

2024

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Gültekin, Adil (2024) "Effect of sealed water curing and fiber length on compressive strength and fracture energy of fly ash-based geopolymer mortars," *Journal of Sustainable Construction Materials and Technologies*: Vol. 9: Iss. 4, Article 9.

<https://doi.org/10.47481/jscmt.1607851>

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Research Article

Effect of sealed water curing and fiber length on compressive strength and fracture energy of fly ash-based geopolymers mortars

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ARTICLE INFO

Article history

Received: 05 July 2024

Revised: 13 December 2024

Accepted: 16 December 2024

Key words:

Fracture energy, geopolymer, PVA fiber, sealed curing, water curing

ABSTRACT

In this study, the effect of the curing method and polyvinyl alcohol (PVA) fiber inclusion on some engineering properties of fly ash-based geopolymer mortars was examined. In this context, six fly ash-based mortars were produced using sodium hydroxide and sodium silicate solution. The fracture energy values were determined with notched samples of 50×50×240 mm dimensions, and a clip-on gage was used to measure the crack mouth opening displacements. The notch width and notch height were 3 mm and 10 mm, respectively. Specimens were cured in hot water (80 °C) for 18 hours. Before curing, one series of samples was sealed with three layers of polyvinyl chloride (PVC) cling film and two layers of duct tape, while the other was not. The results showed that sealing the specimens during curing increased the compressive strength, and these increases were 18% for the reference mortar and 18% and 12% for mortars produced with 6 mm and 12 mm PVA fiber, respectively. Sealed curing enhanced fracture energy and peak loads and reduced the rate of capillary water absorption. With fiber inclusion, increases of up to 1508% in fracture energy values were achieved. The results revealed that sealing samples during curing significantly affects the mechanical properties.

Cite this article as: Gültekin, A. (2024). Effect of sealed water curing and fiber length on compressive strength and fracture energy of fly ash-based geopolymer mortars. *J Sustain Const Mater Technol*, 9(4), 365–373.

1. INTRODUCTION

Concrete is the most widely used building material in the world. However, the production of Portland cement consumes significant energy and emits substantial quantities of carbon dioxide. The cement industry is estimated to be responsible for approximately 8% of global carbon dioxide emissions. Alternative materials are required to reduce these drawbacks. One potential alternative to cement is geopolymers [1].

In the production of geopolymers, aluminosilicate powder materials like fly ash and slag are used with the activator solution. This activator is generally produced using sodium hydroxide, sodium silicate, potassium hydroxide,

and potassium silicates, and the resulting reaction between aluminosilicate and activator is known as geopolymerization [2]. Geopolymers offer several advantages, including significantly reducing cement-related carbon dioxide emissions, possessing high mechanical properties and good durability, providing the disposal of waste materials, exhibiting low shrinkage, and having good sulfate and corrosion resistance [3].

Although geopolymers have many advantages, these materials are brittle, similar to traditional concrete. Different types of fibers can reinforce geopolymers like fiber-reinforced cement-based concrete, and numerous studies have addressed this subject [4]. Fibers reduce brittleness and microcracking, thereby increasing the fracture tough-

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ness of geopolymer concretes. Research has been conducted studies on the use of various fiber types, such as steel, polyvinyl alcohol (PVA), glass, polypropylene, carbon, and natural fibers, in the production of fiber-reinforced geopolymers [5]. Yurt [6] examined the effect of fiber inclusion on the mechanical properties of blast furnace slag-based alkali-activated concretes and reported that flexural strengths increased with adding fibers. In a similar study, Faris et al. [7] investigated the effect of steel fiber geometry on the properties of fly ash-based fiber-reinforced geopolymer concretes. They stated a substantial increase in the flexural strength of the concretes with the addition of fibers, reaching up to 144%. The optimal fiber dosage was found to be 1% in terms of flexural strength, and it was demonstrated that hooked end fibers are particularly advantageous in enhancing flexural behavior. Wang et al. [8] reported that incorporating 0.05% basalt fiber by volume increased the peak load and fracture energy of fly ash-based geopolymer concretes by 37% and 56%, respectively. Deepa et al. [9] explored the influence of steel fiber inclusion on fly ash-based geopolymer concretes. Researchers reported that the first crack load, peak load, fracture toughness, and fracture energy values increased with fiber addition, and the facts were more pronounced with higher fiber content. It was stated that the fracture energy increased by 345% compared to the control sample produced without fiber addition when the fiber content reached 0.75%.

