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RESEARCH ARTICLE

EXPLORING THE POTENTIAL OF SLAG WASTE GENERATED AFTER ZINC METAL RECOVERY IN GEOPOLYMER MORTAR PRODUCTION

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Abstract

During the purification of zinc metal, the slag waste generated causes various problems such as environmental pollution and storage. It is necessary to evaluate these wastes in different areas for effective management. In this study, the effect of slag from zinc production facilities on the mechanical properties of mortars obtained by substituting slag for fly ash in geopolymers at a replacement rate of 10-50% by weight was evaluated. Mortar mixtures were subjected to various tests including workability, flexural strength, compressive strength, water-absorption, and void ratio. Mortar mixtures containing NaOH with a liquid/binder ratio 0.40 were subjected to thermal curing at 90°C for 24 hours. Flexural and compressive strength tests were conducted on 7 and 28 days of samples. As a result of the tests, it was determined that the flexural strengths of mortars produced with slag ranged from 3.0 MPa to 5.8 MPa after 28 days, while the compressive strengths ranged from 28.2 MPa to 45 MPa. Mortar mixtures containing slag achieved 48-136% higher compressive strength values than the control mixture containing fly ash (19MPa). High-temperature tests (400, 600, 800°C) revealed that mortar mixtures containing up to 30% slag achieved higher flexural and compressive strengths than the control. As the amount of slag in the mortar increased, water absorption and void ratios also increased. These results indicate that slag waste can enhance the mechanical performance of geopolymers.

Keywords

Geopolymer,
Fly ash,
Slag waste,
Mechanical properties

Time Scale of Article

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1. INTRODUCTION

Geopolymers are promising inorganic polymer materials that stand out for their high mechanical performance and durability, as well as their potential for environmental protection and energy saving. Additionally, they boast lower greenhouse gas emissions compared to Portland cement production [1]. Geopolymer rely on the dissolution of alumina and silica-containing materials obtained from industrial waste or by-products in alkaline solutions, forming of 3D polymer chains connecting Al-Si-O structures. Various industrial wastes such as metakaolin, fly ash, blast furnace slag, and waste ceramic powders are among these materials. This chemical process offers a more environmentally sustainable method for material production [2-3]. Fly ash, a significant raw material in geopolymers production, is a waste product obtained from the capture of micron-sized particles emitted into the atmosphere from thermal power plant chimneys during coal combustion, collected in ash silos through electrostatic or mechanical filters [4]. This ash is widely used in geopolymer production, and the increasing demand for its use has led to increased costs due to the distance between production sites and thermal power plants. However,

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the classification of fly ash and its expanding usability in this field has enabled its use as an alternative mineral additive, aggregate, or filler material in cement, concrete, soil improvement, and road applications. While many European countries are moving towards closing coal-fired power plants, Turkey is encouraging investments in renewable energy sources and restrictions on fly ash production are anticipated in our country's future [5-6].

Today, in the pursuit of sustainable construction materials, the use of waste powders as a fundamental material in the production of geopolymer mortar and concrete has emerged. This approach not only proves significant in terms of sustainability but also contributes to the recycling of waste generated in the relevant sector. Studies in the literature have evaluated how various types of waste, such as red mud [7-8], waste powders from brick production [9], waste from ceramic factories [5, 10-11], stone cutting waste [12], and ash-slag waste [13, 14], affect the performance of geopolymers. These evaluations highlight the potential of various post-production wastes from different sectors to contribute to the physical and mechanical properties of geopolymers.

The use of geopolymers in conjunction with waste presents an innovative approach to solving waste management issues and contributes to recycling, ensuring the efficient utilization of resources. Therefore, studies focusing on the utilization of different wastes in geopolymers hold significant potential in both industrial waste management and construction materials. In our study, we utilized waste from the Çinkur Factory (Çinkom, Kayseri) as an alternative material in place of fly ash in fly ash-based geopolymers.

