

PERFORMANCE OF HIGH CALCIUM FLY ASH BASED GEOPOLYMER CONCRETE IN CHLORIDE ENVIRONMENT

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ABSTRACT: This paper presents research on geopolymer concrete performance using high calcium fly ash in a chloride environment. The experiment, since there is no specific geopolymer concrete standard up to the present, uses Ordinary Portland Cement concrete standard. Concrete used in chloride environment requires a minimum of 35 MPa concrete strength and maximum water-cement ratio of 0.4. In this study, three compositions of geopolymer concrete were prepared, namely mix-1, mix-2, and mix-3. The mixes were produced by mixing fly ash, alkaline activator, sand, gravel, and water. Sodium metasilicate granular pentahydrate and sodium hydroxide flake were used as the alkali activators. Two percent of sucrose was added to the fly ash for improving geopolymer concrete workability. Geopolymer concrete curing was performed by placing test specimens into a closed plastic box at 28-30°C room temperature for 7, 14, and 28 days. Workability tests showed that mix-3 had a slump of 227 mm, which was only slightly larger than mix-1 and mix-2. Permeability test showed that the three mixes had almost the same quality at 28 days. Based on resistivity test, mix-3 had the best performance at 7, 14, and 28 days. In term of compressive strength, mix-3 had the highest strength at 7, 14, and 28 days. Furthermore, mix-3 also had the highest pH compared with the other two mixes.

Keywords: Performance, Geopolymer Concrete, Alkali Activator.

1. INTRODUCTION

Geo-Polymer Concrete (GPC) is an innovative material, which doesn't use Ordinary Portland Cement (OPC) in its manufacturing process. In the process of GPC manufacture, the use of OPC is replaced with Fly Ash (FA) material, kaolin, and other materials by adding alkali activators, which were sodium silicate solution (Na_2SiO_3) and sodium hydroxide solution (NaOH). GPC manufacturing process provides some benefits. These are the utilization of FA waste materials, the reduction of CO_2 emissions occurring during the POC production, and the reduction of OPC usage for the future [1-3].

GPC possesses 8%-12% higher compressive strength performance and it exhibits better resistance in seawater environment. However, there is some controversy about its corrosion resistance. Based on the half-cell potential test, GPC is more susceptible to corrosion compared with Ordinary Portland Cement Concrete (OPCC) [4]. Other studies show that GPC is more resistant to corrosion than OPCC [5]. GPC is proposed as an effective substitute for OPCC in reinforced concrete structures located in a marine environment or one which has extensive exposure to salt or brackish water [6]. GPC delivers greater protection against corrosion compared with OPCC when concrete is contaminated with chloride [7].

The wet method is a commonly GPC manufacturing method used by many GPC researchers. GPC mixing in wet method is carried out through a process of activator solution making

produced from sodium silicate solution and sodium hydroxide solution mixing added with FA, sand, and coarse aggregate [1-3,8-14].

The main problem of high calcium FA usage is the concrete setting which is relatively fast [15] since fast setting influences the application of GPC in real structure.

The objective of this study is to produce GPC with reasonable workability for the implementation in a chloride environment. This study uses the normal concrete standard of ACI 318-2011 [16], which requires a minimum of 35 MPa concrete strength and a maximum of 0.4 water-cement factor for concrete used in a chloride environment.

2. RESEARCH METHOD

This research used three compositions of GPC. The analysis of GPC performance covered tests on slump, permeability, resistivity, compressive strength, and pH.

2.1 Specimens

In this study, the dry method of GPC manufacturing was developed to overcome the fast setting issue. The dry method GPC manufacturing began with the calculation of wet method GPC manufacturing composition. The composition can be seen in Table 1. Next, the calculation based on wet method was converted into the calculation based on dry method GPC manufacturing composition. The

converted data is presented in Table 2.

Using the composition shown in Table 2, several numbers of 150 x 300 mm cylinder GPC specimens were made.