Fracture energy can be summarized as the required energy to create a one-unit crack area and can be determined using notched beam specimens [10]. Parameters like water/cement ratio, quantity of aggregate, aggregate size and strength [11], sample and notch geometry, and the presence of fibers [12], as well as aggregate gradation [13], were reported to affect the fracture energy of concrete significantly. Numerous studies have investigated the fracture energies of both cement-based composites and geopolymer/alkali-activated materials. Kozłowski et al. [14] investigated the effect of foaming agent dosage on the fracture energy of Portland cement-based foam composites. Tang et al. [15] examined the effect of partially replacing coarse aggregate with polystyrene on fracture energy in concrete. Celik and Bingol [16] investigated the effect of dosage of polypropylene, glass, and basalt fiber inclusion on the fracture energy of Portland cement-based self-compacting concrete. Ipek and Aksu [17] studied the effect of fiber type, length, and content on the fracture energy of cement-based SIFCON composites. Similar studies have also been conducted on geopolymer/alkali-activated materials. Ding et al. [18] investigated the effects of alkali concentration, alkali solution modulus, and liquid/binder ratio on the fracture energy of fly ash/slag-based geopolymer concretes cured under ambient conditions. Researchers reported that the fracture energy increased with the increase in alkali concentration, activator modulus, and slag amount, while the opposite was true for the liquid/binder ratio. In a similar study, Gomes et al. [19] examined the effect of steel fiber dosage on the fracture parameters of metakaolin-based geopolymer concretes cured under ambient conditions and reported significant improvements in frac-

Table 1. Chemical composition and some physical properties of fly ash

Compound	(%)	Property	Value
SiO ₂	55.9	Specific gravity	2.21
Al ₂ O ₃	23.3	Retained on 32 µm	26.7%
Fe ₂ O ₃	6.3	Retained on 45 µm	20.0%
CaO	5.3	Retained on 90 µm	6.4%
MgO	2.1		
Na ₂ O	0.6		
K ₂ O	2.3		
SO ₃	0.2		
Loss on ignition	2.0		

ture behavior with fiber addition. Liu et al. [20] conducted a study on the effect of steel fiber and silica fume inclusion on the fracture energy of ultra-high-performance geopolymer concretes and reported that the mechanical properties of these concretes were comparable to those of conventional ultra-high-performance concrete.

While various alternatives like steam and ambient curing are possible in geopolymer production, oven curing is commonly employed [21]. Heat curing can be used to achieve the desired strengths [22], and the curing temperatures are generally below 100 °C [23]. Apart from these, different methods such as microwave curing [24], solar curing [25], water curing, and saline water curing [26] are also used. However, it has been reported that alkalis in the geopolymer can leak into the curing environment during water curing, and it was stated that the mechanical properties could be negatively affected [27]. The number of studies addressing water curing in geopolymer or alkali-activated material production is limited. Moreover, most previous studies have focused on water curing at room temperature. Therefore, further research is needed to explore the effects of water curing at relatively high temperatures and the incorporation of fibers in water-cured geopolymers. This study examined the fracture energies, compressive strengths, and sorptivity properties of hot water-cured fly ash-based geopolymer composites. For this purpose, 50×50×240 mm prism specimens with a notch of 10 mm in height and 3 mm in width were used. Curing was applied in two different ways. One series of geopolymer composites was cured directly in water at 80 °C, while another was covered with (polyvinyl chloride) PVC film and duct tape before curing. This approach can prevent the leaching of alkalis from the geopolymer into the curing water.

2. MATERIALS AND METHOD

2.1. Materials

In this study, low calcium-bearing fly ash, a mixture of sodium silicate and sodium hydroxide, and CEN standard sand conforming to TS EN 196-1 [28] were used as the aluminosilicate, activator solution, and aggregate, respectively. The chemical composition and some physical properties of ash are presented in Table 1. The sodium hydroxide was in pellet form with a minimum purity of 98%, and the sodi-

um silicate solution contained 9.1% Na₂O, 28.6% SiO₂, and 62.3% H₂O. In preparing the activator solution, sodium hydroxide pellets (11.4% by weight) were dissolved in sodium silicate solution (88.6% by weight), and the resulting activator was allowed to rest for 24 hours. To reduce the setting time of mixtures, a CEM I 42.5 R type ordinary Portland cement was used. PVA fibers of 6 mm and 12 mm lengths reinforced the mortars.

