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## Doğal Kürleme Şartlarında Alkali Aktivatör Oranının Jeo-Polimer Betona Etkisinin İncelenmesi

Investigation of Alkaline Activator ratio on Geopolymer Concrete under Ambient Curing Regime

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### Doğal Kürleme Şartlarında Alkali Aktivatör Oranının Jeo-Polimer Betona Etkisinin İncelenmesi

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#### Öz

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#### Anahtar Kelimeler

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#### Keywords

Geopolymer concrete Alkali-activate ratio Compressive strength Tensile strength Ambient curing Jeopolimerizasyon, çeşitli atık malzemeleri sıradan betona kıyasla daha iyi mekanik ve dayanıklılık özelliklerine sahip yapı malzemelerine dönüştürebilmektedir. Bu çalışmada, cüruf (GGBFS) uçucu kül (FA) le karıştırılarak ve farklı alkali-aktivatörler (AL) farklı bağlayıcı mineral/puzolanik malzemeler (B) aktivatör oranları (AL/B) ile harmanlanarak, jeopolimer beton (GPC) hazırlanmasında kullanılmıştır. Sodyum silikat (Na<sub>2</sub>SiO<sub>3</sub>) ve sodyum hidroksit (NaOH) çözeltisi, 12 M lik sabit NaOH konsantrasyonu ve Na<sub>2</sub>SiO<sub>3</sub>/NaOH oranı 2.5 ile alkali-aktivatörü olarak kullanılmıştır. GPC karışımları, farklı AL/B oranlarıyla (0.33, 0.37, 0.40 ve 0.42) hazırlanmış ve GPC ye aktivatör oranının etkisinin incelenmesi açısından, sabit bir GGBFS/FA oranı kullanılmıştır. Sertleşmiş GPC iki farklı yaşta (7, 28 gün) ve yoğunlukta basınç ve çekme mukavemeti deneylerine tabi tutulmuştur. Bütün karışımlar doğal kürleme şartlarında sertleştirilmiştir. Deneysel sonuçlar, çeşitli AL/B oranlarının GPC'nin basınç dayanımı üzerinde etkili olduğunu göstermiştir. AL/B oranını düşerken basınç dayanımı artmaktadır. 28 günde 57.76 MPa'ya kadar basınç dayanımı gösteren 0.375 optimum AL/B oranıdır. Ayrıca, GPC nin yoğunluğu 2203 ila 2279 kg/m<sup>3</sup> arasında değişmektedir. Sonuç olarak, basınç dayanımı ve çekme dayanımı arasında bir ilişki önerilmiştir.

#### Investigation of Alkaline Activator ratio on Geopolymer Concrete under Ambient Curing Regime

#### Abstract

Geopolymerisation allows the reuse and refurbishment of an extensive range of waste materials into building elements with excellent mechanical and durability properties, giving it potential as an environmentally friendly alternative to conventional cement concrete. In this research paper, ground granulated blast furnace slag (GGBFS) was blended with fly ash (FA) to form binder content (B) and combined at different ratios with a solution of various alkaline activators (AL) to make Geopolymer concrete (GPC). The AL solution consisted of a mixture of sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) and sodium hydroxide (NaOH), with a stable NaOH concentration of 12 M (Molar) and a Na2SiO3/NaOH mass ratio of 2.5. The GPC mixes were prepared with different AL/B ratios (0.33, 0.375, 0.40 and 0.42), and a constant partial GGBFS/FA replacement ratio was used. All of the mixes were cured under ambient conditions. To analyses the impact of the AL/B ratio on the GPC, the compressive and splitting tensile strengths of hardened GPC were investigated and the density was measured at two different ages, 7 and 28 days. The experimental results presented that varying the AL/B ratio had an influence on the compressive strength of GPC. Compressive strength was higher at an AL/B ratio of 0.375 than at both higher and lower ratios. At this optimum AL/B ratio, compressive strength of up to 57.76 MPa was recorded on the 28th day. Additionally, the hardened density of GPC ranged between 2203 and 2279 kg/m<sup>3</sup>. Finally, a relationship is proposed between compressive strength and tensile strength.

