

# Influences of high temperature on mechanical properties of fly ash based geopolymer mortars reinforced with PVA fiber

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## Abstract

In this study, a geopolymer composite containing PVA fiber was produced to recycle waste fly ash and obtain an eco-friendly binder. Mechanical properties of geopolymer mortars, produced by using F class fly ash which was activated with NaOH (sodium hydroxide), and reinforced by PVA (polyvinyl alcohol) fiber were investigated after high temperature effect. Geopolymer mortar samples produced by mixing with fly ash, sand, water and NaOH were placed in standard molds of 40×40×160 mm<sup>3</sup>. PVA fibers were used at percentages of 0.5%, 1% and 1.5% by volume in the experiment. Tests were performed on mortars exposed to high temperatures of 200°C, 400°C, 600°C and 800°C for physical and mechanical properties. For the specimens not subjected to high temperatures, an increase in the compressive strength of mortars containing PVA fibers was observed in comparison to mortar without PVA fiber. On the other hand, it was concluded that losses in compressive strength were less for mortar without PVA fiber when compared with the mortars containing PVA fibers. As a result of the modeling, the PVA ratio, which gives the optimum flexural and compressive strength, was determined as 1.47%. As a result of melting of PVA fibers under the effect of high temperature, 83.58% loss of compressive strength was determined in samples containing 1.5% PVA after 800°C temperature.

**Keywords:** geopolymer, fly ash, polyvinyl alcohol, high temperature, flexural strength, compressive strength.

## Introduction

Greenhouse gases are one of the most important problems today. Greenhouse gases, especially CO<sub>2</sub>, cause global warming and threaten human health. As of 2017, the world's CO<sub>2</sub> emissions reached 36.5 billion tons annually (Ritchie et al. 2021). It has been stated that 2018 cement production in the world has reached 4.1 billion tons, and 1.5 billion tons of the total CO<sub>2</sub> emission were caused by the cement sector (Global Carbon Atlas, 2020; Applications, Science, 2020). In addition, the use of fossil fuels to meet energy needs also causes CO<sub>2</sub> emissions. As of 2018, CO<sub>2</sub> emissions of coal-based fuels reached 14.6 billion tons (Applications, Science, 2020) Fly ash released during the combustion of coal threatens human health. The storage is a problem in terms of sustainable environmental protection. Due to the environmental problems of the cement sector, efforts are continuing to produce an alternative binder to cement. Industrial wastes such as blast furnace slag, fly ash, floor ash and silica fume are activated with various alkalis (Bingöl et al. 2020; Şahin, Mahyar & Erdoğan 2016). As a result of this chemical activation, cement-like binder can be produced. Materials such as lime, metakaolin (Bernal et al., 2010), red mud (Muraleedharan & Nadir 2021), andesite dust (Çelikten, 2021) can also react with alkalis and gain binding properties. The binder formed with these alkaline activated materials is called geopolymer (Davidovits, 1989).

Geopolymer concretes produced by activation of alkalis have high flexural and compressive strength (Atiş et al., 2015; Kaya et al., 2020; Ling, Zhang, et al., 2019; Neupane, 2018). Activator ratio (Shi et al., 2018; Wang et al., 2020), aggregate properties (Abdullah et al., 2015; Keke et al., 2019), water/binder ratio (Aydın, 2010), curing time and curing temperature (Durak et al., 2021), binding properties (Kaya et al., 2020) are the factors affecting the mechanical strengths of geopolymers. The SiO<sub>2</sub>/Na<sub>2</sub>O ratio, defined as the silicate modulus of the activator, is also effective on strength (Aydın, 2010; Bernal et al., 2010). High temperature is required for rapid reaction in geopolymer concretes produced with binders such as blast furnace slag and fly ash (Durak et al., 2021; Kaya et al., 2020). The most common known activators are sodium hydroxide (Durak et al., 2021; Rifaai et al., 2019) potassium hydroxide (Okoye, Durgaprasad & Singh 2015; Rahmiati et al., 2014) sodium silicate (Atabey et al., 2020), and so on. In many studies, activation is achieved by mixing NaOH and sodium silicate in different ratios (Atabey et al., 2020; Balcikanli & Ozbay 2016; Ugur Durak et al., 2021; Kaya

et al., 2020). The reaction called geopolymerization is complex and has not been fully understood to date. In addition, the reaction has been reported to consist of steps such as dissolution of aluminosilicate solids, gel formation, polycondensation and geopolymer formation (Zhang et al., 2016).

Geopolymer concrete has positive properties such as high mechanical strength (Atiş et al., 2015; Kaya et al., 2020; Ling, Wang, et al., 2019; Neupane, 2018), high abrasion resistance (Atabey et al., 2020; Atiş et al., 2015), high temperature resistance, low shrinkage (Çelikten, Sarıdemir, & Özgür Deneme 2019; Kaya et al., 2018), high freeze-thaw resistance (Atabey et al., 2020; Atiş et al., 2015; Aygörmüş, 2021). However, it has insufficient properties such as low sulfate resistance (Aydın, 2010; Elyamany, Abd Elmoaty & Elshaboury, 2018). In a study in which lightweight alkali activated concrete was produced with basaltic pumice, fly ash and blast furnace slag, it was stated that the samples showed very good durability against acid attacks compared to the control samples with portland cement (Sarıdemir & Çelikten 2020). The development of geopolymer concrete has increased rapidly over the last decade (Diaz-Loya, Allouche & Vaidya 2011; Ryu et al., 2013; Sofi et al., 2007). Alomayri (2019) reported that the flexural strength, flexural modulus, fracture toughness, compressive strength and impact strength increased in the geopolymer paste produced by using fly ash with Nano- $\text{Al}_2\text{O}_3$  added at different ratios. Kaur et al. (2018) activated mixtures with 0 to 10% nano-metakaolin substitutes in replacement of fly ash with NaOH. They also stated that an increase in compressive strength of the geopolymer mortars containing 4% nano-metakaolin was observed. Ravitheja & Kumar (2019), observed that compressive strength was improved in geopolymers, containing fly ash, produced by nano clay.