2.2. Production of Samples

Mortar mixtures were prepared using a mortar mixer. CEN standard sand, fly ash, cement, activator solution, and water were sequentially placed into the mixer bowl, and the mixer was operated at 62.5 rpm for 90 seconds. Fibers were added to the bowl using the sprinkling method for approximately 30 seconds during this initial mixing stage. After 90 seconds of mixing, materials adhering to the bowl's walls were scraped off with a spoon, and mixing was continued at the same speed for another 90 seconds.

After the mixing process, the flow diameters of the mixtures were determined according to the TS EN 459-2 [29] Standard. The mortars were placed in 50×50×240 mm metal prismatic molds in two layers. Each layer was compacted with 25 jolts using a jolting table. The mortars were allowed to be set under laboratory conditions for six hours. Due to the low CaO content in the used fly ash, the setting time under laboratory conditions was significantly prolonged (approximately one week). To address this issue, portland cement was incorporated to accelerate the setting process. After the setting period, the samples were demoulded. The demoulded samples were divided into two groups. One group of mortars was tightly sealed with three layers of PVC cling film and two layers of duct tape, as shown in Figure 1, while the other group was not sealed.

The samples were cured in tap water at 80 °C for 18 hours. At the end of the curing period, the samples were removed from the water, and once the specimens cooled to room temperature, tests were conducted. The reference mortar and mortars containing short and long fibers are abbreviated as Ref, SF, and LF, respectively. Samples cured with PVC cling film and duct tape were called "Sealed" samples, while the others were designated as "Unsealed." For instance, the sample specified as SF-Sealed represents the series produced using 6 mm PVA fiber and cured after being sealed with PVC and duct tape.

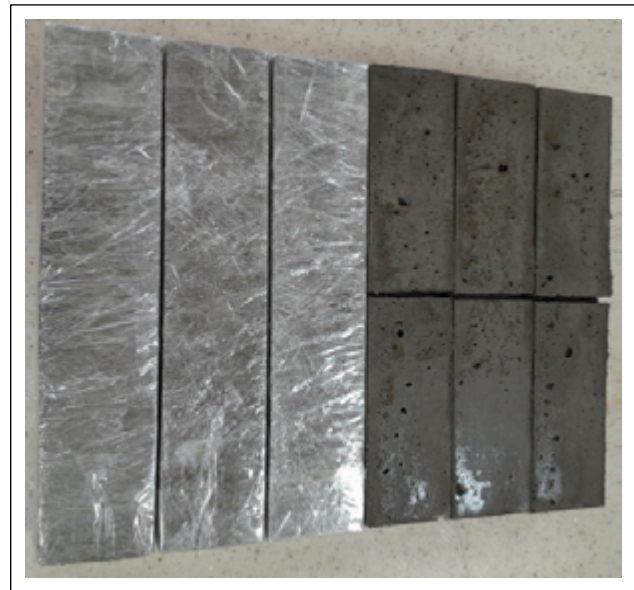


Figure 1. Sealed and unsealed specimens before water curing.

2.3. Tests

Notched samples, as shown in Figure 2, were used for the fracture energy tests. A clip-on gage was used to measure the crack mouth opening displacements (CMOD). For this purpose, two metal blades were fixed to the sample to attach the clip-on gauge. The experiment was conducted using a 3-point bending test setup. A displacement-controlled universal test device was used, and the crack opening rate was set to 0.05 mm/min. The test was automatically stopped for each sample when a 95% reduction in peak load was observed. The load-CMOD graph was plotted, and the area under the curve (W_0) was calculated. Subsequently, Equation 1, suggested by RILEM (1985) [10], was modified and used to calculate the fracture energies. Three samples were tested for each series, and the average fracture energy value was reported.

$$\text{Fracture energy} = (W_0 + mg\delta) / A_{lg} \quad (1)$$

In the equation, mg , δ , A_{lg} represent the weight of the specimen between supports, the maximum crack opening displacement, and the fracture area, respectively.

Compressive strength tests were performed by ASTM C349 [30] Standard, with some modifications using six split samples following the fracture energy tests. These tests were conducted with a 500 kN capacity concrete press. The loading rate was set at 0.9 kN/s, and a compression device with a 50×50 mm frame was used, as shown in Figure 3.

