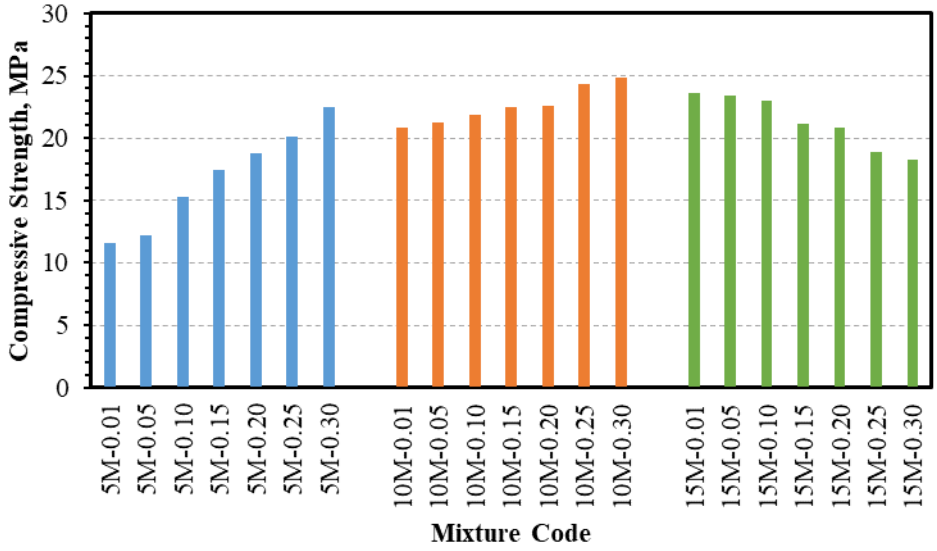

Figure 3 Compressive strength results of RPGMs.

The influence of CaO/SiO_2 ratio and NaOH content on the water absorption of RPGMs is shown in Figure 4 and Table 4. It can be seen from this figure that the water absorption capacity of RPGMs increased as increasing the CaO/SiO_2 ratio independently of the NaOH molarity. Besides, the water absorption capacity of the RPGMs which have CaO/SiO_2 ratio lower than 0.15 decreased gradually as increasing the molarity of NaOH. However, the capacity of the RPGMs designed with the CaO/SiO_2 ratios of 0.20, 0.25, and 0.30 increased as increasing NaOH molarity. These results were compatible with the U_{pv} results. These results are likely to be related to the better activation of BFS at low NaOH molarity resulting

in low pore volume for the RPGMs. In a previous work (Yusuf et al., 2014), it was reported that the increase in the Ca content by the inclusion of BFS in the low Ca geopolymer system (ultrafine palm oil fuel ash-based geopolymer was studied in the previous work) caused to increase the density of the products through pore filling effect. As stated in other previous studies, the utilization of BFS by fly ash in the low Ca geopolymer systems, gave a decrease in total porosity (Provis et al., 2012) and caused to decrease in the water absorption capacity (Sarıdemir & Çelikten, 2020). In the present work, the U_{pv} and water absorption results indicate the filling effect of BFS in the RPGMs as stated in the previous works.

