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Effect of High Temperature on the Strength of Geopolymer Concrete Produced from Industrial Tailings

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If the concrete is affected by high temperature, significant decreases in strength are observed. The temperature of the concrete at 250-300 °C is considered as a limit. It is stated that the damage occurring below this temperature is insignificant. As the temperature progresses into the concrete during the fire, the regions where the temperature rises above these limits are damaged and the carrier part of the structure decreases gradually.

In this study, we researched the strength properties of the geopolymer concretes produced from the mortar, which is a mixture of slag, fly ash and sodium silicate (Na_2SiO_3) solution. Uniaxial compressive strengths (UCS), point load strengths (PLS), and the stress-strain curves were revealed and the optimal condition for geopolymer concrete production were determined. After the optimum conditions were selected, the concrete specimens were exposured to high temperatures (400, 500 and 600 °C). Effect of the high temperatures on strength of the produced specimens were examined.

Keywords: Geopolymer, Concrete, High, Temperature.

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1. Introduction

Although the amount of energy obtained from renewable energy sources such as sun, wind, wave, stream, biomass and geothermal is increasing day by day, the use of fossil fuels, which are the main source of energy production, will continue for the next 100 years in line with the increasing energy demand. In addition, advances in flue gas treatment methods with advancing technology make the use of fossil fuels more environmentally sensitive. In particular, applications to bring fly ash to the industry are limited to the use of these materials only in cement-ready concrete plants in Turkey.

In parallel with the development of industrial applications, the utilization of residual materials accumulated more and more is of increasing importance. In the last 20 years, studies on the evaluation of such residual materials have continued on a limited scale in Turkey. Increasing environmental concerns have led to efforts to take measures on a global scale. In Turkey, the legislation on the control and evaluation of waste has been accelerated especially after 2000 with the regulations such as "Regulation on Waste Management", and "Regulation on the Landfill of Waste".

Fly ashes are the particles of powder size obtained from the flue gases with the help of cyclone and electrofilters as a result of burning of pulverized coals in thermal power plants and loss of carbon and volatile contents in these coals. The ash obtained in the lower part of the furnaces in the thermal power plant is called as bottom ash and its dimensions are higher compared to the fly ash [1-3]. Particle sizes of fly ashes vary between 0.5-150 µm. Crude ash can be formed by cooling the coal burned in water in the cyclone type furnaces [4].

Geopolymers are promising materials that can replace Portland cement concretes [5-6]. The major disadvantage of Portland cement is that the amount of CO_2 produced by humans is around 5-7% due to the production of portland cement [7-8]. CO_2 emissions during Portland cement production are not only caused by the calcination process during clinker production, but also by fossil fuels used to provide calcination heat. Both processes are equally responsible for CO_2 emissions [9-10]. Compared with traditional Portland cement, geopolymers have superior properties such as fire proofing, low price, environmental sensitivity, sound absorption [11-12].

The geopolymers described for the first time by Davidovits, who also have patents for most soil mineral based materials,

include AlO_4 and SiO_4 quadrangles that form 3-dimensional poly (sialate), poly (sialate-siloxo) and poly (sialate-disiloxo) polymeric chains [13-14]. The molecular sequences of geopolymer structures are shown in Figure 1. Si-O-Si and Si-O-Al chains are formed by establishing polymeric amorphous or semi-crystalline structures [15]. Geopolymerization reactions occurs between solid aluminasilicate oxides (minerals and inorganic by-products) and alkali metal silicate solutions (NaOH, KOH, etc.), at moderate temperatures (20-150 °C) and high basic conditions. Unlike zeolite crystals, geopolymer chemistry has a more amorphous particle structure. Figure 2 summarizes the procedure for obtaining geopolymer materials.

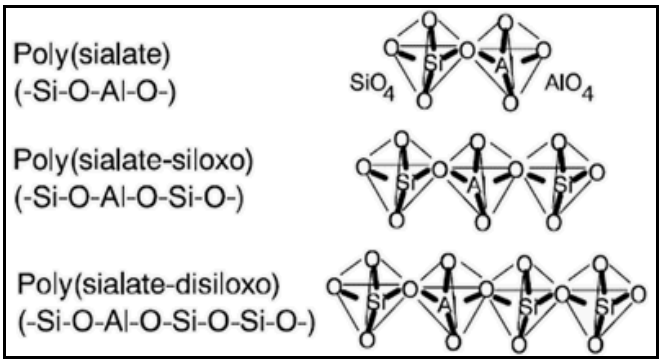

Figure 1. *Molecular sequences of polysialates (Connie et al., 2018)*

Polysialates and hydrosodalites are in the three-dimensional hexagonal molecule sequence. As a result of polycondensation, they give water by dehydratation and dehydration and reach a grain structure similar to zeolite rocks. Unlike zeolite crystals, three-dimensional hexagonal amorphous molecular bond structure of alumino silicate soil materials are geopolymers [16]. The simple formulation of geopolymers is given below.