Table 2. Mixture proportions and some properties of mixtures

Mix	Ingredient (g)						Flow diameter (mm)	Fresh unit weight (kg/m³)	
	Fly ash	Cement	Alkali solution	Sand	Water	PVA fiber			
						6 mm			12 mm
Ref	405	45	213	1350	40	–	–	144	2212
SF	405	45	213	1350	40	7.2	–	129	2174
LF	405	45	213	1350	40	–	7.2	120	2161

PVA: Polyvinyl alcohol; SF: Short fiber; LF: Long fiber.

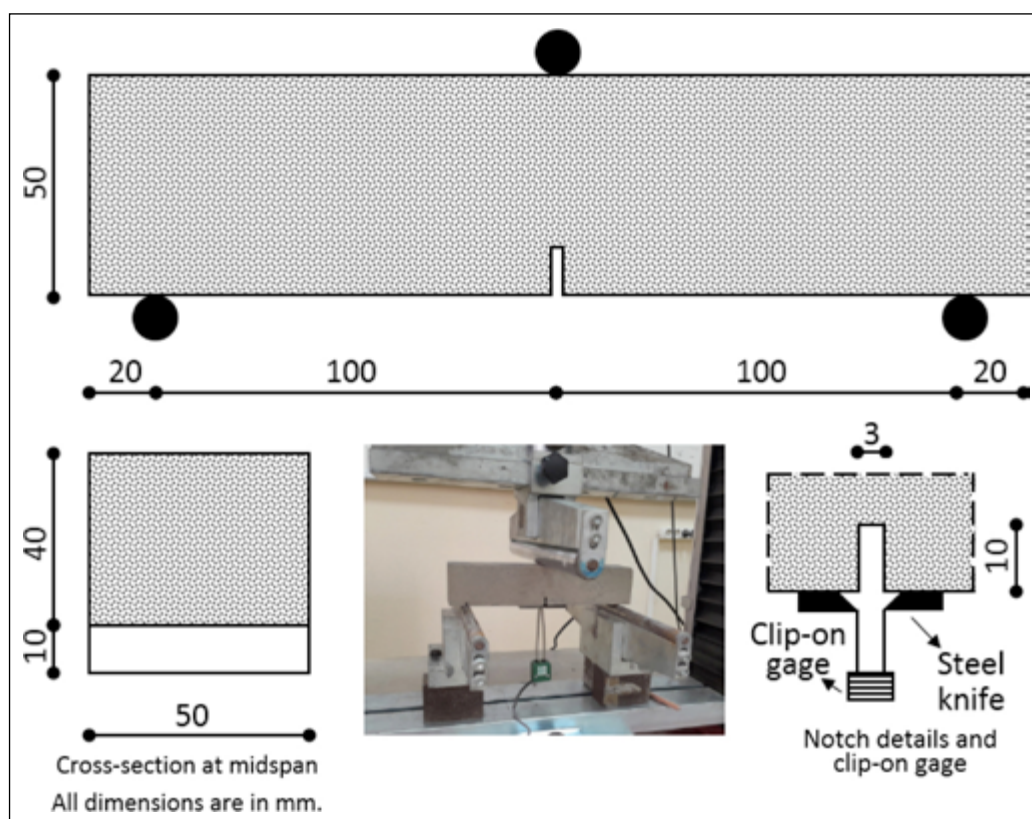


Figure 2. Geometry of specimen, notch details, test set-up, and test photograph.

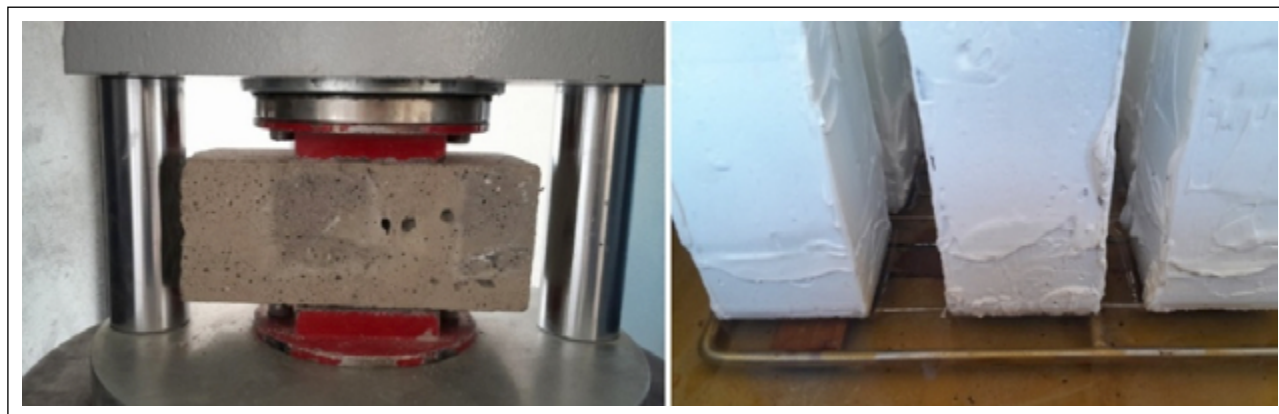


Figure 3. Compressive strength and sorptivity test photographs.