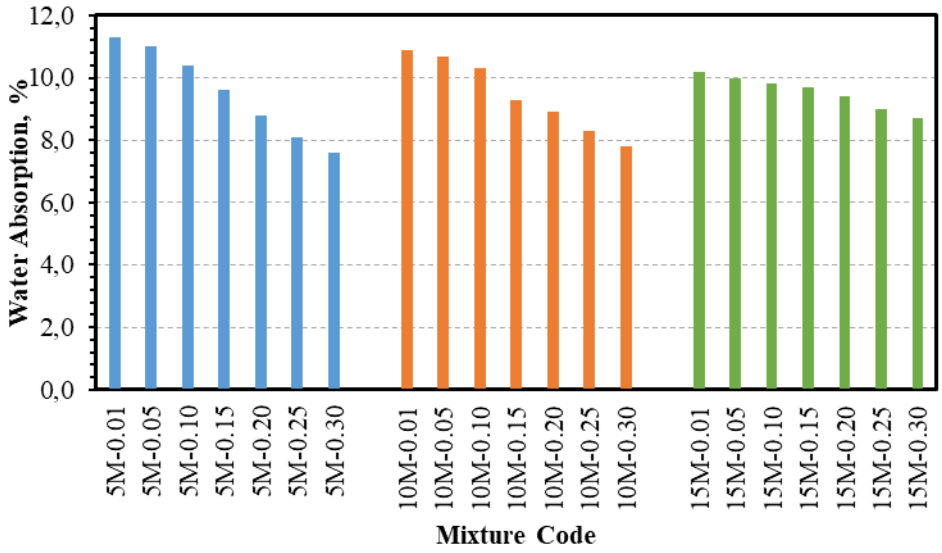

Figure 4 Water absorption results of RPGMs.

The acid resistance of the RPGMs was evaluated based on the change in f_{cs} of mortar specimens after exposure to HCl solution (pH=2) for 90 and 180 days, separately. Figure 5 and Table 4 show the residual f_{cs} and final f_{cs} results of RPGMs after immersion of the HCl solution, respectively. The residual f_{cs} of the 5M-0.01 RPGMs decreased to about 34.5% and 31% for 90 and 180 days' immersion periods, respectively. The inclusion of BFS increased the residual f_{cs} of the RPGMs. The highest residual f_{cs} values were observed on the 5M-0.30 as 52% and 45.8% after immersion for 90 and 180 days, respectively. The improved durability of the RPGMs to the acid environment with the BFS content can be attributed to

the difficult diffusion of H^+ ions to the inner structure of the mortars. The difficulty in the diffusion of the ions may be originated from the change in the pore structure of the RPGMs with BFS content as supported by U_{pv} and water absorption results. Besides, the residual f_{cs} of the RPGMs decreased as increasing the molarity of NaOH due to the lower activation of BFS in high molarities. The lowest residual f_{cs} values were observed on the 15M-0.01 and 15M-0.05 RPGMs. On the other hand, the f_{cs} values of these RPGMs were still higher than the 5M-0.01 and 5M-0.05 RPGMs after immersion to the HCl solution.

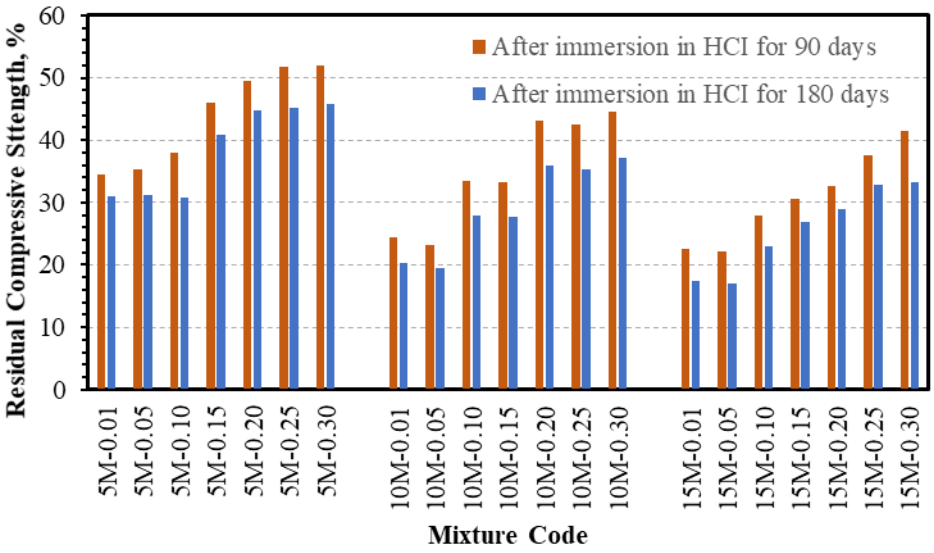

Figure 5 Residual compressive strengths of RPGMs after exposure to acid.