The Zinc Production Factory is a recycling company that processes filter dust from iron and steel factories located in various provinces and regions of Turkey such as Iskenderun, Izmir, Osmaniye, Canakkale, and Bursa, to produce enriched zinc concentrate. This enriched zinc concentrate, which is entirely produced for export purposes and provides significant foreign currency inflow to our country, is processed as an intermediate product for use in metal production, although it is not a final product [15]. At the Çinkom Plant, zinc ore is processed in a rotary kiln with coke and limestone at temperatures between 900-1300°C to obtain zinc oxide. During this process, the zinc oxide is separated, and the residual material is removed from the furnace as slag in the form of iron calcium silicate and stored. Each year, after obtaining the desired metal compositions from the rotary kiln, granulated slag is obtained [16]. However, research on the use of zinc ore waste is quite limited. Studies by Alwaeli [17] examined the use of granulated lead-zinc slag in concrete by replacing sand at ratios of 25-50-75 and 100 percent and compared it with standard concrete. As a result of these studies, it was found that the slag exhibited higher strength in concrete samples and also attenuated gamma radiation [18]. Bayer Ozturk et al. [16] investigated the usability of the same slag in the concrete sector at certain ratios instead of aggregate and its effects on concrete performance. Similar strength results were obtained with aggregate concrete. In another sector, the effects of waste dust processed as raw and calcined pigment forms on the color performance of wall tile glazes were examined. It was found that it could be used to obtain brown tones on glazed tile surfaces without causing defects such as pinholes or cracks [19].

In a few limited studies involving the use of slag generated after zinc extraction in geopolymers, the compressive strength and toxic leaching of this slag-based geopolymer have been investigated. Geopolymer mortars produced with pure chemical reactants were used to study the solidification mechanism of heavy metals to eliminate the effects of components on the slag. Geopolymers containing ingredients such as metakaolin, cement, slag, and coal gangue were examined for their solidification behaviour by analysing their ion potential and electronegativity values [20-21].

This study aims to investigate the effect of zinc plant slag on the mechanical performance of geopolymer mortar. In this context, separate mortars were prepared with 12M NaOH at water-to-binder ratios of 0.40 and then cured at 90°C for 24 hours. The resulting samples were evaluated for physical and mechanical properties such as workability, unit weight, water absorption, void ratio, flexural strength, and compressive strength. The experiments were conducted to thoroughly analyse the impact of this slag on the performance of geopolymer mortars. By focusing on the mechanical performance of geopolymer mortar incorporating zinc plant slag, the study fills a gap in understanding how this specific material affects the strength and durability of mortar formulations. This study aims to fill the existing comprehensive research gap regarding the inclusion of zinc ore slag in geopolymers.

2. EXPERIMENTAL

2.1. The used Material Properties

Slag obtained from the Zinc production facility (Çinkom, Kayseri), Sugözü thermal power plant fly ash (Class F), sodium hydroxide as the alkali activator, water, and river sand was used to obtain geopolymer mortars. The purity of sodium hydroxide used as activator is 98.2%. The saturated dry surface density of the river sand used in the study is 2.71, and the water absorption is 1.81% (TS EN 1097-6) [22]. The purity of the sodium hydroxide used as an activator is 98.27%. Tap water was used for preparing the mortars (TS EN 1008) [23]. The slag obtained from the zinc production facility was ground in a ring mill and sieved through a 125-micron sieve before use. The chemical analysis of the slag waste and fly ash is given in Table 1. The slag contains 14.18% SiO_2, 19.32% Fe_2O_3, and 31.93% CaO [19]. The fly ash contains 55.10% SiO_2, 27.41% Al_2O_3, and 5.93% Fe_2O_3. The sieve analysis of river sand is presented in Table 2. The phase analysis (X-ray diffraction) graphs of the slag and fly ash used are presented in Figures 2 and 3. In the phase analysis of fly ash, mullite, quartz, hematite, and low-intensity CaO peaks are notable, while in the phase analysis of slag, riversiderite, wustite, and calcium iron oxide phases were obtained. The occurrence of the identified phases in the waste, which contains high amounts of iron (Fe), calcium (Ca), and relatively high amounts of silicon (Si) in its chemical composition, is an expected condition.