Sorptivity tests were conducted on 50×50×240 mm samples without notches, following the ASTM C1585 [31] Standard with some modifications. After the curing period, the samples were dried in an oven at 50 °C for 72 hours. Subsequently, all parts of the samples except the bottom area (50×50 mm) were covered with a waterproof material. The amount of water absorbed by the samples was measured over 6 hours. The $I-s^{0.5}$ graphs were plotted, and the capillary water absorption rates were determined using the best-fit line passing through these points.

2.4. Mixtures

The amount of ingredients, flow diameters, and fresh unit weight values of the mortars are presented in Table 2. Due to the prolonged setting time of fly ash at room tem-

perature, mixtures were prepared with 10% cement in the total powder content to expedite the setting process. The fiber content was maintained at 0.6% of the total mortar volume. It was noted that adding fiber reduced both the mortars' flow diameters and fresh unit weights.

3. RESULTS AND DISCUSSION

3.1. Compressive Strength

The compressive strengths of the mortars are presented in Figure 4. Among all series, the compressive strength of the sealed samples was higher than that of the unsealed samples. Sealing the samples with PVC cling film increased the compressive strength of the reference mixture by 18%. Similarly, these increments were 18% and 12% for the short and long

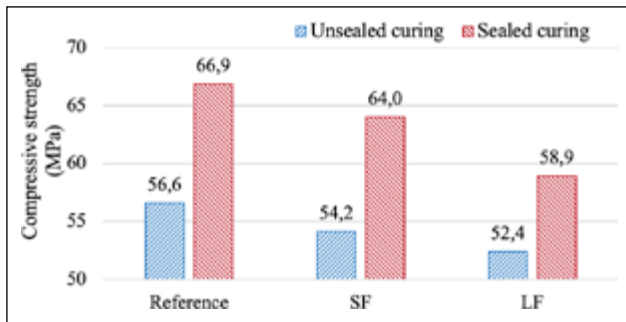


Figure 4. Compressive strength test results.

fibers including mixtures. The difference in strength between sealed and unsealed mortars prepared from the same mix suggests a greater degree of geopolymerization in the sealed samples. Despite limited prior research on sealed water curing, Giasuddin et al. [26] reported that the compressive strengths of fly ash-based geopolymers cured in tap water and two different concentrations of saline water (8% and 15%) were 48, 61 and 65 MPa, respectively. However, the compressive strength of samples sealed with silicone and plastic sheets increased to approximately 90 MPa. Researchers suggested that sealing the samples hindered possible ion transfer between water and the sample, enhancing the compressive strength. In a similar study, Kannangara et al. [32] investigated the effect of curing methods on the compressive strength of fly ash-based geopolymer pastes. Different composite series were produced using various alkaline solution/fly ash ratios and sodium silicate/sodium hydroxide ratios at 60 °C for 24 hours using a climatic test chamber. One series was covered with a polymeric film layer, while the other was left uncovered. It was found that the compressive strengths of the film-covered series were 4% to 179% higher compared to the uncovered samples. The researchers stated that the phenomenon occurred due to the initial dehydration of the matrix in the uncovered series. This dehydration led to deterioration in the matrix structure, and water loss negatively affected the dissolution and gelation processes. It was also emphasized that carbonation may had an impact on the situation.