Polyvinyl alcohol (PVA) was first produced in 1924 (Jones, 1973). PVA is a material used in many industries such as textile, paint, medicine, food. It is also used as a binder and catalytic agent in the construction sector, and as a fiber in the cementitious composite (DeMerlis & Schoneker 2003; Xu et al., 2018). In recent years, there have been studies on treated cementitious composites. PVA fibers are reported to increase compressive strength in the geopolymer matrix (Xu et al., 2018). PVA fiber has some advantages such as increased energy absorption and toughness in the matrix, increased strength and increased ductility (Arısoy, 2002). Tanyıldızı & Yonar (2016) tested geopolymer concretes, containing different amounts of PVA fiber, exposed high temperatures of 200°C– 800°C. They found that the flexural and compressive strengths of the geopolymers increased with increasing fiber content in the mixture. However, it is also reported that flexural and compressive strengths of samples exposed to high temperature decreased. In another study, it was stated that flexural and compressive strengths of geopolymers containing fly ash were improved by addition 6% nano-silica to the mixture (Arısoy, 2002). A similar result for flexural strength was obtained by Ling et al. and they also reported that nano-silica adversely affected workability of geopolymer (Ling, Zhang, et al., 2019). (Xu et al., 2017) reported that PVA fibers increased flexural toughness of fly ash geopolymer composites.

The study presented here concerns the investigation of the effect of PVA fibers on physical and mechanical properties of geopolymer mortars containing fly ash under high temperatures. The optimum PVA ratio was determined as a result of the modeling made according to the results obtained from the high temperature experiment.

## Methodology

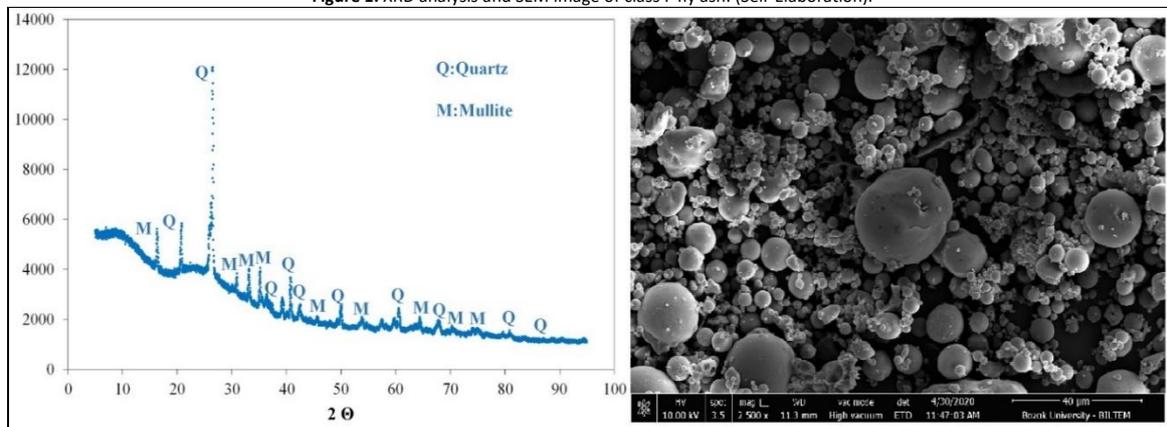
### Materials

Class F fly ash was obtained from Turkey's Süğözü Thermal Power Plant. The maximum particle size of fly ash is 75  $\mu\text{m}$ , and its experimentally obtained physical and chemical properties are given in Table 1. XRD (X-ray diffractometer) results of class F fly ash and SEM (scanning electron microscope) images are given in Figure 1. The PVA used in the study is shown in Figure 2. The properties of the PVA is also given in Table 2. Fine sand of limestone origin with a specific gravity of 2.63 and a water absorption capacity of 0.98% was used in the production of mortar. F class fly ash was activated with NaOH to produce geopolymer mortar samples. The binder/sand ratio was 1/3 and the water/binder ratio was 0.30. All of the geopolymer mortars with and without PVA fiber were prepared by mixing 14% by weight Na (sodium), fly ash and sand with water. Mixing was carried out in a standard cement mixer. The ratios of PVA fibers in fiber mortar specimens were planned as 0.5%, 1% and 1.5% by volume.

**Table 1.** Properties of fly ash. (Self-Elaboration).

Chemical properties	(%)
MgO	1.65
Al <sub>2</sub> O <sub>3</sub>	21.69
SiO <sub>2</sub>	60.51
SO <sub>3</sub>	0.53
Na <sub>2</sub> O	0.92
K <sub>2</sub> O	2.58
CaO	1.52
Fe <sub>2</sub> O <sub>3</sub>	7.85
Free CaO (%)	0.18
Ignation loss	2.57
Physical properties	
Specific weight (t/m <sup>3</sup> )	2.65
45 micron sieve remaining (%)	8.8
Blain specific surface (cm <sup>2</sup> /g)	2360

**Figure 1.** XRD analysis and SEM image of class F fly ash. (Self-Elaboration).



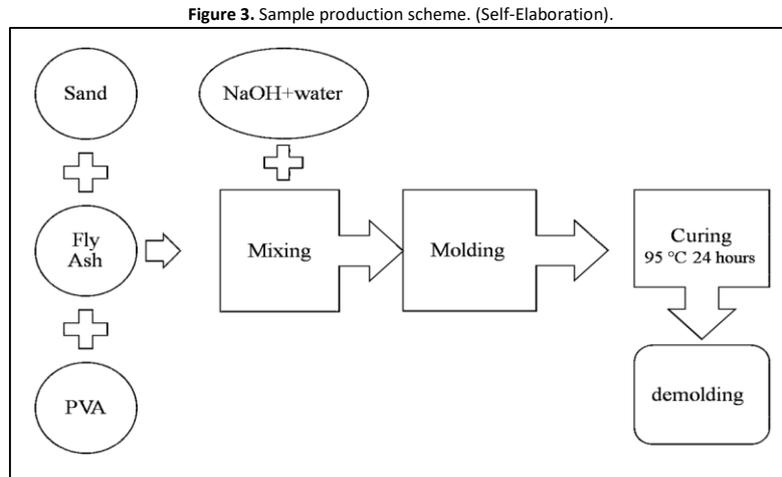
**Table 2.** Properties of PVA fiber. (Self-Elaboration).

Diameter (micron)	Length (mm)	Tensile strength (GPa)	Elastic Modulus (GPa)
40	8	1,6	42

**Figure 2.** PVA used in the study. (Self-Elaboration).



The geopolymer samples were placed in standard molds of dimensions 40x40x160mm<sup>3</sup>. The mixing, molding, heating and curing steps of the mortar are shown in Figure 3. In some geopolymer studies containing fly ash, temperatures between 80°C and 100°C were used for high strength (Atabey et al., 2020; Atiş et al., 2015; Durak et al., 2021; Kaya et al., 2020). All samples were exposed to heat at 95°C for 24 hours. Heat cured samples were kept in the ambient temperature of laboratory (~23-25°C) up to 28 days.