The residual f_{cs} and final f_{cs} values of the RPGMs after exposure to 5% Na_2SO_4 solution for 90 and 180 days are given in Figure 6 and Table 4, respectively. The residual f_{cs} values were in the ranges of 36-55%, 26-53%, and 23-43% for the RPGMs in the series of 5M, 10M, and 15M after exposure to Na_2SO_4 solution for 90 days. After immersion for 180 days, the values were decreased between 4% and 9% with respect to the proportions calculated after 90 days' immersion. The highest residual f_{cs} values were observed on the RPGMs manufactured as the CaO/SiO_2 ratio of 0.30 in all the series. The highest f_{cs} values of 13.0 and 10.8 MPa were achieved on the 10M-0.30 RPGMs after exposure to Na_2SO_4 solution for 90 and 180 days, respectively. Besides, an increase in the molarity of NaOH had a negative influence on the

residual f_{cs} of the RPGMs as reported for the results in the acid environment. The lowest residual f_{cs} of 17.9% was calculated for the 15M-0.05 RPGMs after exposure to Na_2SO_4 solution for 180 days. Additionally, the residual f_{cs} values of the RPGMs after exposure to Na_2SO_4 solution were slightly higher than the values after exposure to HCl solution.

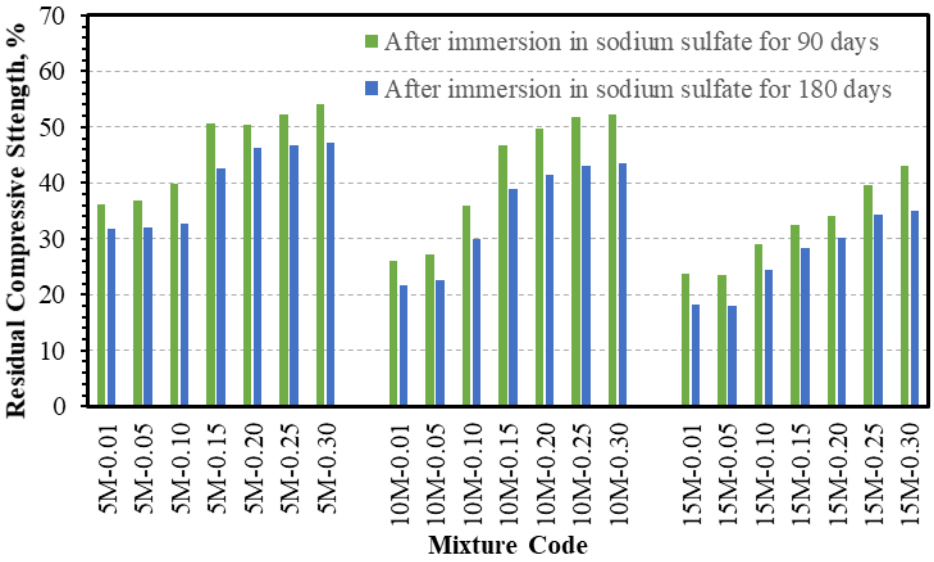

Figure 6 Residual compressive strengths of RPGMs after exposure to Na_2SO_4.