Figure 1. Slag waste site, size reduction, and geopolymer mortars produced with slags

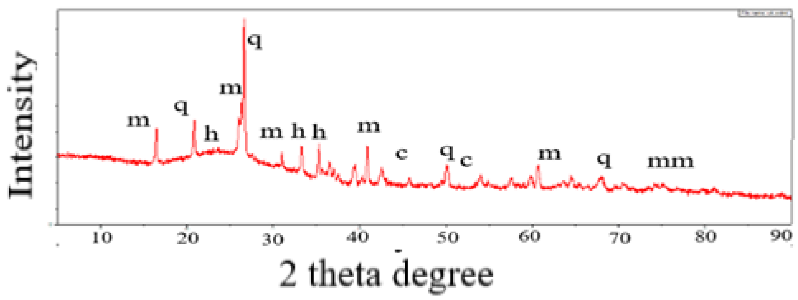

Figure 2. The XRD graph of fly ash (m: mullite, q: quartz, h: hematite, c: CaO)

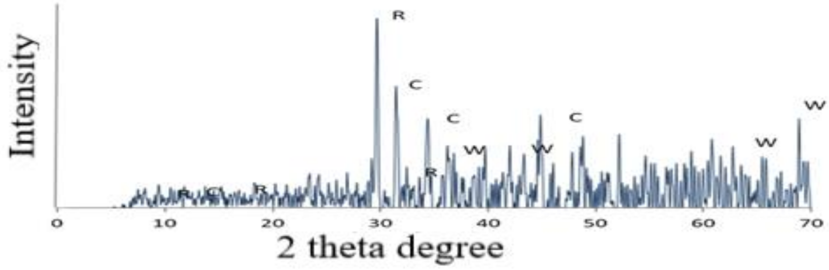

Figure 3. XRD graph of slag waste (R: riversiderite, w: wustite, c: Calcium ferro oxide) [18]

The morphology of fly ash and slag waste was investigated using scanning electron microscopy (FESEM, Zeiss Gemini 500) in Figure 4. The microstructures of the slag waste revealed the presence of irregularly shaped particles, measuring approximately 5-10 µm in size. Spherical particles ranging in size from two to five microns captured interest in the microstructure of the fly ash.

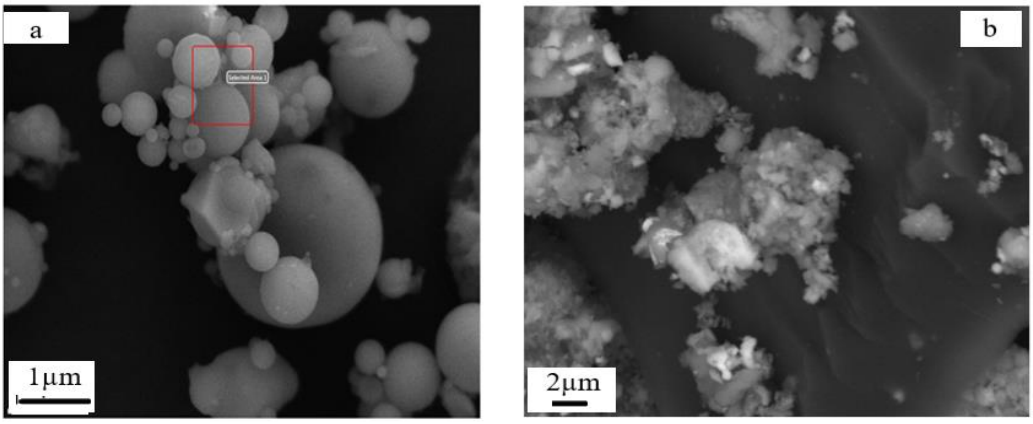

Figure 4. Secondary electron microscopy image of a) fly ash, b) slag waste

Table 1. The chemical analysis of the slag and fly ash used in the mixtures

Oxides	Slag	Fly ash
SiO_2	14.18	55.10
Al_2O_3+TiO_2	6.79	27.41
Fe_2O_3	19.32	5.92
CaO	31.93	2.44
MgO	1.54	1.99
SO_3	3.81	0.20
Na_2O	0.21	0.52
K_2O	0.68	3.44
BaO	0.07	0.08
PbO	2.60	-
ZnO	6.31	-
Loss on ignition	12.0	3.45

Table 2. The sieve analysis values of river sand.