The mix proportions of the mortar samples are given in Table 3. For each testing temperature including ambient temperature, a total of twenty series mortar specimens were produced.

**Table 3.** Mix proportions of mortars. (Self-Elaboration).

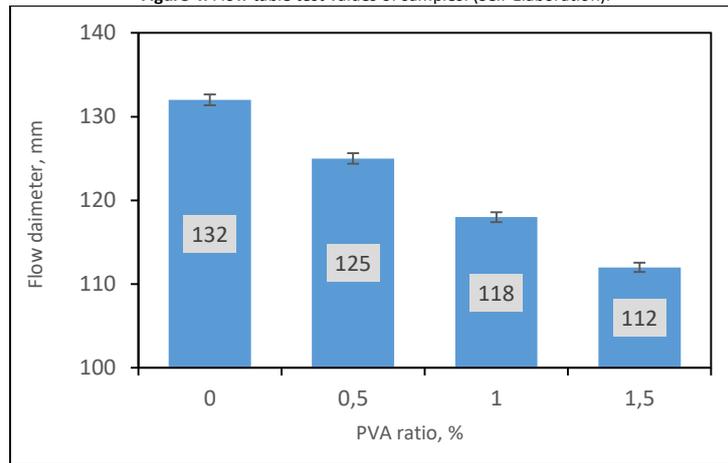
Mix code	Fly ash/sand	Water/fly ash	Sodium/fly ash (%)	PVA volumetric (%)
PVA0	1/3	0.30	14	0
PVA5	1/3	0.30	14	0.5
PVA10	1/3	0.30	14	1.0
PVA15	1/3	0.30	14	1.5

In the scope of experimental study, fresh mortar consistency (with flow table) was measured according to EN 1015-3 (TS EN 1015-3, 1999). Unit weight, water absorption, porosity and ultrasonic pulse velocity (UPV) tests were performed on the hardened mortar samples after the curing process was finished at the end of 28 days. Furthermore, flexural and compressive strength tests were carried out on prismatic samples of 40x40x160mm<sup>3</sup> dimensions with respect to EN 1015-11 (TS EN 1015-11, 2019) standard. After tests of control series specimens kept in ambient temperature were finished, other samples were subjected to high temperatures of 200°C, 400°C, 600°C and 800°C. Samples in each temperature group were allowed to cool for 1 hour after reaching the target temperature, and then allowed to cool in room temperature. Flexural and compressive strength tests were also made on specimens after high temperature effect. After high temperature, weight losses and UPV of the samples were tested.

## Results and discussion

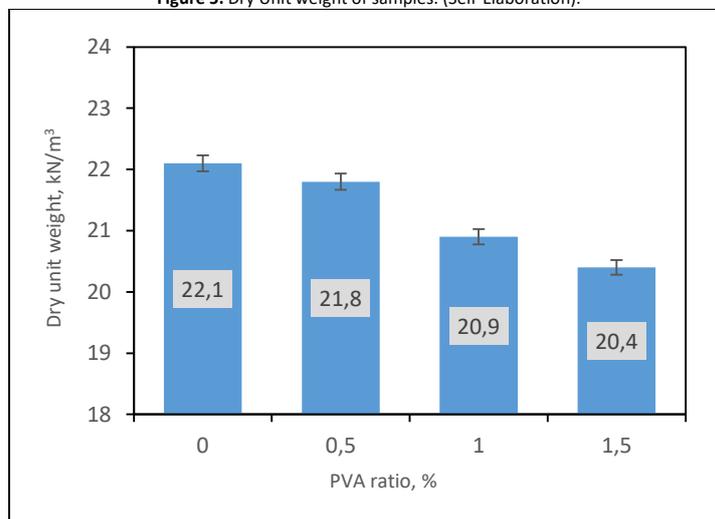
As a result of the consistency test carried out on fresh mortar mixtures, it was determined that the flow table value of the samples without PVA fiber was higher than the samples containing PVA fiber. It means that the workability decreased with an increasing amount of PVA fiber in the mixture. With the increase of fiber amount, a decrease in flow table values was also observed. While the spread value was 132 mm in non-fiber geopolymer samples, 112 mm spread value was determined in samples containing 1.5% PVA fiber. In geopolymer concretes containing PVA, it is stated that the workability decreases significantly with increasing amount of PVA (DeMerlis & Schoneker, 2003). The flow table test results are shown graphically in Figure 4.

Figure 4. Flow table test values of samples. (Self-Elaboration).



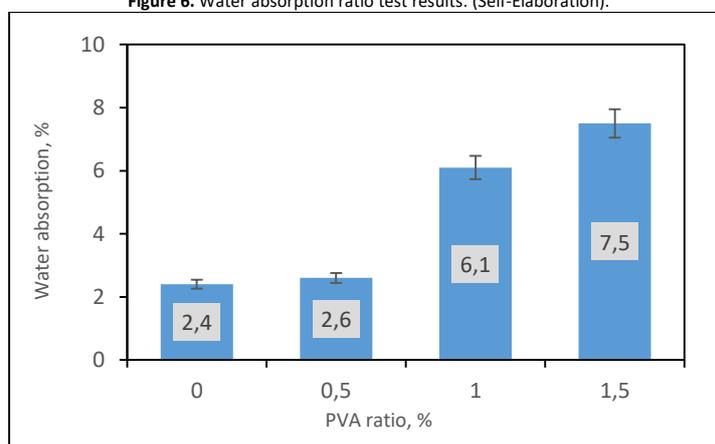
Dry unit weight, porosity, water absorption ratio, flexural and compressive tests were all done on hardened 40×40×160 mm<sup>3</sup> sized samples. Dry unit weights of samples vary between 20.4 and 22.1 kN/m<sup>3</sup>. It is observed that the unit weights decrease with the increase in the amount of PVA. Unit weights of the mortar samples are shown in Figure 5.

Figure 5. Dry Unit weight of samples. (Self-Elaboration).

