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Araştırma Makalesi / Research Article

Strength Development of Heat Cured and Ambient Cured Sodium Hydroxide Activated Fly Ash Based Geopolymer

Sodyum Hidroksit ile Aktifleştirilmiş Uçucu Kül Bazlı Geopolimerlerin Isıl Kür ve Ortam Küründe Dayanım Gelişimi

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ABSTRACT

Compressive strength development of class F fly ash geopolymer activated by sodium hydroxide was compared between initial heat curing at 75ºC for 24 hours and the ambient medium. Class F geopolymeric mortar was produced with standard Rilem sand, sodium hydroxide, and water. Mortar mixtures ratios were 3, 1, and 0.288 for sand, fly ash, and water, respectively. Some samples were cured in laboratory conditions; some samples were heat cured for 24 hours at 75ºC. Ambient curing medium result with non-measurable low compressive strength up to 7 days, however significant strength development observed in longer curing time up to six months. Heat curing developed higher strength at all times than ambient curing did. It was concluded that heat cured geopolymer samples could be utilized in construction materials, while utilization of non-heat cured samples was not practical due to its longer curing duration needs.

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ÖZ

Sodyum hidroksit ile aktifleştirilmiş F sınıfı uçucu kül geopolimerinin basınç dayanımı gelişimi, 75ºC'de 24 saatlik ilk ısıl kürleme ile laboratuvar ortamı arasında karşılaştırılmıştır. F sınıfı geopolimerik harç, standart Rilem kumu, sodyum hidroksit ve su kullanılarak üretilmiştir. Harç karışım oranları kum, uçucu kül ve su için sırasıyla 3, 1 ve 0.288 olarak kullanılmıştır. Üretilen numunelerin laboratuvar ortamında ve 75ºC'de 24 saat ısıda ayrı ayrı bekletilerek kür edilmiştir. Ortam şartlarında bekletilen numunelerde 7 güne kadar ölçülemeyen düşük basınç dayanımları elde edilmiştir. Ancak altı aya kadar daha uzun kürleme süresinde önemli bir dayanım gelişimi gözlenmiştir. Isı ortamında kürlenen numuneler her zaman ortam kürüne göre daha yüksek dayanım sonuçları vermiştir. Isı ile kürlenen geopolimer numunelerin inşaat malzemelerinde kullanılabileceği, ısı ile kürlenmemiş numunelerin kullanımının ise daha uzun kürlenme süresi ihtiyaçları nedeniyle pratik olmadığı sonucuna varılmıştır.

***Sorumlu yazar**

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1. INTRODUCTION

Geopolymers were presented as an alternative to the utilization of Portland cement (OPC) in the concrete industry; mainly, geopolymers produced with fly ash got the high potential to be an alternative to the application of Ordinary Portland Cement (OPC) [1].

Studies were carried out to investigate the properties of alkali-activated fly ash or fly ash-based geopolymers; the number of studies is increasing and going on an accelerated manner.

Geopolymerization is a chemical process similar to polymeric reaction; as a result of chemical reaction, it forms alumina and siliceous chain with strong binding properties. Geopolymeric reaction takes place between amorphous silica and alumina in an alkali activator medium with a high pH value. This reaction is generally called alkali activation, which converts the amorphous glass content of geopolymeric source materials into the solid matter with strong binding properties [2,5].

Studies have shown that role of molarity of alkali activator materials in the process of geopolymeric reaction is significant. Alkali medium with high pH value, primarily, dissolves the amorphous glassy contents of geopolymeric source materials (i.e., fly ash, silica fume, metakaolin etc.), at the end of a chemical reaction; geopolymer chains made of sodium, alumina, and silica are formed [6,10]. In brief, amorphous glassy contents of geopolymeric source materials are dissolved in the presence of high pH value provided by strong alkali activators (i.e. NaOH, KOH). Later on, geopolymeric reaction forms a solid matter made of sodium-alumina-silicate with strong binding property [6].

As it is stated before, geopolymerization is a chemical reaction and process; it takes place in time. Apart from this, it is known that geopolymeric reaction of fly ash-based geopolymer mixtures at ambient temperature is quite slow. For these reasons, the temperature of curing medium as well as curing durations are prominent and dominant parameters that influence the reaction kinetics of geopolymerization [1, 4, 6, 9, 11, 16]. In addition, amount of alkali or molarity of alkali medium is another specific crucial parameter for geopolymerization process since geopolymeric reaction takes place in the presence of strong alkali activating solution [10, 17, 19].