Sieve diameter, mm	Sieved sand,%
5	100.0
4	96.0
3	88.3
2	77.3
1	54.9
0.60	26.5
0.45	24.5
0.33	10.9
0.19	3.4

2.2. Method

Fly ash was used at a rate of 450 g in single usage, while in combination with slag, it was replaced with slag at rates of 10-20-30-40-50%, resulting in weights of 405 g, 360 g, 315 g, 270 g, and 225 g, respectively. Codes were assigned to mortar mixtures based on the percentage of waste included in the mixture. For example, in the control mortar with only fly ash, labelled SM-0 (slag mortar-0), there was no slag present, whereas SM-10 indicates that 10% (45 g) of slag waste was used (Table 3). In the created mixtures, the liquid-to-binder ratio was determined as 0.40. The amount of water was added to glass jars to prepare the solution, and sodium hydroxide was added on top. The chemical was completely dissolved by shaking for approximately 1 minute. Since the reaction of sodium hydroxide with water is exothermic, it was expected to cool down to room temperature. Mortars were prepared by pouring the prepared solutions into moulds with dimensions of 40×40×160 mm according to TS EN 196-1[24].

Table 3. Mixture Ratios and codes in Mortars

Sample/	Slag Waste %	Fly ash %	Sand (g)	Water (g)	NaOH (g)
Control-SM-0	0	100	1350	180	87
SM-10	10	90	1350	180	87
SM-20	20	80	1350	180	87
SM-30	30	70	1350	180	87
SM-40	40	60	1350	180	87
SM-50	50	50	1350	180	87

After mixing the solution, ash, and slag in the mixing vessel for the first 30 seconds, river sand was added during the second 30 seconds. Then, it was mixed at high speed for 30 seconds under the standards. The mixing ceased and the mixture on the walls of the vessel was gathered in the center for the first 30 seconds, and then it was left for a total of 90 seconds. Subsequently, mixing continued at high speed for an additional 60 seconds to complete the mixing process. The mixed mortars were subjected to workability tests according to the (TS EN 1015-3) [25] standard. The samples for workability assessment were poured into a 3-part mould with dimensions of 40x40x160 mm. The pouring process was carried out in 2 stages. The poured samples were subjected to thermal curing in an oven at 90°C for 24 hours. After being removed from the oven, the mortars were subjected to flexural and compressive strength tests according to the (TS EN 1015-11) [26] standard after waiting for 7 and 28 days. The water absorption and void ratio of the samples waiting for 28 days were examined. Additionally, after the 28-day waiting period, the strengths of the mortars were examined after high-temperature application in a high-temperature furnace at 400, 600, and 800°C for 30 minutes with a temperature increase of 10°C/min.

3. RESULTS AND DISCUSSIONS

In the study, workability tests were initially applied to the freshly produced mortars, and the results are shown in Figure 5. The workability results of the samples produced with 10-40% slag waste ranged from 173-110 mm. When compared with the control sample containing 100% fly ash (225 mm), it was observed that the workability values decreased. Previous studies have indicated that factors such as specific surface area, particle shape, activator ratio, chemical composition, and reaction rate affect workability [12, 27-28]. In mortar mixtures where the activator ratio is constant, the use of slag, due to its irregular physical shapes (as shown in Figure 4) and differences in high Ca and Fe content, reduced workability compared to the smooth flow of fly ash with spherical particles.