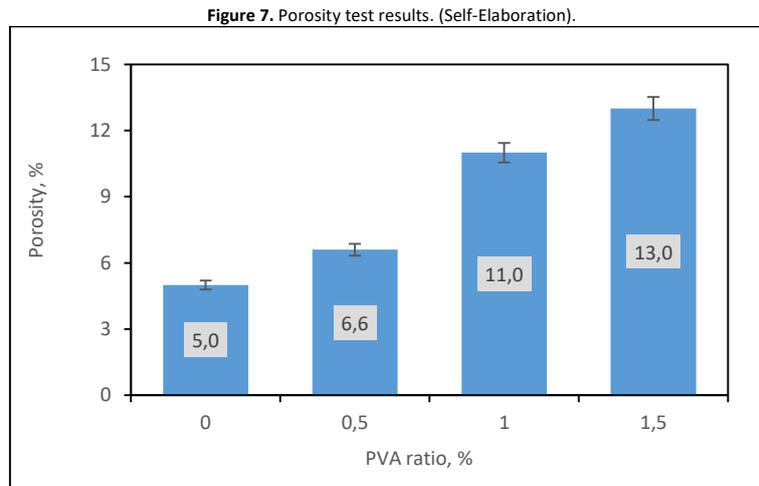


An increase in the amount of PVA fiber and an increase in water absorption ratios were observed. The lowest water absorption ratio was 2,4% for samples without PVA fiber and the highest water absorption ratio was 7,5% for samples containing 1,5% PVA fiber. The water absorption ratios of the samples are given in Figure 6. This increase in water absorption may be due to the voids result in clumping of PVA fibers during mixing and placing.

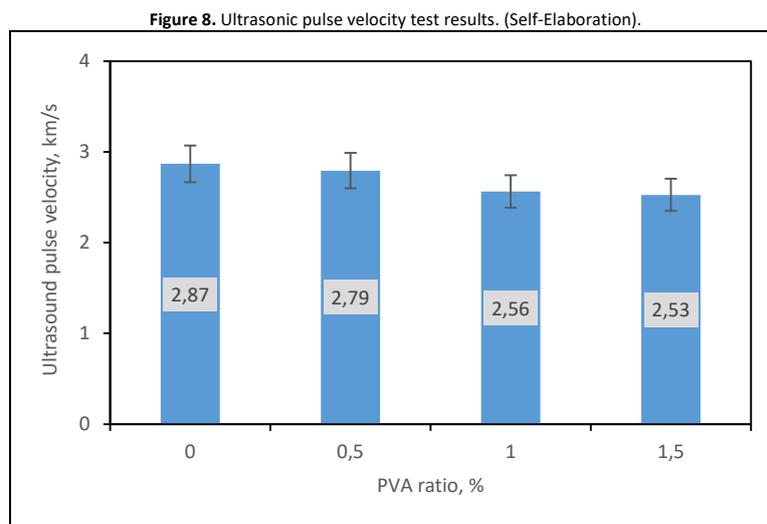
Figure 6. Water absorption ratio test results. (Self-Elaboration).



As the amount of PVA fiber increased, a certain increase in the porosity was observed. The porosity of samples without fiber was 5%, while the porosity of samples containing 1.5% fiber was 13%. The porosity test results are given in Figure 7. With the increase in the amount of PVA fiber in the mixture, the increase in water absorption ratio is similar to the literature (Xu et al., 2018). The porous structure of the geopolymer causes an increase in the amount of water absorption (Atabey et al., 2020; Kaya et al., 2018).

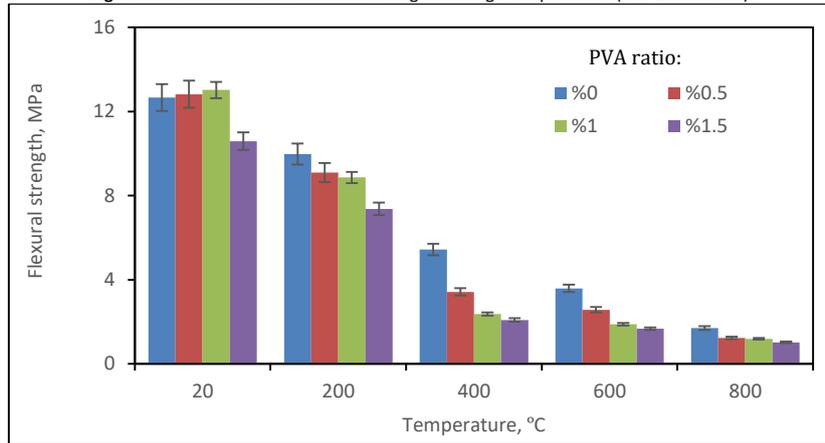


The UPV decreased with the increasing amount of PVA fiber. In fibreless samples, UPV was determined as 2.87 km/s. In samples containing 1.5% fiber, UPV was 2.53 km/s. The UPV of the samples is given in Figure 8. As the ultrasonic will take time to pass through the void with the increasing void ratio, the UPV decreases. An increase in the cavity ratio and a decrease in UPV is an expected result.

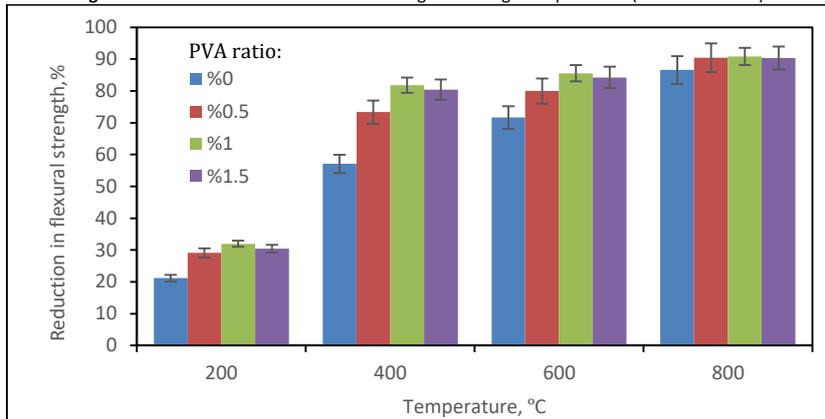


As the amount of PVA fiber increased, a certain increase was observed in flexural strength. Flexural strength of 12.66 MPa was observed in fiber free samples, while 13.02 MPa flexural strength was determined in samples containing 1% fiber. The relation between flexural strength and high temperature is shown in Figure 9. In other studies, it has been found that flexural strength increases with the increase of PVA fibers in the geopolymer composite (DeMerlis & Schoneker 2003; Tanyildizi & Yonar 2016). In general, it is expected that all types of fibers used in mortars and concrete improve the flexural strength and toughness. As a result of the high temperature effect, flexural strength of all mixtures decreased. It was observed that the flexural strengths of PVA free samples were higher than PVA containing samples at all temperatures. Flexural strengths of PVA free samples ranged from 1.70 MPa to 9.98 MPa. The flexural strength was determined as 7.37 MPa after the temperature of 200°C and 1.02 MPa after the temperature of 800 °C in the samples containing 1.5% PVA fiber. Ratio of reduction in flexural strength after high temperature in Figure 10. After high temperature PVA fiber free samples after 800°C, flexural strength decreased by 86.57% and in 1% PVA fiber samples 90.86%. The higher the PVA fiber content, the greater the loss of flexural strength as a result of high temperature was observed (Xu et al., 2018).