#### **1. INTRODUCTION**

The conventional cement industry consumes a large quantity of energy due to the use of rotary kilns. These kilns ensure the continuous production of cement but consume a large amount of fuel, resulting in carbon dioxide ( $CO_2$ ) emissions that contribute to global warming. Worldwide, the annual contribution of the

manufacturing of Ordinary Portland Cement (OPC) to greenhouse gas emissions is estimated at approximately 1.350 billion tons, or nearly 7.0 % of global greenhouse gas emissions [1, 2]. At the same time, approx. one billion tons of fly ash (FA) is wasted annually world-wide in coal-fired electric power stations [3]. In the best-case scenario this material is stockpiled, but more often than not, it is merely landfilled. In both cases, it creates a serious environmental hazard. One suggested use for fly ash is as an artificial pozzolan, by mixing it with cement at specified percentages to produce normal and high-strength concrete [4, 5]. A second waste material called ground granulated blast furnace slag (GGBFS) is available in huge quantities sufficiently large for industrial use throughout the world. It is an accidental by-product of the steel fabrication process and includes lime and calcium–magnesium aluminosilicate. GGBFS can be used as a pozzolanic material in ordinary concrete, but in Geopolymer concrete (GPC), it can be used as a binder (B), resulting in reduced greenhouse gas emissions in the world [6, 7].

The question of how to develop an eco-friendly construction material with mechanical and chemical properties similar to or even better than OPC has been a focus of research, and Geopolymer has been celebrated as one of the most promising materials. Developed by the French scientist and engineer Prof. Joseph Davidovits in 1970, alkali-activated or "Geopolymer" concrete is an environmentally friendly alternative to traditional ordinary Portland cement concrete made from by-product aluminosilicate components such as FA, GGBFS, bottom ash, and rice husk ash with an alkaline activator (AL). Davidovits reported variations in the physical and chemical behaviour of GPCs produced with FA from different sources and also studied the effect of different activators [8–11]. Following the publication of Davidovits' research results, many researchers followed suit, advancing the development of GPC. Several types of AL have been used to stimulate the aluminosilicate binders in order to produce GPC. A mixture of sodium and potassium hydroxide (NaOH and KOH) or liquid sodium and calcium silicate (Na2SiO3 and Ca2SiO3) is commonly used to stimulate aluminosilicate in the manufacture of GPC. Fernández-Jiménez & Palomo [12] has been noticed that the AL properties are dependent on the concentrations and types of AL elements. Vora & Dave [13] mixed two different types of ALs in solution, 85% NaOH 8 M (Molar) and 15% Na<sub>2</sub>SiO<sub>3</sub>, to make GPC. All GPC samples were cured at a fixed temperature of 85°C with a 20-hour cure interval. They noted that the GPC produced with the mixed AL solution exhibited greater compressive capacity than that produced with a single AL solution. In addition, they observed that the bond strength of GPC is greater than that of OPC. They also considered the impacts of several factors affecting the compressive strength of GPC [13]. Lloyd et al. [14] focused on the analysis of microstructural GPC with silicate-based and NaOHbased solutions. They used Class F FA mixed with a Na<sub>2</sub>SiO<sub>3</sub> or NaOH solution, river sand and crushed aggregate to make GPC. A more homogeneous microstructure was detected when silicate-based solution was used. They using Class F FA and mixed of Na<sub>2</sub>SiO<sub>3</sub> and NaOH solutions to making Geopolymer also they used river sand and crushed aggregate. The samples were cured using a furnace at 24 different temperatures and for different durations. Muñiz-Villarreal et al. [15] studied the impact of curing temperatures on the Geopolymer specimens. They documented that the ideal geopolymerisation process utilizes furnace curing at 60°C. Al-Rawi and Tayşi [16] used both FA and GGBFS as the source components and stimulated them with a combination of Na<sub>2</sub>SiO<sub>3</sub> and a constant concentration of NaOH solution to produce self-compacting Geopolymer concrete (SCGC). FA was substituted with GGBFS at five levels of replacement, being 0, 25, 50, 75, and 100% by weight. They concluded that using GGBFS in the mixes significantly improved the compressive strength.