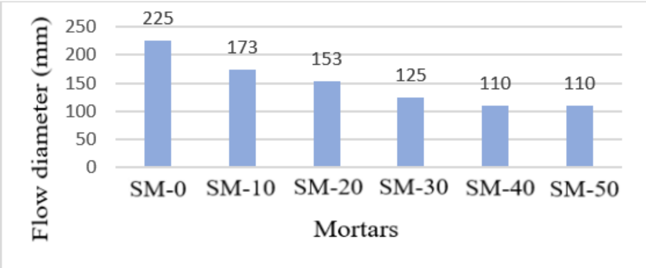

Figure 5. Workability results of mortars

The flexural strengths of the produced samples at 7 and 28 days are shown in Figure 6. The highest strength value at 7 days was 4.82 MPa in sample SM-20, and the highest strength at 28 days was 5.8 MPa in sample SM-20. The flexural strength values of samples containing 10%, 20%, and 30% slag at 28 days were 5.7 MPa, 5.8 MPa, and 3 MPa, respectively. When examining the strengths at 7 days, values of 3.79 MPa, 4.82 MPa, and 3.11 MPa were reached. The flexural strength of sample SM-50 was lower compared to the control sample at 7 and 28 days. For samples with 10-20% slag additives, the flexural strengths at 7 days were 45.21-84.67% higher than the control sample, and at 28 days, they reached 46.15-48.71% higher strength. The good bonding between the geopolymer mortar and aggregate is achieved through strong Si-O-Si interactions. While Si plays a significant role in alkali gel formation alongside Al and Ca, an increase in slag content leads to a decrease in the Si and Al content of the structure [27]. Therefore, the flexural strengths of mortars decrease when slag substitution exceeds 30% instead of fly ash.

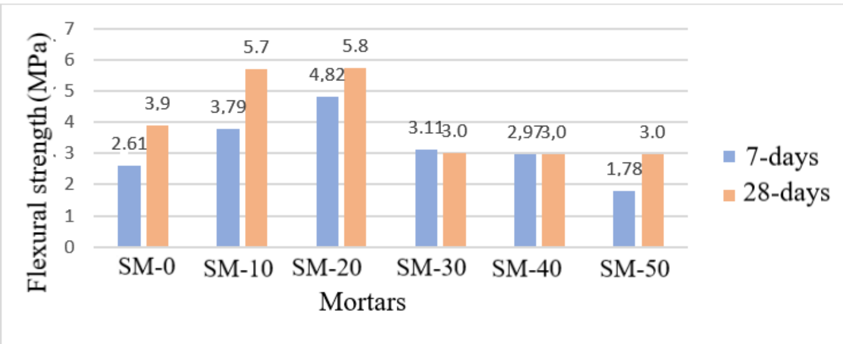

Figure 6. Flexural strength results of mortars

The compressive strength results of the mortars are shown in Figure 7. As a result of the analyses, strength increases ranging from 77-106% were observed in samples SM-10-SM-50 up to a 30% substitution rate during the 7-days period. However, a decreasing trend in strengths was observed once the 30% substitution rate was exceeded; however, in sample SM-50 (at a 50% substitution rate), a 23% increase compared to the control sample was determined. When comparing the 28-day compressive strength results with the control sample, it was found that samples SM10-SM50 reached compressive strengths 51-170% higher than the control sample. In the literature on geopolymer mortars, many studies indicate that the use of materials rich in Ca increases strength [29-33]. Additionally, an increase in the SiO_2/Al_2O_3 ratio may lead to an increase in strength by leading to Si–O–Si bonds, which are stronger than Si–O–Al bonds [28, 34-36]. In this context, the findings supporting the increase in flexural and compressive strength values of samples, particularly at substitution rates up to 30%, due to these oxides in the slag content, are supported.