Researchers, working on alkali-activated geopolymeric binder using fly ash as geopolymeric source material, obtained and reported compressive strength of geopolymer mortar and geopolymer concrete in the range of 10 MPa and 120 MPa values depending on fly ash used, amount of alkali activator, activator type, heat curing temperature and duration [8, 10, 18, 20, 25].

Although it is reported that geopolymeric reaction of class F fly ash-based geopolymer is very slow at ambient laboratory condition, there are no studies how curing duration influence the strength development of fly ash-based and NaOH activated geopolymer mixture cured at laboratory condition. This work focuses on long-term compressive and flexural strength development of fly ash-based alkali-activated geopolymer mortar cured at ambient laboratory conditions and temperatures. To achieve this target, fly ash based alkali-activated fresh geopolymer mixtures were prepared and cured in laboratory conditions until six months of curing duration.

For the aim of comparison of the influence of heat curing and ambient temperature curing at the laboratory, some mortar mixtures were initially heat cured for 24 hours at 75^{o}C, some of the mortar mixtures were cured at laboratory without initial heat curing. After 24 hours of initial heat curing, geopolymer mortar samples were stored in laboratory conditions and further cured until six months duration. At the end of the selected curing duration, compressive and flexural strength of samples were measured and evaluated. Unit weight and workability of mixtures were also measured.

2. MATERIAL AND METHOD

In obtaining geopolymeric binder, class F fly ash was used. Fly ash was taken from Sugozu thermal power plant in Yumurtalık county of Adana city in southern Turkey. Chemical oxide compositions of fly ash were presented in Table 1.

Fly ash used has complied with the European Norm standard of [26] and American standard of ASTM C 618 [27]. It is classified as class F fly ash according to ASTM C618 standard due to its total amount of SiO_2+Al_2O_3+Fe_2O_3 oxide value, which is higher than 70% and the amount of CaO which is less than 10% (low lime). The specific gravity of fly ash used was 2.39, and the amount of remaining on 45-micron sieve was 12%.

Sodium hydroxide is used as an activator; it was a technical grade chemical and its purity more than 97%. The amount of activator was selected as 10% of the mass of fly ash in the mixture. For the preparation of mortar mixture, standard Rilem sand was employed. The number of materials needed for standard mortar mixture to obtain three 40x40x160 mm^3 sized prismatic samples were given in Table 2.

Table 1. Chemical compositions for fly ash

Oxide	SiO_2	$Al2O_3$	$Fe2O_3$	CaO	SO_3	Na_2O
(%)	61.81	19.54	7.01	1.77	0.31	2.43

Table 2. Mixture compositions (g)

Sand	Fly ash	NaOH	Water
1350	450	78	150

3. RESULTS AND DISCUSSIONS

3.1 Unit weight and workability of geopolymer mortars

Fresh alkali-activated geopolymer mortar was measured as 200 mm from mini flow test for mortar. There was no workability problem during the compaction of fresh mortar. Hardened mortar unit weight was found to be about 2.2-2.4 g/cm^3.

3.2 Compressive strength of geopolymer mortars

Compressive strength measurement results according to TS EN 1015-11 [28] obtained from alkali-activated class F fly ash-based geopolymer mortar were presented in Table 3. Initial heat cured samples showed satisfactory compressive strength development beginning from 24 hours till six months. Initial heat curing for 24 hours at 75^{o}C developed 32.4 MPa compressive strengths at the first day. However, samples continued to development of strength for further curing duration until six months at laboratory conditions. For further six months curing after 24 hours of first heat curing at 75^{o}C, geopolymer mortar samples developed an average 45.5 MPa compressive strength value.

It is observed that geopolymer mortar samples cured at ambient temperature could not develop proper compressive strength till the 7th day; however, it developed 2.1 MPa compressive strength at 28 days curing duration.

In long term curing duration (90-day and 180-day) at ambient condition, geopolymer mortar samples developed 17.2 MPa and 27.1 MPa compressive strength for 90-day and 180-day, respectively (see Table 3 and Figure 1).

A comparison between initial heat cured samples and without heat cured samples were made. The comparison showed that at the end of three months, curing duration, samples cured at ambient conditions developed 40% of compressive strength of heat-cured mortar samples. In addition, at the end of six months' curing duration, samples cured at ambient conditions developed 60% of the compressive strength of heat-cured mortar samples. Geopolymer samples cured at ambient conditions could not develop compressive strength in a short-term curing duration of up to 7 days; however, it developed entirely satisfactory compressive strength in longer-term curing duration.