**Figure 9.** Relations between flexural strength and high temperature. (Self-Elaboration).



**Figure 10.** Ratio of reduction in flexural strength after high temperature. (Self-Elaboration).



A certain increase in compressive strength was observed with the increasing amount of PVA fiber. Compressive strengths were obtained 40.73 MPa in samples without PVA fiber and 51.95 MPa in samples containing 1.5% PVA fiber, respectively. The compressive strength of the samples is shown graphically in Figure 11. 8.57% increase in compressive strength was observed in non-fiber samples after 200 °C temperature. This is thought to be due to the non-activated activator in the sample. Some studies have shown an increase in compressive strength after high temperature effects in geopolymer mortars (Bernal et al., 2010; Kaya et al., 2018). After 800°C, the loss of compressive strength of PVA free samples is 68.5%. In the samples containing 0.5%, 1% and 1.5% PVA, the loss of compressive strength was 79.5%, 81.9%, 83.58% in the samples. The increase in PVA fiber ratio caused a decrease in the strength after high temperature. It is thought that after high temperature PVA fiber melting increases the void structure and deterioration of the internal structure causes the strength decrease. It was observed that compressive resistance losses were less in all temperature groups of PVA free samples compared to PVA containing samples.

**Figure 11.** Relations between compressive strength and high temperature. (Self-Elaboration).

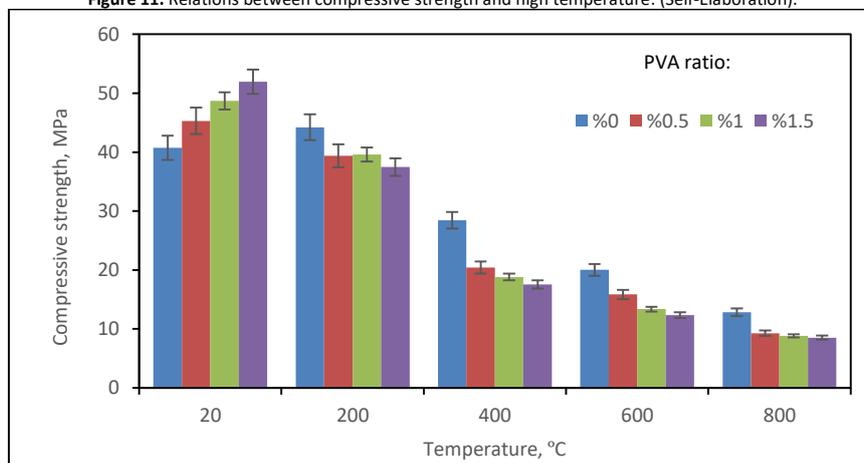
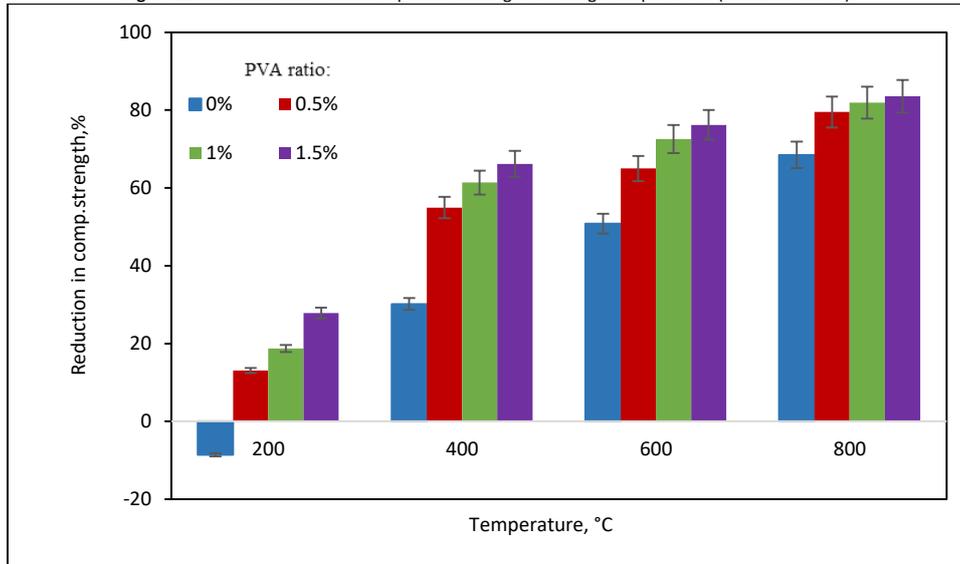


Figure 12. Ratio of reduction in compressive strength after high temperature. (Self-Elaboration).



In the study performed by (Tanyildizi & Yonar 2016) on geopolymer concretes containing PVA fiber, it is stated that the flexural and compressive strengths of the samples containing PVA fiber exposed to high temperature effect decreased. In the same study, it was reported that samples cured at 60°C showed better flexural and compressive strengths than samples cured at 80°C and 100°C. Geopolymer samples produced by curing at low temperature around 50°C are known to have high temperature resistances compared to samples produced by curing at high temperature around 90 °C. They show good performance in low temperature cured samples in terms of high temperature resistance due to the presence of unactivated activator. Since they have activators in low temperature cured samples, they perform well in terms of high temperature resistance (Kaya et al., 2018). In this study, it is considered that geopolymer samples produced at 95°C have significant strength losses.