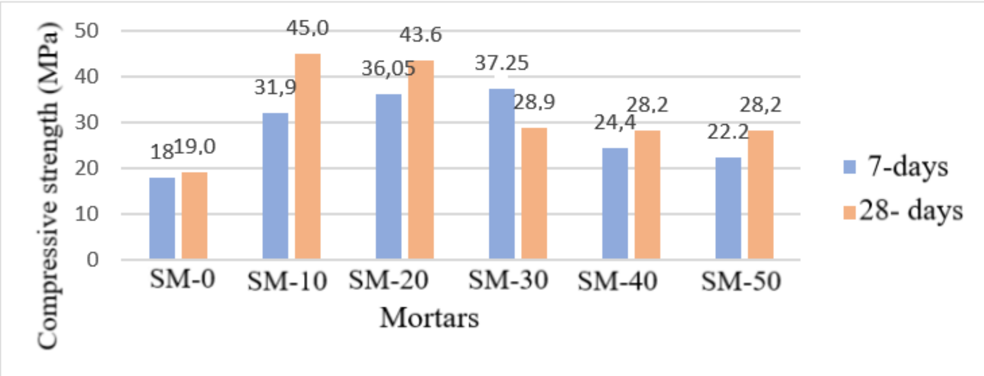

Figure 7. Compressive strength results of mortars

The strength graphs of the produced samples before and after high temperatures (400-600-800°C) are shown in Figures 8 and 9. It is observed that the strengths increased up to a 30% slag substitution rate when compared to the control sample (SM-0). It can be seen that the strength results increased at 400°C, while the strength results of the mortars subjected to 600°C and 800°C decreased.

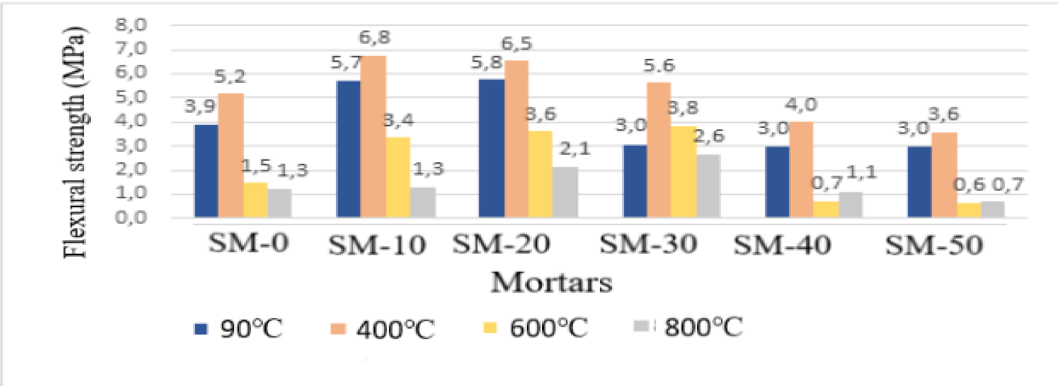

Figure 8. Flexural strength results of mortars after high temperature exposure

After the exposure to 400°C temperature, it was detected that the flexural strengths of mortars with slag additives up to 30% increased by 7.7-30.76% compared to the control sample. At the same time, it was found that the flexural strengths decreased in samples with 40% and 50% additives. After the exposure to 600°C high temperature, an increase of 126-153% was determined in flexural strengths up to a 30% substitution rate, and at 800°C, an increase of 61-100% was observed in flexural strength for 20-30% additions compared to the control sample, after which a decrease in flexural strength was observed after 30% addition.

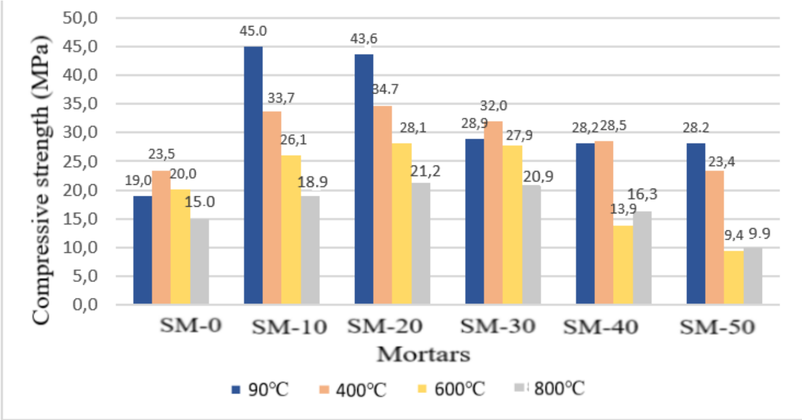

Figure 9. Compressive strength results of mortars after high temperature exposure.