$$X_q[-(SiO_2)_a\text{-}AlO_2]_q.nH_2O \quad (1)$$

Here; X is the metal cation (Ca^{2+}, K^+ or Na^+), a is 1, 2 or 3, and q is the degree of polycondensation.

Fly ashes, metakaolines, glass residues, red sludge, ashes of agricultural industry by-products (coconut, sugarcane, rice paddy, etc.), ferrochrome slag, concrete demolition residues are some materials containing aluminacilicate used to produce geopolymer concrete [17-24].

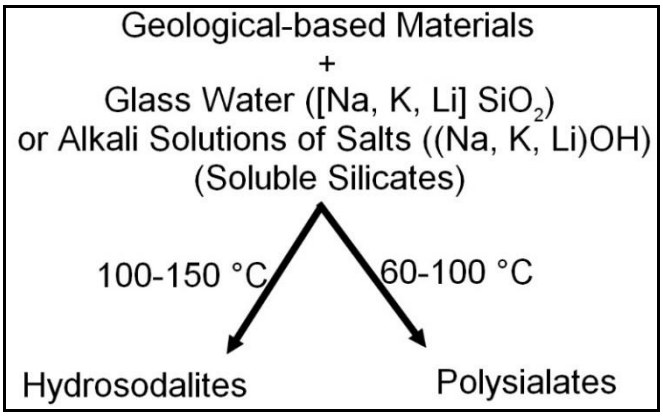

Figure 2. *Flow chart for producing geopolymer material*

The University of Queensland's Global Change Institute building was built entirely with geopolymer concretes from fly ash and blast furnace slag. Brisbane West Wellcamp Airport in South Queensland was established using 70000 tons of geopolymer concrete and was put into service in 2014 [25]. There are dozens of companies that produce geopolymer concrete worldwide [26-29].

In this study, we researched the strength properties of the geopolymer concretes produced from the mortar, which is a mixture of slag, fly ash and sodium silicate (Na_2SiO_3) solution. Uniaxial compressive strengths (UCS), point load strengths (PLS), and the stress-strain curves were revealed and the optimal condition for geopolymer concrete production were determined. After the optimum conditions were selected, the concrete specimens were exposured to high temperatures (400, 500 and 600 °C). Effect of the high temperatures on strength of the produced specimens were examined.

2. Materials and methods

Fly ashes were taken from Sugözü Thermal Power Plant in Yumurtalık District of Adana. All the fly ashes of Sugözü are less than 80 µm in size. Ferrochrome slags from Antalya and Elazığ ferrochrome plant and arc furnace slag from İskenderun were used for aggregate production. The slags were crashed by a laboratory type jaw crusher. In accordance with TS 802 standard, aggregates were prepared in accordance with the maximum grain size determined to be 12 mm [30]. A minimum of 95% sodium meta silicate (anhydrous) was obtained from TEKKİM chemical Inc. to provide geopolymerization. Sodium meta silicate contains 49.5% Na_2O and 46% SiO_2. XRF analyses were applied to the slags (Table 1). The density of the fly ash was determined as 2.33 g/cm^3 by a pycnometer. When the XRF analysis of the arc furnace slag obtained from the iron and steel plant is examined, it is seen that the slag contains a high percentage of iron. The iron content is followed by high calcium content.

Table 1. XRF analyses of the samples.