Several previous studies have been limited to the use of alkali activated concrete in the construction of ready mixed concrete members. Therefore, the development of a method for curing GPC at ambient temperatures will broaden its applications in the construction of a vast range of structures and facilities. Many benefits can be obtained through the use of ambient cured GPC, including reduced  $CO_2$  emissions, reduced costs and in-situ casting. This paper presents a concise summary of the experimental data, including material properties, mix method, properties of specimens and testing procedures. The aim of this

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investigation is to investigate the impact of AL/B ratios on the properties of GPC with respect to the workability, compressive and splitting tensile strengths and density of ambient-cured GPC at different ages (7 and 28 days). Finally, a correlation between the splitting test results and compressive capacity is proposed, to predict the tensile strength and compared with different code provisions and previous studies.

#### 2. EXPERIMENTAL APPROACHES

#### 2.1 Materials

The raw components used in GPC mixing consisted of five parts: alkaline activators, binder, crushed sand, crushed rock and superplasticizer. The AL was a uniform sodium-hydroxide-based solution prepared by mixing sodium hydroxide solution (NaOH) with sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>). The NaOH was procured in flake form with 98% purity, and the Na<sub>2</sub>SiO<sub>3</sub> consisted of Na<sub>2</sub>O, 10.6 %; SiO<sub>2</sub>, 26.5% and H<sub>2</sub>O, 66.1%; with a fresh density of 1.390 g/ml at 25°C. FA and GGBFS obtained from Iskenderun, Adana, Turkey, corresponding to ASTM C 618 and ASTM C 989, respectively, were used as the binder components. Their chemical compositions were investigated via X-ray fluorescence (XRF) analysis. The results of this analysis are presented in Table 1. Commercial local crushed sand and crushed rocks were used in the mixes. Figure 1. Illustrates the physical properties and gradation curves of the aggregates. Finally, high-range water-reducing MasterGlenium® RMC 303, a new generation of polycarboxylic-based superplasticizer, was used in all mixtures.

SO <sub>3</sub> LOI	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	CaO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	Materials
0.1 2.07	0.1	2.45	0.7	1.76	25.79	7.85	21.14	62.4	Fly ash (%)
0.68 2.74	0.68	0.17	2.4	7.63	34.19	1.28	10.6	40.4	GGBFS (%)
0.00	0.00	0.17	2.1	7.05	54.17	1.20	10.0		LOI: Loss of ig

Table 1. The chemical composition of GGBFS and FA.

#### 2.2 Mix proportions

The concentration of NaOH solution used was 12M, as this was the optimal concentration reported from previous studies [18–21]. To prepare the NaOH solution, sodium hydroxide flakes were mixed with tap water. The solution was left to settle for 18 hours, after which we prepared the AL solution by mixing the NaOH and Na<sub>2</sub>SiO<sub>3</sub> solutions together and leaving them to settle again for approximately 4 hours. In general, we noted that the AL was ready to use after 4 hours but we have important limit should be taken on account to prevent flash setting when prepare the GPC mixture. Additionally, based on the literature studied, a GGBS/FA ratio of 75/25 was used to increase the initial setting time and workability of the mixes, and the Na<sub>2</sub>SiO<sub>3</sub>/NaOH mass ratio was set at 2.5 [17]. The details of the studied GPC mixtures are summarized in Table 2. For the GPC specimens, the crushed sand and aggregate were mixed for 2 minutes using a high shear capacity concrete mixer, then the FA and GGBFS were added, followed by another 2 minutes of mixing. Then, AL was added to the dry mix, and the wet components were mixed together for approx. 4 to 5 minutes. Finally, high-range, water-reducing superplasticizer was added gradually.