When the compressive strength results of the samples after high-temperature exposure were examined, it was determined that the change in strength increased by 43.40%, 47.65%, 36%, and 21.27% as the amount of additive increased in samples exposed to 400°C temperature. The SM-50 sample was found to be the same as the control sample at 400℃ (23.5≈23.4). The increase in strength in samples containing 10-30% slag at 600°C temperature ranged between 30.5-40.5%. The increase in strength in mortars containing 10-40% slag exposed to 800°C temperature ranged between 8.6-41.33%. It was observed that as the temperature increased after the high-temperature application, the strengths decreased, but compared to the control sample, an increase in strengths was obtained for all three temperatures up to 30% slag addition. This indicates the substitutability of slag for fly ash up to 30%.

The decrease in compressive strength in mixtures can be attributed to several factors: the rise in temperatures (e.g., 600 and 800°C), reduced Si/Al content resulting from slag substitution exceeding 30%, and the formation of porosities due to reactions at elevated temperatures.

The measured water absorption and void ratio results of the mortars after 28 days are presented in Figure 10. The water absorption of mortars containing slag ranges from 3.24% to 7.63%, while the void ratio ranges from 6.39% to 14.83%. The water absorption rate of the control sample is 3.73%, while the void ratio is 7.41%. It is observed that as the percentage of slag in the mortar increases, both the void ratio and water absorption increase compared to the control mortar. It is noted that the water absorption in sample SM-10 is lower than that of the control sample. The reason for the increase in water absorption and void percentages in SM30-SM50 mortars compared to the control sample is the amount of CaO and Fe_2O_3 in the slag content. The increased presence of these components has enhanced the pozzolanic effect, altering the physical and internal structure of the mortar, thus increasing its water absorption capacity.

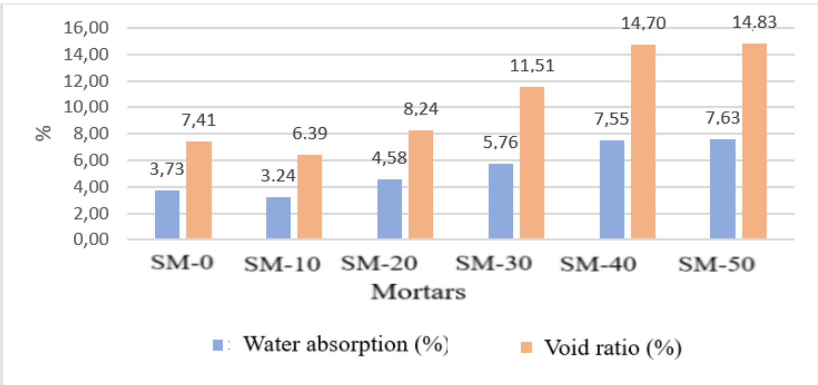

Figure 10. The water absorption and void ratio results of the mortars

4. CONCLUSIONS

This study evaluated the effect of slag waste generated after zinc recovery on the physical and mechanical properties of fly ash-based geopolymers, and reached the following conclusions:

• The workability values of produced geopolymers decreased as the slag waste content increased.
• Samples with slag substitution achieved higher flexural and compressive strength values compared to the control mix. This upward trend in strength continued up to a 30% waste addition, but decreased after the addition of 30% waste.
• It was observed that flexural strengths increased at 400 ℃ when the mortar was exposed to high temperatures. However, after exposure to 600 and 800 ℃, a downward trend in the flexural strength of the mortars was observed. Nevertheless, the strength results of mortars containing slag were higher than those of the control sample.
• The compressive strength results, which were examined for the effect of high temperatures, showed a similar trend to the flexural strength values.
• Compared to the control sample, the apparent porosity and water absorption values of mortars with slag were slightly higher.
• The use of slag in fly ash-based geopolymers could be a significant step towards reducing environmental impacts in the construction sector and producing high-strength geopolymers. This utilization can contribute to environmental sustainability by promoting the recycling of waste materials and more efficient use of resources.

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CONFLICT OF INTEREST

The author(s) stated that there are no conflicts of interest regarding the publication of this article.

CRediT AUTHOR STATEMENT

Zahide Bayer Öztürk: Supervision, Resources, Writing - original draft, Visualization, Conceptualization, **Mehmet Engür:** Resources, Investigation, Methodology, Validation.

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