Content	SiO_2	Al_2O_3	CaO	Fe_2O_3	MnO	K_2O	TiO_2	SrO	BaO	ZrO_2	SO_3	Na_2O	CuO	MgO	Cr_2O_3	LOI#
Sugözü Fly Ashes	55.20	24.10	5.50	8.11	LA*	1.62	1.44	0.27	0.26	0.19	2.34	LA*	LA*	LA*	LA*	2.34
Elazığ Ferrochrome Slags	38.00	22.50	1.56	1.22	0.30	0.23	0.37	LA*	LA*	LA*	0.64	LA*	0.02	29.00	6.03	0.34
Antalya Ferrochrome Slags	41.70	14.20	25.45	1.22	0.10	LA*	0.25	LA*	LA*	LA*	0.31	LA*	0.02	12.30	3.56	1.41
Arc Furnace Slags	11.59	7.63	31.90	37.38	3.95	LA*	0.30	LA*	LA*	LA*	0.39	LA*	LA*	LA*	1.34	0.01

*Under detection limit, #Loss on ignition

In the slags obtained from Elazığ ferrochrome plant, which produces high carbon ferrochrome, major oxides are silicium, magnesium and aluminum oxides. Calcium content is very low and loss on ignition value is lower than Antalya ferrochrome slag.

Resistance of slag aggregates against thermal and degradation was determined by using saturated magnesium sulfate solution according to TS-EN 1367-2 Turkish Standard [31]. Samples weighing approximate 500 g between 10-14 mm in size were used from each aggregate group. The samples which were kept in saturated magnesium sulfate solution for 17 hours were filtered and dried in the oven for 24 hours and then immersed in the solution for 17 hours. In this way, the process was repeated 5 times and then sieved through a 10 mm sieve and the ratio of the portion passing through the sieve to the total sample mass and thermal and degradation resistance were calculated.

According to TS-EN 1097-2 Turkish Standard, Loss Angeles Abrasion Test was applied to the slags [32]. Slag aggregates between 8-12 mm were used in this test. 10 balls weighing 4263 g were placed in the Loss Angeles tester and the device was rotated 500 revolutions. The sample was then extracted and sieved through a 1.6 mm sieve. Wear loss values were found by proportional to the total mass used. Each experiment was repeated 2 times to obtain the mean values. In Table 2, when the los angeles wear values of the slag are examined, it is seen that the most resistant to slag is the arc furnace slag.

Table 2. Wear, thermal resistance and density values of slags.

Aggregate types	Antalya Ferrochrome Slags	Elazığ Ferrochrome Slags	Arc Furnace Slags
Los Angeles Abrasion (%)	16.46	15.28	15.08
Density (g/cm³)	3.12	3.25	3.99
Sulfate Soundness (%)	9.51	12.48	12.57

The slag with the highest density is again the arc furnace slag. According to the results of the magnesium sulphate test, the most resistant slag against thermal and climatic degradation were determined as Antalya ferrochrome slag.

The alkali solution containing 5M Na_2SiO_3 was used for geopolymerization. Tap water was used in the preparation of the solutions. The solutions were prepared 30 minutes before the mortar mixture was formed. The mortar mixture was poured into 5 x 5 x 5 cm molds immediately after preparation. The mixture was stirred for 5 minutes before pouring into the molds. A thin layer of grease was used for easy removal of concrete blocks from the molds. The molds were struck for 3 minutes to remove any air trapped in the mortars. After pouring the mortars into the molds, the molds were placed in the oven at predetermined temperature and left in the oven for 1 day. The curing process was then continued at room temperature. The ratio of aggregates to fly ash was mixed to be 0.66.

In order to measure uniaxial compressive strength, ELE brand concrete press was used. In accordance with ASTM C109 / C109M standard, cubic samples were placed in the loading section and loading process started by computer [33]. The loading speed was selected as 200 kgf/sec. When the cubic sample was broken, the compressive strength test was terminated. UCS values were determined by Equation 2.

$$\sigma = F / A \quad (2)$$

Where; F is pressure (kgf) and A is area (cm^2).

UCS values were converted to MPa. Point load strength of concrete blocks were measured with ELE brand point load resistance device. Protherm (PLF model) ash furnace was used for high temperature resistance tests.

3. Results and discussions

3.1. Effect of slag aggregate type on strength of geopolymer concrete

During the experiments, it was determined that the relative humidity rate ranged from 55 to 70%. The aggregates were mixed with Sugözü thermal power plant fly ashes, where the ratio of aggregates to fly ash was 0.66, which was best achieved in previous experiments. Then 5 M Na_2SiO_3 solution was added to the mixture to form the mortar and the mortar was poured into molds.