Table 1 GPC wet method composition kg/m³

Composition	Mix-1	Mix-2	Mix-3
Fly Ash	468	468	550
Na ₂ SiO ₃	180	126	115.5
NaOH	72	126	115.5
Fine aggregate	672	672	446.15
Coarse Aggregate	1008	1008	1025.65
Water	-	-	-
Superplasticizer	9.36	9.36	11

Table 2 GPC dry method composition kg/m³

Composition	Mix-1	Mix-2	Mix-3
Fly Ash	468	468	550
Na ₂ SiO ₃	96.41	67.49	61.86
NaOH	31.97	55.94	51.28
Fine aggregate	672	672	446.15
Coarse Aggregate	1008	1008	1025.65
Water	123.62	128.57	117.86
Superplasticizer	9.36	9.36	11

In this experiment, mix-1 was made from 70 percent of combined aggregate (fine aggregate and coarse aggregate) and 30 percent of geopolymer binders. The composition of the geopolymer binders consisted of 65 percent of fly ash and 35 percent of mass of activator (Na₂SiO₃+NaOH). The activator composition ratio of Na₂SiO₃: NaOH was 2.5:1. Two percent of sucrose by weight of FA was added for improving GPC workability.

Mix-2 was made from 70 percent of combined aggregate and 30 percent of geopolymer binders. The composition of geopolymer binders was 65 percent of fly ash and 35 percent of the mass of activator (Na₂SiO₃+NaOH). The activator composition ratio of Na₂SiO₃: NaOH was 1:1. For improving GPC workability, two percent of sucrose by weight of FA was then added to this mix.

Finally, mix-3 was produced from 65 percent of combined aggregate and 35 percent of geopolymer binders. The composition of geopolymer binders was 70 percent of fly ash and 30 percent of (Na₂SiO₃+NaOH) activator masses. The ratio of activator composition of Na₂SiO₃: NaOH was 1:1. Like mix-1 and mix-2, two percent of sucrose by weight of FA was added.

2.2 Materials

2.2.1 Fly ash

The FA used in this study was obtained from PT. Paiton Jawa Power Probolinggo. Table 3 presents the chemical composition of FA used in the experiment, which was determined by X-Ray Fluorescence (XRF). The XRF test result indicated that the CaO level was higher than 10%. Therefore, based on [17] the fly ash can be categorized as type C fly ash.

Table 3 Chemical composition of Fly Ash

Oxide	(%)	Oxide	(%)
SiO ₂	33.89	SrO	0.0955
CaO	19.86	Cr ₂ O	0.039
Fe ₂ O ₃	17.68	Cl	0.0213
Al ₂ O ₃	12.54	ZnO	0.0162
MgO	9.023	ZrO ₂	0.011
SO ₃	2.16	CuO	0.008
Na ₂ O	2.04	NiO	0.006
K ₂ O	1.03	As ₂ O ₃	0.006
TiO ₂	0.691	V ₂ O ₅	0.006
BaO	0.215	Rb ₂ O	0.003
MnO	0.168	Br	0.003
P ₂ O ₅	0.154		

2.2.2 Sodium hydroxide

The experiment utilized sodium hydroxide (NaOH) 98% flake for the materials of the alkaline activator. The specification of sodium hydroxide can be seen in Table 4.

Table 4 Specification of NaOH

Parameter	Value	Unit
NaOH	98.11	%
Na ₂ CO ₃	0.26	%
NaCl	122.32	Ppm
Fe	4.88	Ppm

2.2.3 Sodium silicate

Another alkaline activator used in this study as the alkaline activator was Sodium Metasilicate Pentahydrate (granular). The specification is presented in Table 5.

In the final process of making GPC, FA materials, NaOH, and Sodium Metasilicate Pentahydrate were grounded together in a ball mill machine to produce geopolymer cement.

Table 5 Specification of Sodium Metasilicate Pentahydrate (granular)

Parameter	Value	Unit
Sodium Oxide (Na ₂ O)	29.04	%
Silica (SiO ₂)	29.12	%
Water insoluble	0.02	%
Fe	57	Ppm
Whiteness	93	%
Bulk Density	0.96	g/cc
PH of 1% Solution	12.5	
Melting Point	72.2	°C
Particular Size	96	%

2.2.4 Fine aggregate

The sand material used in this study was collected from PT. WIKA Beton precast concrete company. From sieve analysis, the sand can be classified as zone 2 (medium size sand), as shown in Fig.1.