When the effect of fiber inclusion on compressive strength is examined, it is observed that there was a slight decrease in strength with the addition of fiber. While the reductions in compressive strength for unsealed-cured short and long fiber-reinforced mortars were 4.2% and 7.4%, respectively, in sealed samples, these values were 4.3% and 12%. The negative effect of using long fibers on compressive strength is greater than that of short fiber addition. Many researchers have investigated the mechanical properties of fiber-reinforced geopolymers, and it has been proven that fiber inclusion and fiber dosage can significantly affect mechanical properties. In a similar study, Zhang et al. [33] investigated the effect of PVA fiber addition from 0.2% to 1.0% with 0.2% intervals on the mechanical properties of fly ash/metakaolin-based geopolymer concretes. Researchers reported that low fiber dosages increased the compressive strength, but the strengths gradually decreased with increasing fiber content. Lower compressive strength values were obtained at dosages of 0.8% and 1% compared to the control mixture. In

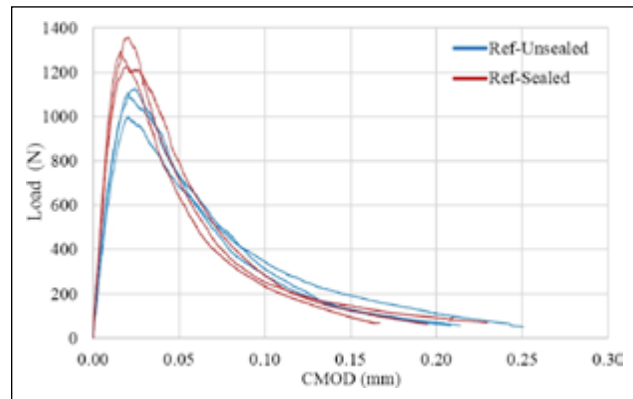


Figure 5. Load-CMOD curves of reference mortars.

CMOD: Crack mouth opening displacement.

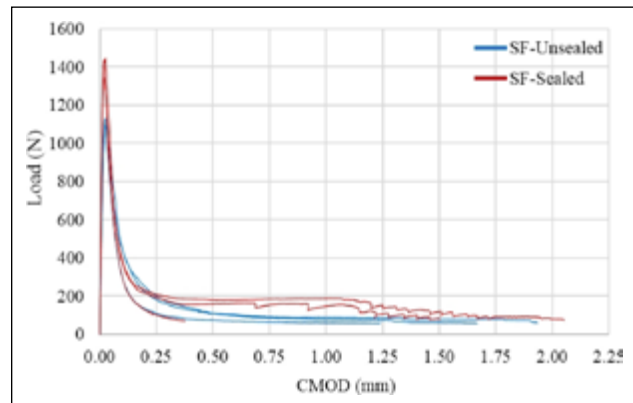


Figure 6. Load-CMOD curves of short fiber-reinforced mortars.

a similar study, Manfaluthy and Ekaputri [34] reported that the addition of 0.3%, 0.5%, and 0.8% PVA fiber by volume increased the compressive strength of fly ash-based geopolymer concrete by 2.4%, 3.7%, and 9.8%, respectively. Zhang et al. [35] stated that using PVA fiber at an appropriate dosage in geopolymer composites increases strength by providing crack control. Still, the use of an inappropriate dosage has a negative effect on the strength by creating pores within the structure. Sukontasukkul et al. [36] highlighted the impact of fiber dosage. They emphasized that using 1% polypropylene fiber in fly ash and silica fume-based geopolymer mortars reduces the compressive strength by 14%.

3.2. Fracture Energy

The load-CMOD curves of the three samples from each series after fracture energy tests are presented in Figure 5–7. As expected, the peak load was reached shortly after loading began in all series. After the peak load, the load-carrying capacity of all samples decreased. Due to the absence of fiber reinforcement in the reference samples, the test was finished at low crack openings. However, the ultimate CMOD values were significantly higher in the fiber-reinforced series than in the reference samples. The fracture behavior was more ductile when short fibers were used, and the toughness value increased significantly. With the use of long fibers, these values increased further. Figure 8 illustrates the bridging ability of the fibers.

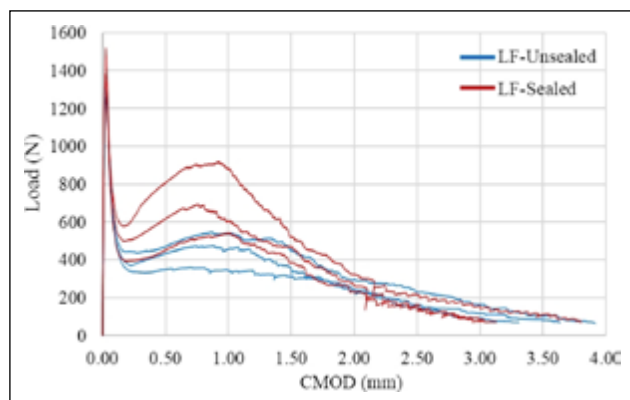


Figure 7. Load-CMOD curves of long fiber-reinforced mortars.