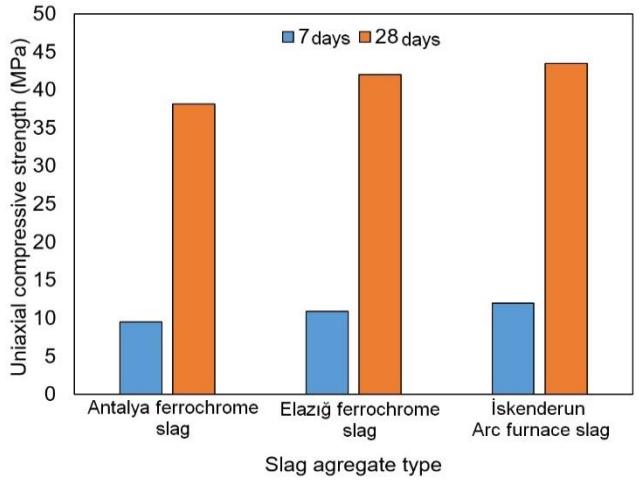

Figure 3. *Compressive strengths according to types of aggregate*

The uniaxial compressive strengths were measured on days 7 and 28 without applying temperature. As it can be seen from Figure 3, the best compressive strength values were obtained in experiments using arc furnace slags as aggregates. These results are consistent with the Los Angeles abrasion test results of the aggregates.

Additional experiments were carried out using arc furnace slag where the highest strength was obtained. In these experiments, arc furnace slag amounts were increased gradually and changes in strength were investigated. After the mortar mixtures were poured into the molds, the molds were cured in the oven at 70 °C for 1 day. The concrete samples were then stored at room temperature. It has been determined that the addition of aggregates up to 20% does not cause a decrease in strength but after the additions it starts to decrease. Especially with the addition of more than 50% of aggregate, strength was reduced by half (Figure 4). It was determined that unit volume weights increased with heavier aggregate ratio and heavier concretes were obtained. Point load strengths of the specimens were also measured. As can be seen in Figure 5, after the addition of 20% arc furnace slag, the strength of concrete samples decreases. These values were found to confirm the compressive strength values. In addition to these findings, the deformations of the concrete samples against strength were calculated.

As it can be seen from Figure 6, although the strength decreases as the aggregate amount increases, the deformation rates are close to each other.

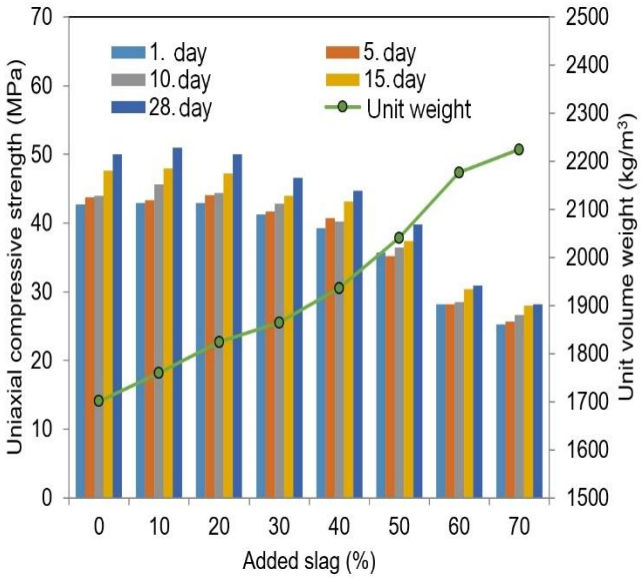

Figure 4. *Compressive strengths according to slag aggregate ratios of arc furnace*

Hard arc furnace slag has a significant effect on the formation of this result. The pre-breakage behavior of the concretes formed with stretchable aggregates such as calcined perlite or pumice is not seen in the concretes prepared by using harder arc furnace slag aggregates.

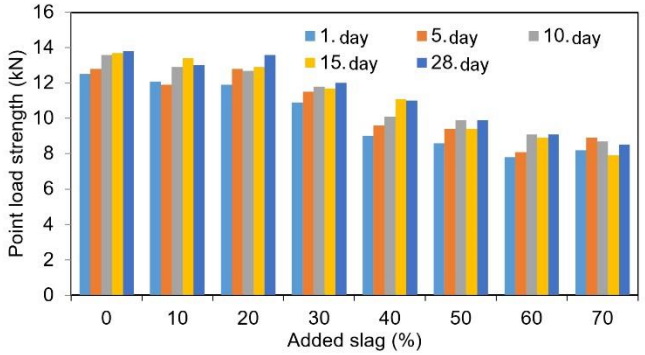

Figure 5. *Point load strengths according to slag aggregate ratios of arc furnace*

3.2. The effect of high temperatures on concrete strength

In order to observe the changes in strength of geopolymer concretes at high temperature, geopolymer concrete samples were exposed to 400, 500 and 600 °C temperature in ash furnace for 1 hour. Although no structural changes were observed in the samples, shallow capillary cracks were detected on their surfaces. The samples were then subjected to uniaxial compressive strength tests (Table 3). There was a decrease in the strength of the samples held at high temperature. While the strength losses at 400 degrees are around 10%, the strength losses at 500 and 600 degrees vary between 20-40%.