Reduction of UPV of samples after high temperature effect are given in Figure 13. With the effect of temperature, a decrease in UPVs of all samples was observed. In PVA free samples, a decrease of 10.77% UPV was observed at 200°C, while samples containing 1.5% PVA decreased by 12.57%. While the loss of UPV was 65.43% in samples PVA free samples at 800°C temperature, the loss of UPV in samples containing 1.5% PVA was determined as 66.22%. A decrease in UPV was observed at all temperatures, with an increase in the amount of PVA in the samples. The increase in the void structure and the decrease of UPV due to post-temperature deterioration is similar to the literature (Kaya et al., 2020). The relationship between the flexural and compressive strengths of the samples is shown in Figure 14.  $R^2=0.99$  shows a strong relationship between flexural and compressive strengths.

Figure 13. Reduction of UPV of samples after high temperature effect. (Self-Elaboration).

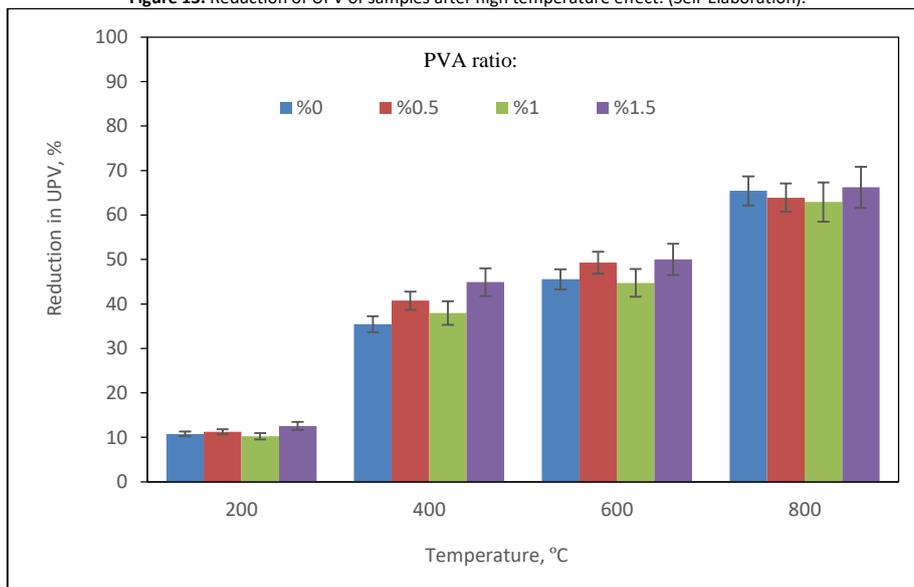
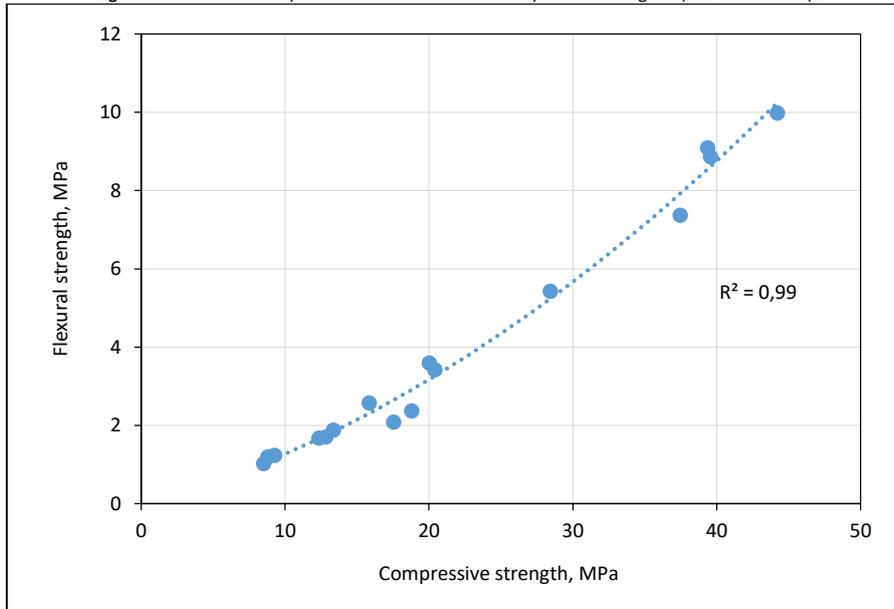
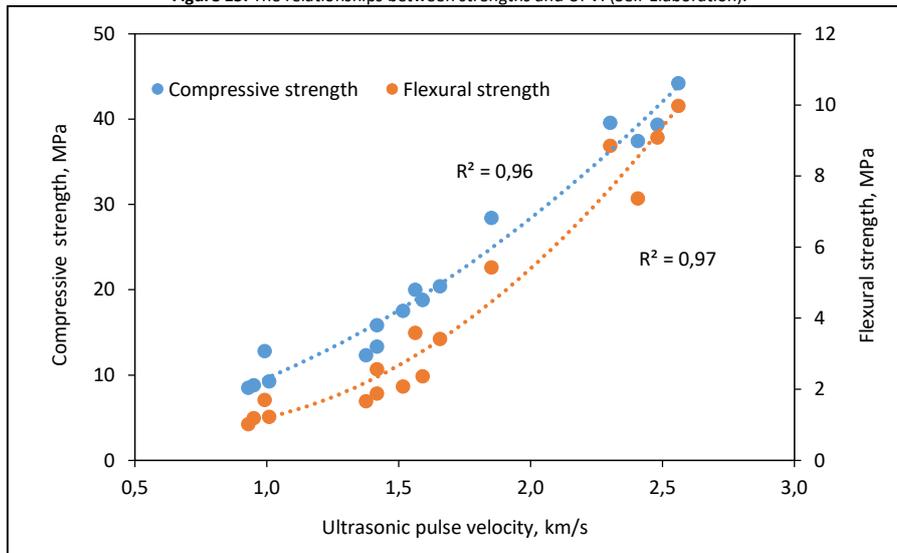


Figure 14. The relationship between the flexural and compressive strengths. (Self-Elaboration).