Table 3. Compressive strength development of mortar (MPa)

Curing time interval (day)	Room temperature curing	Heat curing (75°C)
1	0	32.4
3	0	36.5
7	0	35.1
28	2.1	41.4
90	17.2	42.4
180	27.1	45.5

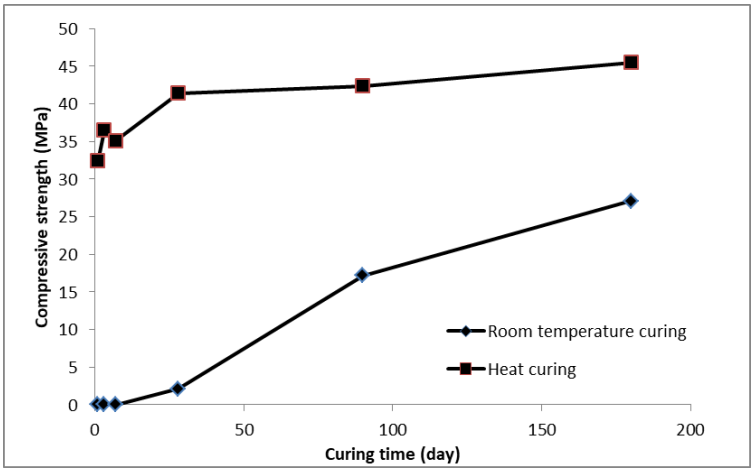

Figure 1. *Compressive strength vs curing time*

In very long term curing duration (1 year or more), geopolymer mortar samples cured at ambient conditions could develop equivalent compressive strength to heat-cured samples. Based on compressive strength development, it can be concluded that heat-cured class F fly ash-based geopolymer mortar with its adequate compressive strength can be employed in construction and building areas as environmentally friendly material. However, utilizing a non-heat cured Class F fly ash-based geopolymer in that area is not practical due to slow compressive strength development.

3.3 Flexural strength of geopolymer mortars

Flexural strengths of alkali-activated class F fly ash-based geopolymer mortar according to TS EN 1015-11 [28] were given in Table 4. Flexural strength of 24 hours initially, heat cured samples was 9.2 MPa. Flexural strength of samples was increased by an increase of further curing time up to six months. However, flexural strength development was found to fluctuate in time, attributed to experimental variations.

For further six months curing after 24 hours of initial heat curing at 75°C, geopolymer mortar samples developed an average 13.3 MPa flexural strength value. Moreover, geopolymer mortar samples cured at ambient temperature could not develop proper flexural strength till the 7th day; however, it developed 2.7 MPa flexural strength at 28 days curing duration.

In long term curing duration (90-day and 180-day) at ambient condition, geopolymer mortar samples developed 8.9 MPa and 10.2 MPa flexural strength for 90-day and 180-day, respectively (see Table 4 and Figure 2).

Table 4. Flexural strength development of mortar (MPa)

Curing time interval (day)	Room temperature curing	Heat curing (75°C)
1	0	9.2
3	0	12.6
7	0	9.9
28	2.7	12.7
90	8.9	15.1
180	10.2	13.3