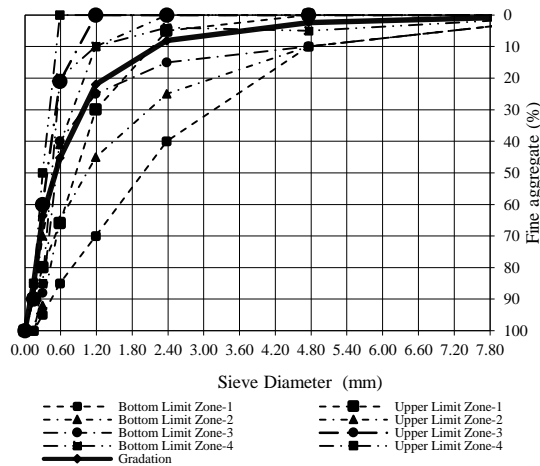


Fig.1 Fine aggregate sieve analysis

The sand properties obtained from the tests are shown in Table 6.

Table 6 Fine aggregate material properties

No	Parameter	Value	Unit
1	Organic content	1.000	
2	Sludge levels	3.080	%
3	Fine modulus	3.260	
4	Volume weight	1.586	ton/m ³
5	Moisture	1.523	%
6	Specific gravity (SSD)	2.714	ton/m ³
7	Absorption	0.402	%

2.2.5 Coarse aggregate

The coarse aggregate material used in this study was also collected from PT. WIKA Beton. The result of sieve analysis test is shown in Fig.2.

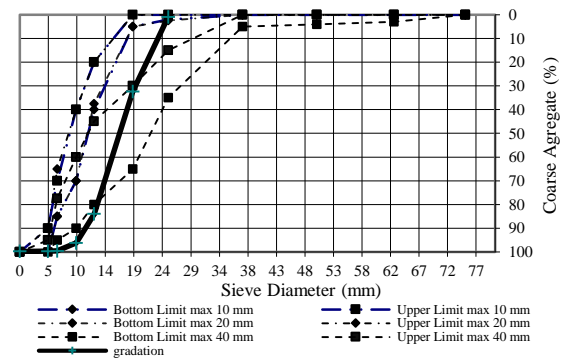


Fig.2 Coarse aggregate sieve analysis

The gravel material properties obtained from the tests is presented in Table 7.

Table 7 Gravel material properties

No	Parameter	Value	Unit
1	Organic content	-	
2	Sludge levels	1.550	%
3	Fine modulus	6.126	
4	Volume weight	1.345	ton/m ³
5	Moisture	1.112	%
6	Specific gravity (SSD)	2.674	ton/m ³
7	Absorption	1.965	%

Finally, the gravel abrasion test result gives the weight loss of 18.44 percent. The procedure of the abrasion test was in accordance with ASTM C131 [18].

Cylindrical specimens of 150 x 300 mm were made. For each mix, 5-7 specimens were prepared. These specimens were then tested at 7, 14, and 28 days. The tests performed in this study covers:

- Slump
- Permeability
- Resistivity
- Compressive strength
- pH

2.3 Mixing and Curing

GPC mixing was conducted by applying a dry method. The process started by inserting gravel into a concrete mixing machine. Water was added into it, and it was mixed for three minutes at the speed of 33-34 RPM. In this process, sucrose was inserted into the mixtures and the mixtures were mixed for one minute. When the mixtures became reasonably homogeneous, geopolymer cement was added into the mixtures and continuously mixed for two minutes. Finally, water was added. The mixtures were then mixed for another three minutes until the homogenous mixture was produced.

Fig.3 shows the appearance of homogenous mixtures of GPC after a slump test was carried out,

whereas Fig.4 shows the appearance of GPC after it was taken off from the molds.



Fig.3 Slump test of geopolymer concrete mixtures



Fig.4 Geopolymer concrete

After being kept in the molds for 24 hours, GPC specimens went for curing at room temperature. After the curing, they were inserted into a closed plastic box with 28-30°C temperature. This process can be seen in Figure 5.

Curing was conducted for 7, 14 and 28 days. After reaching the intended age, GPC was taken out from the closed box for testing.



Fig.5 Curing of GPC

2.4 Result

2.4.1 Slump test

The slump test based on ASTM C143 2015 [19] was performed to obtain GPC workability. The results are given below:

Table 8 Slump test

No.	Series	Compressive strength at 28 days (MPa)	Slump (mm)
1	Mix-1	24.50	220
2	Mix-2	37.25	217
3	Mix-3	49.70	227

Table 8 shows that mix-3 possesses 227 mm of slump. This value was only slightly higher than the slump of mix-1 and mix-2.