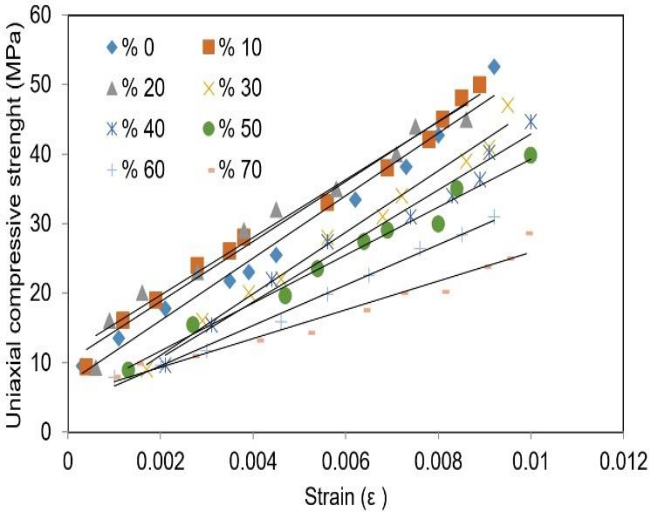

Figure 6. *Stress-strain curves according to added aggregate proportions*

As it can be seen in Figure 7, it is determined that the decreases in the strengths decrease as the amount of slag aggregates increases, even though the strength decreases with the increase in temperature.

Table 3. Changes in concrete strength at high temperatures (MPa).

Temp.	Aggregate amount (%)							
	0	10	20	30	40	50	60	70
-	50.03	51.02	50.03	46.59	44.73	39.82	30.9	28.15
400 ˚C	45.12	46.12	44.16	43.12	41.16	37.15	28.16	26.15
500 ˚C	34.15	36.15	37.46	34.46	34.46	30.46	24.46	23.16
600 ˚C	27.46	27.78	28.16	26.96	26.16	24.48	20.99	19.99

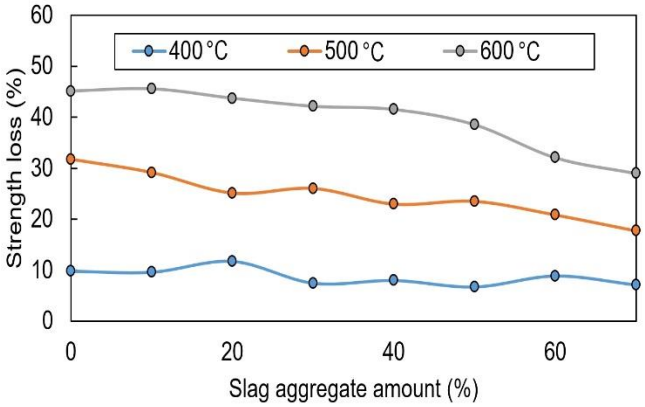

Figure 7. *Strength losses after heat treatments*

4. Conclusions

Exposure to fire is one of the most destructive process that concrete can face. The decrease in the strength of concrete under high temperature depends on the exposure time and the exposure temperature [34]. After the 500 °C threshold is passed, the compressive strength of concrete generally drops by 50% to 60%, and the concrete is considered fully damaged [35].

In this study, we researched the strength properties of the geopolymer concretes produced from the mortar, which is a mixture of slag, fly ash and sodium silicate (Na_2SiO_3) solution. Firstly, 3 different slag aggregates were used in geopolymer concrete and the concrete samples were formed. Slag aggregates used in the highest strength concrete were determined as arc furnace slags. It was determined that the addition of aggregates up to 20% does not cause a decrease in strength but after the additions it starts to decrease. In order to observe the changes in strength of geopolymer concretes at high temperature, geopolymer concrete samples were exposed to 400, 500 and 600 °C temperature in ash furnace for 1 hour. While the strength losses at 400 degrees are around 10%, the strength losses at 500 and 600 degrees vary between 20-40%. it was determined that the decreases in the strengths decrease as the amount of slag aggregates increases.

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