The residual f_{cs} percentages and final f_{cs} values of the RPGMs after immersion in 5% $MgSO_4$ solution for 90 and 180 days are given in Figure 7 and Table 4, respectively. While the lowest residual f_{cs} of 17.4% was seen on the 15M-0.01 RPGMs after immersion to the solution for 180 days, the highest f_{cs} of 46.8% was observed on the 5M-0.25 RPGMs at the same condition. As seen on the figure, the residual f_{cs} percentages of the RPGMs increased as increasing BFS content as observed after exposure to HCl and Na_2SO_4 solutions. The better activation of BFS at lower NaOH content also increased the residual f_{cs} percentages of the RPGMs in the series of 5M with respect to the other series. Besides, the durability test results indicate that the HCl (pH=2) and 5% $MgSO_4$ solutions had a more detrimental effect on the f_{cs} of the RPGMs than the 5% Na_2SO_4solution. The major loss in the f_{cs} of the RPGMs was noted after immersion to the acid or sulfate solutions for 90 days. The decrease in the f_{cs} values of the mortars was more limited from 90 to 180 days than the 0 to 90 days.

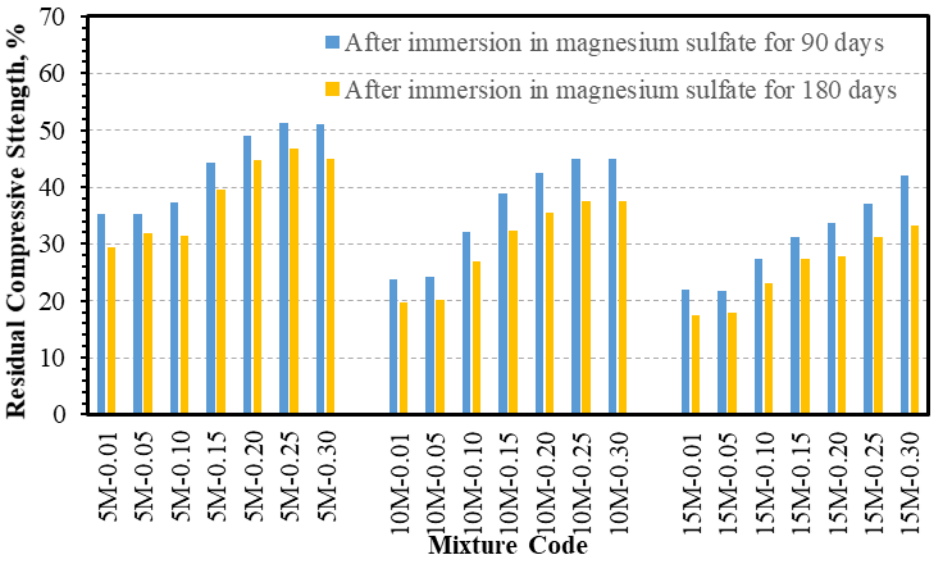

Figure 7 Residual compressive strengths of RPGMs after exposure to $MgSO_4$.

CONCLUSION

The mechanical and durability properties of blast furnace slag incorporated ground perlite-based geopolymer mortars are investigated in this study. With regard to the analysis of results obtained from this work, the following conclusions are revealed from these results:

- The optimum BFS content in the ground perlite-based geopolymer mortars is variable dependent on the molarity of NaOH. The best results in respect of mechanical and durability properties were achieved on the 10M-0.30 mortars.
- The effect of NaOH concentration changed with the precursor combination (CaO/SiO_2 ratio). The mechanical properties of ground perlite-based geopolymer mortars improved as increasing NaOH molarity. The optimum NaOH molarity was determined as 10M for the blast furnace slag incorporated ground perlite-based geopolymer mortars.
- Water absorption capacity of ground perlite-based geopolymer mortars decreased as increasing the blast furnace slag content independent from the NaOH molarity. The influence of blast furnace slag was more distinct at low NaOH content.

- The flexural strength/compressive strength ratio of the ground-perlite based geopolymer mortars decreased gradually as increasing the blast furnace slag content.
- Blast furnace slag incorporation in the ground perlite-based geopolymer mortars increased the acid and sulfate durability of these mortars, significantly.
- Magnesium sulfate and hydrochloric acid had a more detrimental influence on the strength of ground perlite-based geopolymer mortars with respect to the sodium sulfate.

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