Freshly mixed GPC was poured into 100 mm cubes and  $100 \times 200$  mm cylindrical moulds. After the casting process, the specimens were covered with heavy-duty nylon bags to prevent shrinkage. The moulds were left for 24 hours in the lab climate and then they were de-moulded. After that, the actual treatment conditions at construction sites were simulated, which was done by leaving the samples in the laboratory

until the testing appointment. After 28 days, the GPC samples were weighed to determine the hardened density. The samples of GPC were tested at 7 and 28 days depending on concrete material test standards.



Figure 1. Gradation curves of crushed sand and aggregate.

Mix Code	Slag	Fly Ash	AL	Crushed Sand	Coarse Agg.	Water Reducer %	Alkaline / Binder %
G-1	318.75	106.25	140	760	1187	5	0.3
G-2	318.75	106.25	160	752.19	1174.81	5	0.375
G-3	318.75	106.25	169	748.68	1169.32	5	0.4
G-4	318.75	106.25	178	745.17	1163.83	5	0.42
Ν	NA	NA	NA	560	1087	1	0.44
NA: Not	t required, m	ix N its norm	al concrete	e with cement 36	65 kg/m <sup>3</sup>		

Table 2.	GPC mix	proportions	$(kg/m^3)$	).

#### **3. RESULTS AND DISCUSSIONS**

#### 3.1 Workability Test

Previous studies reported lower workability values for GPC than for OPC, attributing this to clingy properties of the GPC mix caused by the chemical composition of the silicate. Here, the workability of mixtures was measured according to ASTM C134. The mixtures containing a low AL/B ratio were noted to be coarser and stiffer, resulting in a relatively lower slump value in comparison with conventional concrete mixtures made with cement. The workability index ranged between 80 and 138 mm for the fresh GPC it can be noticed in Figure 2.



Figure 2. Effect of AL/B ratio on the slump value.

#### 3.2 Compressive Strength

According to building codes, compressive capacity is one of the central properties characterizing normal concrete. Mix design is the process of designing a concrete mix to provide a given strength. A compressive strength of 25 MPa at 28 days is well established as the primary criterion of concrete quality, representing a fundamental key to meet the requirements for use in construction. In this work, compressive strength was determined by testing concrete cubes with a 3000 KN BESMAK uniaxial compression machine. A rate of loading of 0.33 MPa/S was used in the test of compressive strength, in accordance with BS-1881. The experimental test results for compressive capacity are illustrated in Figure 3.



Figure 3. The compressive strength of GPC under ambient curing conditions at 7 and 28 days.

In general, the compressive capacity of the GPC increased as the AL/B percentage increased from 0.3 to 0.375. The 7-day compressive strengths of G1, G2, G3, G4 and ordinary concrete were 31.13, 46.53, 26.83, 25.26 and 28.44 MPa, respectively. It can be observed from Figure 3. That the compressive strength was highest when the AL/B ratio was 0.375. Then, at even higher AL/B ratios of 0.40 and 0.42, the compressive strength decreased. The reason for this reduction in compressive strength can be identified as the higher AL/B ratios in G3 and G4. Excess alkali activator can increase the amount of water in the mixture, which interferes with the process of geopolymerisation. The percent increase in compressive capacity from 7 to 28 days were approximately 25.13%, 19.44%, 22.92%, 25.11% and 22.32% for G1, G2, G3, G4 and N, respectively. Mix G2 was identified as the optimum mix, summarized as having an excellent compromise between workability and compressive and tensile strengths. Overall, the GPC mixtures exhibited acceptable corrosion resistance and good compressive strength compared with the minimum compressive strength of 35 MPa defined by building codes.