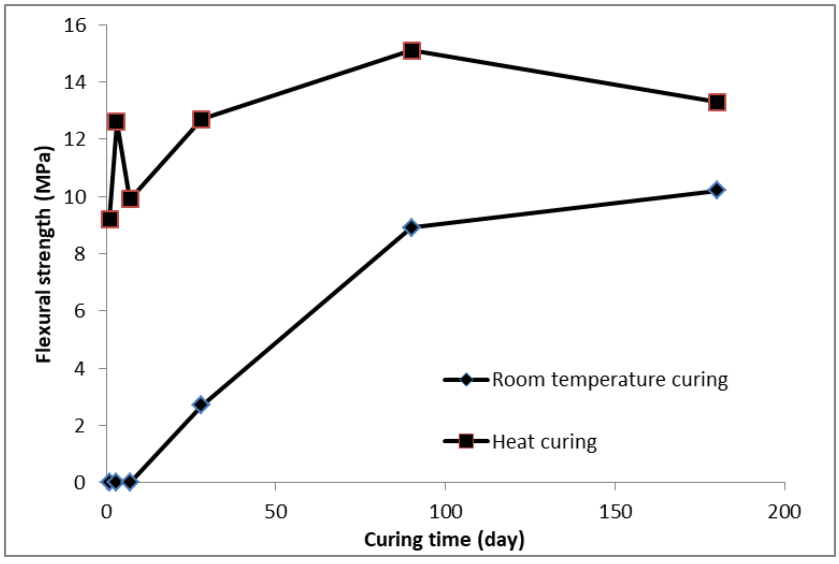

Figure 2. *Flexural strength vs curing time*

Comparison between initial heat-cured flexural strength and non-heat cured flexural strength showed that at the end of three months and six months, curing duration, mortar cured at ambient conditions developed mora than 50% of flexural strength of heat-cured mortar samples.

Geopolymer samples cured at ambient conditions could not develop flexural strength in short term curing duration up to 7 days; however, it developed entirely adequate flexural strength in longer-term curing duration.

In very long term curing duration (1 year or more), it is expected that geopolymer mortar samples cured at ambient conditions could develop equivalent flexural strength to heat-cured samples.

Flexural strength development also proved that heat cured geopolymer mortar mixtures studied in this work could be utilized in construction and building area with satisfactory flexural strength development. However, utilizing non-heat cured geopolymer mixtures in that area is not practical due to slow flexural strength development.

A comparison was made between strength obtained from the study and published work. This is given in the following.

Olivia and Nikraz [20] activated class F fly ash with 14 molar concentration of sodium hydroxide and sodium silicate solution at 75^{o}C heat curing for 24 hours. They obtained compressive strength in the order of 50-60 MPa, flexural strength in the order of 7-8 MPa.

Ryu et al. [21] activated class F fly ash with 9 molar concentration of sodium hydroxide and sodium silicate solution at 60^{o}C heat curing for 24 hours, they obtained compressive strength in the order of 40, splitting strength in the order of 2-4 MPa

Vora and Dave [22] reported the compressive strength of geopolymer mixtures between 32 and 46 MPa at 7 days, the lowest value corresponds to 8 molar sodium hydroxide concentrations, and the highest value corresponds to 14 molar sodium hydroxide concentrations. They reported 35 MPa compressive strengths for 10 molar concentration of sodium hydroxide. The cured mortar samples at 75^{o}C for 24 and 48 hours.

De Vargas et al. [23] reported compressive strength between 8 and 28 MPa, for alkali-activated class F fly ash-based geopolymer mortar cured at 80^{o}C for 24 hours initial heat curing. Further curing at laboratory conditions was applied up to 3 months.

Skvara et al. [24] activated low calcium brown fly ash with sodium hydroxide and sodium silicate solution and prepared geopolymer mortar. The compressive strength of the geopolymer produced was 40 MPa at 28 days of laboratory curing after 12 hours of initial heat curing at 80ºC.

Gorhan and Kurklu [15] reported compressive strength in the order of 15 and 25 MPa and flexural strength in the order of 5, and 8 MPa for alkali-activated fly ash mortar. Molarities of activators were 3, 6, and 9. Heat curing was applied at 65 and 85ºC for 24 hours.

Compressive and flexural strength obtained in the study was in the order of 40 MPa and 12 MPa, respectively. The compressive strength reported by Olivia and Nikraz [20] was obtained using 14 molar activators and was found to be somewhat higher than the current results obtained using 10 molar activators; thus, the difference was attributed to higher molarity of their work. Compressive strength of this work was found to be in-line with results reported by Ryu et al. [21] and Vora and Dave [22] and Skvara et al. [24] However, the current compressive strength value was higher than the strength reported by De Vargas et al. [23] and Gorhan and Kurklu [15].

4. CONCLUSIONS

The following conclusions were made from the study.

1. NaOH activated geopolymer mortar mixtures made with fly ash and cured for 28 days at ambient condition did not gain adequate compressive and flexural strength. Therefore, it is proved and concluded that the geopolymerization reaction of alkali-activated class F fly ash-based geopolymer is quite slow.
2. Samples cured at room temperature developed significant compressive and flexural strength at long term curing duration.
3. It was seen that heat cured samples continuously developed high compressive and flexural strength of 24 hours up to six months.
4. It is concluded that heat cured fly ash based geopolymer samples can be utilized in construction materials area while utilization of non-heat cured samples is not practical due to its longer curing needs.

CONFLICTS OF INTEREST

No conflict of interest was declared by the authors.

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