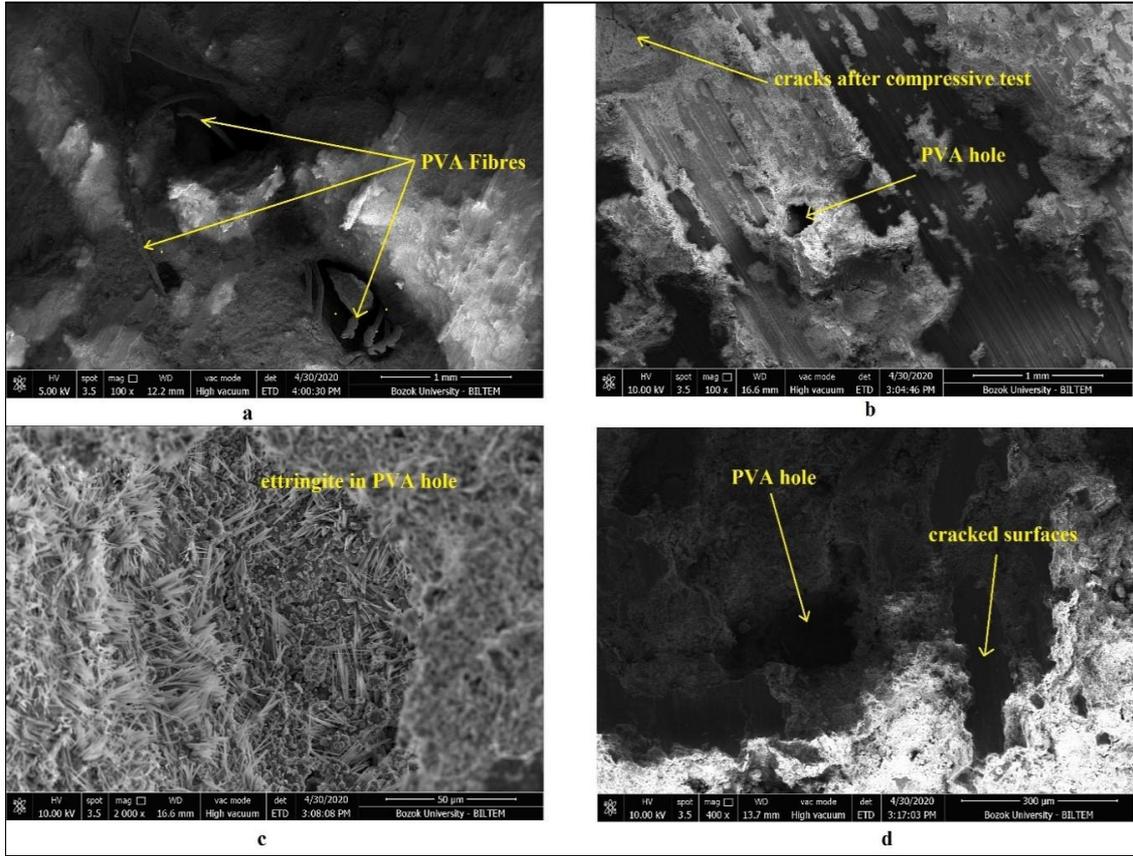
The relationship between the strengths of the samples before and after the high temperature with the ultrasonic pulse velocity ratio is shown in Figure 15. It is clearly seen that there is a strong relationship between flexural strength and UPV with the coefficient of determination of  $R^2=0.97$ . The relationship between compressive strength and UPV with the coefficient of determination of  $R^2=0.96$ . The relation shows that compressive strengths increase due to the increase in UPV ratio with the decrease of porosity.

Figure 15. The relationships between strengths and UPV. (Self-Elaboration).



The SEM images of the geopolymer samples containing 1.5% PVA before and after the temperature test are given in Fig. 16. In Fig. 16a, the image of the sample containing 1.5% PVA fiber before applying the temperature is given. In Figures of 16b and 16c, SEM images are given after 400°C temperature. A SEM image of PVA fiber sample, after 600°C temperature, is shown in Fig. 16d. It was exposed to 400°C and 600°C temperature, and the samples could not be seen due to the melting of PVA fibers in the samples. It was observed that there was hole in the place where PVA fibers were located. In addition, ettringite formation was observed in the sample (Figure 16c) exposed to 400°C temperature in the gap. In previous studies, it has been stated that in samples containing high-temperature fly ash, ettringite occurs with the effect of temperature (Chindapasirt et al., 2013).

Figure 16. SEM images of geopolymer mortar at 1.5% PVA; a) 200°C, b, c) 400°C, d) 600°C. (Self-Elaboration).



### Regression analysis of test results

Regression analyses were made by using Response Surface Methodology (RSM) for flexural strength and compressive strength test data. The aim of regression analyses was to observe the changes in strengths (residual strengths) depending on PVA contents at elevated temperature effect.

RSM uses mathematical and statistical methods to establish a model by analyzing of a set of input (dependent variables) and output (independent variables or responses) data (Braima, Anozie, & Odejobi, 2016) Models established for responses used to find optimum solutions for responses (Derringer & Suich 1980; Montgomery, 2005). In this experimental design, independent variables were PVA ratios (0%, 0.5%, 1% and 1.5%) and testing temperatures (200°C, 400°C, 600°C and 800°C). A total of  $4 \times 4 = 16$  design points were used in establishing the models for responses (compressive and strength flexural strengths) PVA ratios and testing temperatures are coded as *PVA* and *T*, respectively.  $f_c$  and  $f_f$  illustrate the compressive and strength flexural strengths, respectively.

A model for each strength was first established by using RSM incorporating best fitting surfaces depending on independent variables of *PVA* and *T*. To obtain the response surfaces, variance analysis (ANOVA) was performed by using design points. A 2 factor interaction (2FI) model for strength flexural strength and a quadratic model for compressive strength were used to obtain maximum  $R^2$  (coefficient of determination) values.

Response surfaces of models, obtained for residual strengths of  $f_c$  and  $f_f$ , were given in Fig. 18. Expressions of models are as follows:

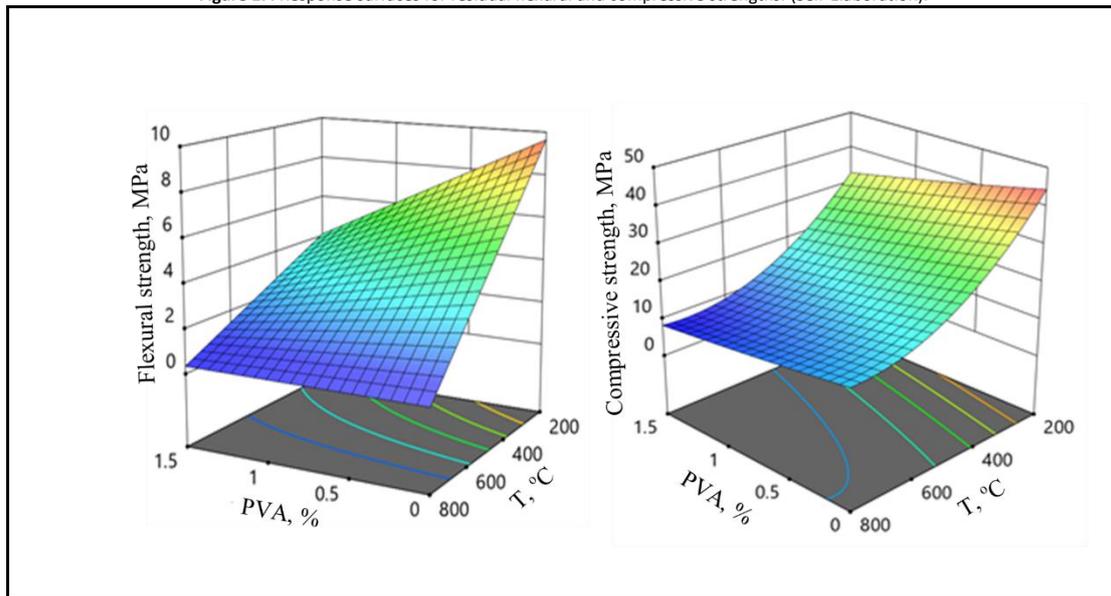
$$f_f = 12.74 - 4.90x(PVA) - 0.016x(T) + 0.006x(PVA)x(T) \quad (1)$$

$$f_c = 68.06 - 9.59x(PVA) - 0.135x(T) + 0.0054x(PVA)x(T) + 0.534x(PVA)^2 \quad (2)$$

The coefficients of determination ( $R^2$ ) of regression models, build for  $f_f$  and  $f_c$ , were found as 0.83 and 0.97. As a result of the answers received using independent variables (*PVA* and *T*), eq. (1) and eq. (2) were obtained. Figure 17. shows the residual strength values predicted by models and values obtained experimentally. The models, for prediction of residual strengths of PVA mortars, obtained by using test results of this experimental study have a good correlation

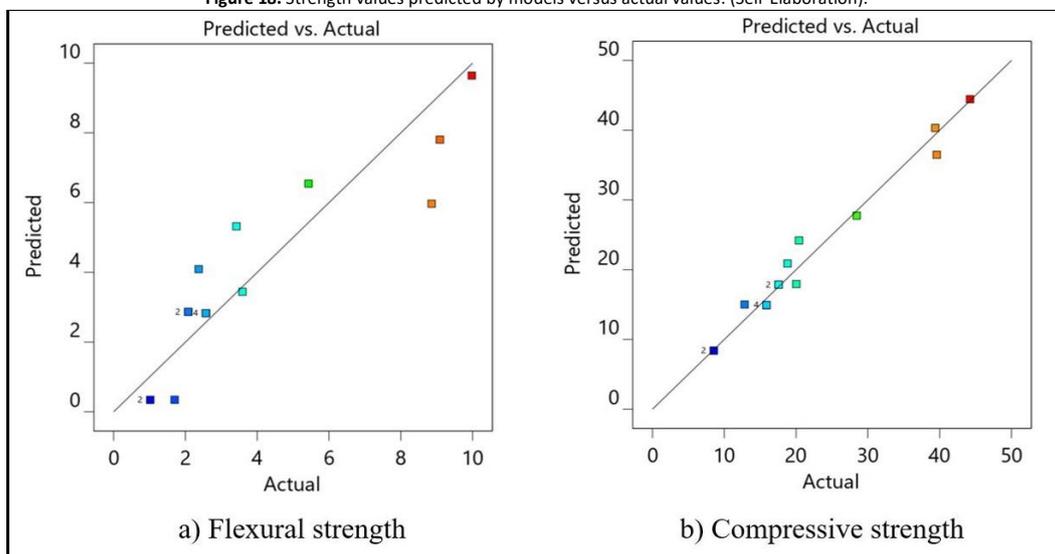
between temperature and PVA content. Before the mix design of PVA mortars to be subjected to the elevated temperatures, the models can be useful for estimation of residual strength depending on PVA content.

Figure 17. Response surfaces for residual flexural and compressive strengths. (Self-Elaboration).



When the response surface for the compressive strength ( $f_c$ ) and flexural strength ( $f_f$ ) in Figure 17 is examined, It was observed that the compressive strength was high at low temperature values, and the compressive strength decreased with increasing temperature value. The compressive strength increases with the increase of PVA ratio at 200°C. However, the compressive strength decreases between 600°C-700°C. For 1.5% PVA, compressive strengths are 34 MPa and 8.5 MPa at 200°C and 800°C, respectively. The highest flexural strength at PVA-free at 200°C was observed as 9.7 MPa. However, the lowest flexural strength as 1,02 MPa was obtained at the temperature range of 600°C-700°C at 1.5% PVA. Figure 18. shows the strength values predicted by models versus actual values.

Figure 18. Strength values predicted by models versus actual values. (Self-Elaboration).



## Conclusions

In this research, the effect of high temperature on mechanical properties in fly ash geopolymer mortars containing PVA fiber was investigated. The following results were obtained from the experimental studies:

- Flexural strengths of samples that do not contain PVA fiber after high temperatures showed the highest values. As PVA fiber ratio increased, it was observed that the loss of flexural strength as a result of high temperature was more.

- The compressive strengths of the samples which do not contain PVA fiber at all temperatures showed the highest values. It was observed that compressive strength losses were less in all temperature groups of PVA free samples compared to PVA containing samples.
- PVA fiber additive has no positive contribution to flexural and compressive strengths after 200°C. The reason of strength losses of fiber-containing samples can be explained by the degradation of PVA after 200°C.
- Although it is known that geopolymer mortars perform well in terms of resistance to high temperatures (Kaya et al. 2018), the high loss of strength in this study is thought to be caused by the deterioration of limestone based aggregate after 600°C temperature.
- The models, for the prediction of residual strengths of PVA mortars, have a good correlation between temperature and PVA content. Before the mix design of PVA mortars to be subjected to the elevated temperatures, the models can be useful for estimation of residual strength depending on PVA content.
- PVA fiber can be used when recycling fly ash in geopolymer production. PVA fibers contribute to the geopolymer strength at ambient temperature. However, no positive contribution to strength was observed under the high temperature effect.

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