#### 3.3 Splitting Test

The indirect Brazilian test is one of the most common processes for determining tensile stress capacity. As we know, normal concrete is relatively low in tensile strength, which is the reason for using different types of reinforcement to improve its tensile strength capacity. However, the tensile strength of concrete is not as reliable as the compressive strength, it is responsible for the overture and stretch of cracks, shearing, steel anchor behavior and temperature effects in concrete elements. In GPC its same situation will repeated. The splitting test was performed on the cylindrical moulds as per ASTM C496, which provides for the testing of cylindrical samples with a loading rate between 0.70 to 1.40 MPa/min. In this paper, a load rate of 1 MPa/min was used. Figure 4. Illustrates the influence of the AL/B ratio on tensile capacity at different specimen ages. It can be noted that the tensile strength of mix G2, which had a 37.5% AL/B ratio, was higher than those of the other mixtures. The average tensile capacity of mix G2 was 4.03 MPa.



Figure 4. Tensile strength at 7 days and 28 days.



Figure 5. The transition zone in a G2 specimen.

The transition zone and aggregate particles of a G2 specimen are shown in Figure 5. As the figure shows, the failure mechanism of the GPC specimen occurred along the paste and through the aggregates; that is to say, the transition area of Geopolymer paste had strong bonds and bonded the aggregate particles [22].

#### 3.4 Density

The GPG proportions were based constructed on a target density of 2300 kg/m<sup>3</sup> and constructed by varying the ratios of AL/B ratio (0.33, 0.375, 0.40 and 0.42) and constant ratio of fine to coarse aggregate (0.4 and 0.6). The 28day densities were calculated from standard modules. It can be noted from the hardened values that the hardened density of mix G2 increased with a decrease in the AL/B ratio but began to decline when the AL/B ratio was increased to 0.42. The different of AL/B ratio can increase the amount of water in the mixture as a result, this change in densities occurred. However, the GPC mixes have acceptable hardened densities when compared with ordinary concrete. The hardened densities of GPC and normal concrete are presented in Figure 6.



Figure 6. The average hardened densities of GCP Mixtures.

#### 3.5 Relationship of mechanical properties

Building codes specify two important values; the first is compressive strength and the second, related to compressive strength, is called splitting tensile strength. The latter can be measured using a splitting tensile test or estimated using empirical equations. In this section, a relationship is established between compressive and splitting tensile strengths depending on experimental data. The empirical equations obtained by previous studies and specification standards are summarized in Table 3. Based on the basic equations reported by [23-26], several researchers have proposed other constituent equations [27-30]. Depend on existing standards and previous studies [23–31], the compression strength values measured in this study were converted to fit with the equations included in Table 3. It can be noted that the splitting tensile strength was proportional to the square root of compressive capacity. It can be seen from the experimental work and correlation results presented here that the splitting tensile strength established for the GPC is lower relative to the compressive strength than is proposed by ACI building code 318-14 [23], ACI 363R-92 [24], and EC-2 [25]. Additionally, the relationship approaches the one obtained from the empirical equations of FIB model code [26] and Oluokun et al. [27], whereas the equations of Sofi et al. [28], Ryu et al. [29] and Lee & Lee [30] were less than the results obtained from laboratory tests. As a result, it is found that the constituent equations could be applied to GP concrete with a slight percentage error. Accordingly, an equation is proposed to estimate the splitting tensile strength of GPC cured under an ambient regime. The most striking results emerge from the experimental data, the empirical equations reported in Table 3. And the proposed formula in Figure 7. Illustrating the acceptability of the proposed equation.