The peak loads and fracture energy values of the mortars are presented in Figure 9 and Figure 10, respectively. The average peak loads of sealed specimens, regardless of fiber inclusion or fiber length, were higher than those of the unsealed series. This increase was 20% in the reference sample, 27% and 8% in the series produced with short and long fiber inclusion, respectively. This situation was attributed to the higher strength of the matrix in the sealed samples. In addition, the possible increase in fiber-matrix bond is also thought to affect the situation. When the effect of fiber usage on peak loads was examined, it was observed that peak loads increased due to fibers' ability to bridge stresses, as expected. This increase was recorded as 3% and 22% in the short- and long fiber-reinforced series produced without sealing, respectively, while it was recorded as 9% and 10% in the sealed-cured series. In this context, long fibers provided higher peak loads than short fibers. Previous studies on similar topics have proved the contribution of fiber inclusion and curing regimes. The effects of fiber addition and curing regime on the mechanical properties of geopolymer composites have been the subject of numerous studies, yielding similar results. Nath and Sarker [37] investigated the effect of curing time on the mechanical properties of geopolymer composites. They stated that the fracture energy and peak loads increased as the curing duration increased from 28 days to 90 days in fly ash-based geopolymer concretes produced by ambient curing and activated with sodium silicate and sodium hydroxide. In a similar study, Wang et al. [8] investigated the effect of basalt fiber addition on the fracture energy of fly ash-based geopolymer concrete. The researchers reported that the peak loads achieved in the fiber-reinforced series were 17–37% higher than the control series due to the effect of basalt fiber addition on crack propagation. It was also reported that fracture energies increased with the addition of basalt fibers, with the highest increase observed being 56% at 0.05% fiber addition [8]. It was reported that using fiber in appropriate dosages improves fracture behavior and increases fracture energy in geopolymer composites due to mechanisms such as bridging, fiber fracture, and fiber pull-out [38]. Cai et al. [39] investigated the effect of PVA fiber and PVA powder on fly ash-based geopolymer composites and obtained SEM images illustrating the three aforementioned fiber working mechanisms.

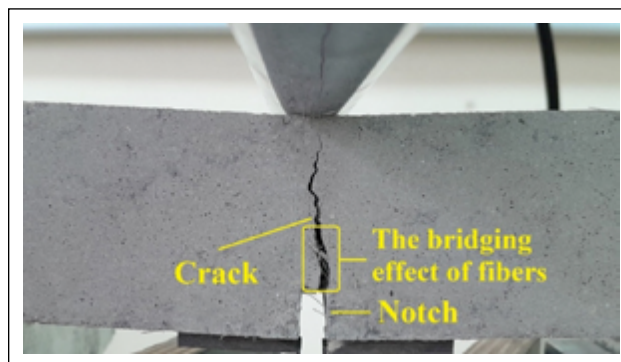


Figure 8. Fracture energy test and bridging effect of fibers.

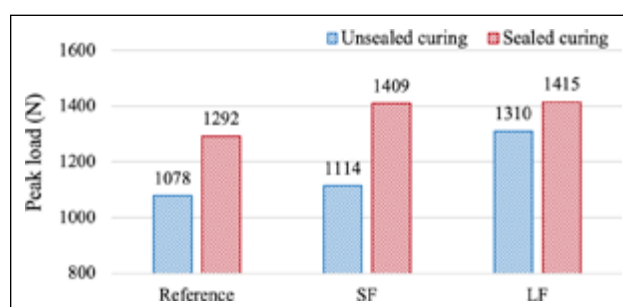


Figure 9. Average peak loads in the fracture energy test.

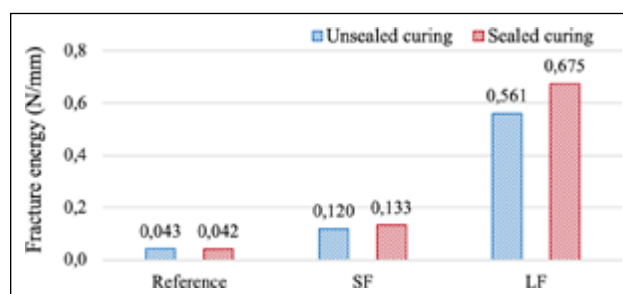


Figure 10. Average fracture energy values in the fracture energy test.

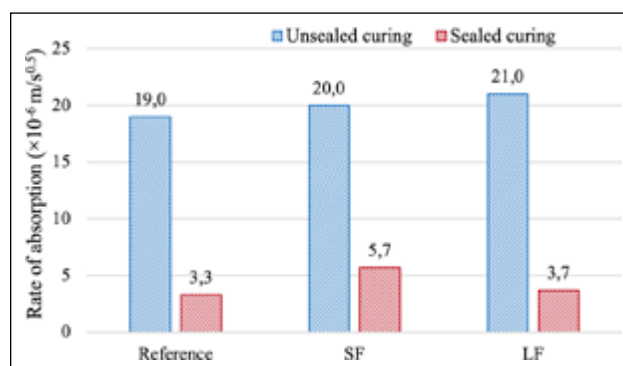


Figure 11. Initial rate of water absorption values.

3.3. Sorptivity

The rate of initial water absorption values of the mortars is presented comparatively in Figure 11. It is observed that the values for both the reference mortar and the fiber-reinforced mortars were very close to each other. Although

there was no significant difference between the values, considerable variations were evident between the sealed and unsealed samples. Specifically, the initial rate of water absorption value of unsealed reference mortar, $19 \times 10^{-6} \text{ m/s}^{0.5}$, decreased by 83% to $3.3 \times 10^{-6} \text{ m/s}^{0.5}$ in the sealed series. Similarly, 72% and 82% reductions were observed in the short and long fibers series, respectively. Thokchom et al. [40] investigated the effect of Na_2O amount on the properties of fly ash-based geopolymer mortars. The researchers observed that increasing the Na_2O ratio enhanced geopolymerization, resulting in a denser internal structure, increased compressive strength, and reduced water sorptivity. In a similar study, Shaikh [41] examined the effect of sodium hydroxide concentration (14 and 16 M) and the sodium silicate/sodium hydroxide ratio (2.5, 3.0, and 3.5) on the sorptivity properties of fly ash-based geopolymer concretes. The researcher reported that higher concentrations of sodium hydroxide or an increase in the sodium silicate/sodium hydroxide ratio led to decreased sorptivity, which was attributed to denser sodium aluminosilicate gel formation. In this study, it is thought that the significantly lower rate of absorption values observed in the sealed samples resulted from increased geopolymerization reactions. The observed increase in compressive strength supports this hypothesis.

4. CONCLUSIONS

This study investigated the effect of the curing method, fiber inclusion, and fiber length (6 and 12 mm PVA fiber) on fly ash-based geopolymer mortars' compressive strength and fracture energy. One series of samples was cured in water at 80 °C in direct contact with water, while another was first sealed with PVC cling film and duct tape and then cured in the same medium. Considering the materials used and the experiments conducted, the following results can be drawn:

- The curing method has significant effects on the mechanical properties. The compressive strengths and peak loads in the fracture energy test of the sealed-cured mortars are higher than those of the unsealed-cured series. Sealed curing increased compressive strength from 12% to 18% in this context. Furthermore, the peak load and fracture energy increased by up to 27% and 20%, respectively, due to sealing.
- With the addition of fibers, peak load, and fracture energy values increased; however, compressive strength values decreased. It was observed that long fibers had a more significant positive effect on peak load and fracture energy in both sealed and unsealed curing conditions. Specifically, long fibers provided 368% and 408% higher fracture energy values than short fibers under unsealed and sealed curing conditions. This improvement is attributed to the better bridging capacity of long fibers.
- It was determined that the fiber inclusion did not contribute positively to compressive strength and, in fact, decreased compressive strength by up to 7% and 12% under unsealed and sealed curing conditions, respec-

tively. In this context, it was also observed that the negative effect was more pronounced when long fibers were used. One of the reasons for this situation is the reduced workability, as also provided in the flow-diameter test.

- The water absorption rate at the sorptivity test of the sealed-cured series is significantly lower than that of the unsealed series. The reduction rates were 83%, 72%, and 82% in the fiber-free reference mixture, short and long fiber, respectively.

ETHICS

There are no ethical issues with the publication of this manuscript.

DATA AVAILABILITY STATEMENT

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

CONFLICT OF INTEREST

The author declares that he has no conflict of interest.

FINANCIAL DISCLOSURE

The author declared that this study has received no financial support.

USE OF AI FOR WRITING ASSISTANCE

Not declared.

PEER-REVIEW

Externally peer-reviewed.

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