<b>Empirical Equations</b>	Range (MPa)	Comments	
$f_{sp} = 0.590 \sqrt{f_c}$	$21 \le f_c \le 83$		
$f_{sp} = 0.560 \sqrt{f_c}$	$f_{ck} \leq 50$		
$f_{sp} = \frac{1}{3} f_{ck}^{2/3}$			
$f_{sp} = 0.3 f_{ck}^{2/3}$	<i>f</i> <sub>c</sub> < 83		
$f_{sp} = 0.294^{0.69}$	$3.5 \le f_c \le 63$	Normal weight concrete	
$f_{sp} = 0.48 \sqrt{f_c}$		Alkali-activated concrete	
$f_{sp} = 0.17^{3/4}$		Fly ash-based geopolymer	
$f_{sp} = 0.45 \sqrt{f_c}$		Alkali-activated concrete	
$f_{sp} = 0.2751 f_c^{0.697}$		Geopolymer Concrete*	
ength (MPa), $f_c$ , $f_{ck}$ : Cylir	der Compressive stre	ngth (MPa)	
	$f_{sp} = 0.590 \sqrt{f_c}$ $f_{sp} = 0.560 \sqrt{f_c}$ $f_{sp} = \frac{1}{3} f_{ck}^{2/3}$ $f_{sp} = 0.3 f_{ck}^{2/3}$ $f_{sp} = 0.294^{0.69}$ $f_{sp} = 0.48 \sqrt{f_c}$ $f_{sp} = 0.17^{3/4}$ $f_{sp} = 0.45 \sqrt{f_c}$ $f_{sp} = 0.2751 f_c^{0.697}$	$f_{sp} = 0.590 \sqrt{f_c} \qquad 21 \le f_c \le 83$ $f_{sp} = 0.560 \sqrt{f_c} \qquad f_{ck} \le 50$ $f_{sp} = \frac{1}{3} f_{ck}^{2/3} \qquad$ $f_{sp} = 0.3 f_{ck}^{2/3} \qquad f_c < 83$ $f_{sp} = 0.294^{0.69} \qquad 3.5 \le f_c \le 63$ $f_{sp} = 0.48 \sqrt{f_c} \qquad$ $f_{sp} = 0.17^{3/4} \qquad$ $f_{sp} = 0.45 \sqrt{f_c} \qquad$ $f_{sp} = 0.2751 f_c^{0.697} \qquad$	

 Table 3. Building codes and published empirical equations of splitting tensile strength and Proposed formula.

\* Geopolymer Concrete with GGBFs/FA ratio is 75/25 % with 12 M NaOH and Na<sub>2</sub>SiO<sub>3</sub> at a mass ratio of 29:7 with different Al/B ratio and cured at ambient conditions.



Figure 7. Comparison of the proposed equation with different building codes and published studies at age 28 days.

#### 4. CONCLUSIONS

This study focused on the impact of AL/B ratios on the mechanical properties and workability of GPC and a method for curing two-part GPC mixes at ambient temperature using GGBFS and FA (75% and 25% by weight of the overall binder, respectively) as a binder in combination with the available chemical component Na<sub>2</sub>SiO<sub>3</sub> blended with NaOH as an AL. Based on the data obtained from experimental work and correlation studies, the following conclusions can be summarized.

• GPC workability increased with the increase in liquid content in mixtures with a higher AL/B ratio. On the other hand, it was noted that these mixtures are in accordance with the specifications limit.

• The compressive capacity of GPC increased significantly as the AL/B ratio increased from 0.3 to 0.375, and decreased at higher ratios. Additionally, the percentage of GGBFS in the binder had a considerable influence on the compressive capacity at initial ages. We noted that at 28 days, the compressive strength of GPC containing a binder of 75% GGBFS and 25% FA, an AL/B ratio of 0.375, and a Na<sub>2</sub>SiO<sub>3</sub>/NaOH ratio of 2.5 reached up to 57.76 MPa when cured in the lab climate for which the recorded temperature ranged from 19 to 24°C.

• The GPC tensile strength, as measured by the Brazilian splitting test, increased with the increase of compressive capacity. We found that the splitting tensile strength could be predicted with little error by apply the compressive capacity in proposed equation.

• Compressive capacity and workability as the performance criteria, the highest-performing mixture under ambient curing conditions was mix G–2. It can be called an optimal mixture and meets the requirements for use on construction sites. However, more research is needed that focuses on the early and final setting times of GPC and on improving the setting time to converge with that of normal concrete.

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