2.4.2 Permeability test

Concrete permeability indicates water absorption level (sorptivity) by concrete. This can be measured by examining the increase of the specimen mass-produced by water absorption as a function of time in which only one surface of the specimen is exposed to water [20]. However, in this experiment, a concrete permeability test was carried out to measure geopolymer concrete air permeability. The test was conducted using Torrent Permeability Test, as shown in Fig.6. The test result is provided in Fig.7.



Fig.6 Permeability test

The concrete classification based on Torrent Permeability Test is given in Table 9 [21].

Table 9 Classification of concrete cover quality

	Index	kT [10^{-16} m^2]
Very Bad	5	>10
Bad	4	1.0 – 10
Normal	3	0.1 – 1.0
Good	2	0.01 – 1.0
Very Good	1	<0.01

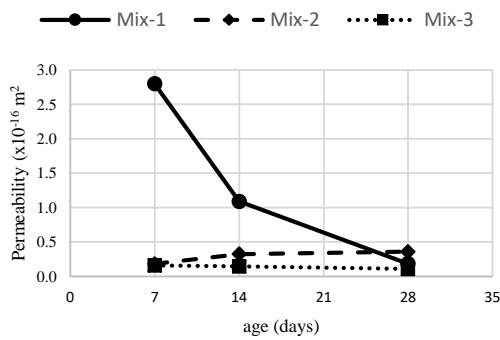


Fig.7 Permeability at 7, 14, 28 days

The concrete permeability of mix-1 at 7 days was $2.799[10^{-16} \text{ m}^2]$, at 14 days was $1.090[10^{-16} \text{ m}^2]$, and at 28 days was $0.185[10^{-16} \text{ m}^2]$, respectively. Mix-1 permeability at 28 days can be classified as Normal quality (index 3) based on value given in Table 9.

For mix-2, the concrete permeability at 7, 14 and 28 days, respectively, was $0.183[10^{-16} \text{ m}^2]$, $0.323[10^{-16} \text{ m}^2]$ and $0.360[10^{-16} \text{ m}^2]$. Similar to mix-1, concrete permeability at 28 days for mix-2 can be categorized as Normal quality (index 3).

Finally, the concrete permeability of mix-3, respectively were $0.158[10^{-16} \text{ m}^2]$ at 7 days, $0.145[10^{-16} \text{ m}^2]$ at 14 days and $0.108[10^{-16} \text{ m}^2]$ at 28 days. Based on this, mix-3 can be classified as Normal quality (index 3), similar to the two previous mixes.

2.4.3 Resistivity test

Concrete resistivity is a parameter that can be used to measure the tendency of corrosion in GPC materials. GPC resistivity value is inversely proportional to GPC permeability. The bigger the value of resistivity of GPC is, the smaller the permeability of GPC. The test of resistivity in this study was conducted by using Resipod Proceq. The equipment used can be seen in Fig.8.



Fig.8 Resistivity test

The measurement of concrete resistivity from the Wenner four-probe system was performed after the concrete had depassivated [22]. The

interpretation of test result is shown in Table 10.

Table 10 Relationship of resistivity with corrosion rate

Resistivity (kΩ-cm)	Corrosion rate
> 20	Low corrosion rate
10 – 20	Low to moderate corrosion rate
5 – 10	High corrosion rate
<5	Very high corrosion rate.

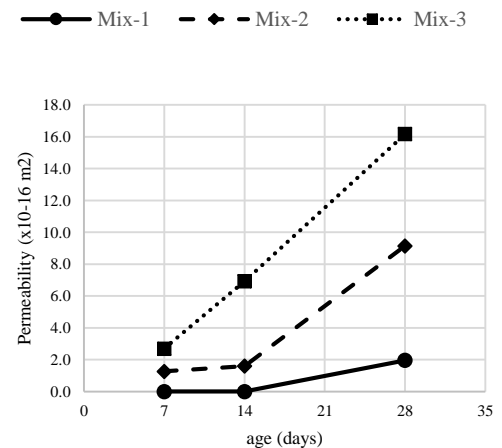


Fig.9 Resistivity for 7, 14, 28 days

The average resistivity of mix-1 at 7, 14, respectively, was smaller than 1 kΩ-cm. However, the resistivity of this mix then increased to 1.96 kΩ-cm at the age of 28 days. These test results indicated that this mix has a very low resistivity, and therefore will probably have a very high corrosion rate, based on guideline given in Table 10.

The average resistivity of mix-2 at 7, 14 and 28 days, respectively, were 1.27 kΩcm, 1.60 kΩcm and 9.15 kΩcm. Based on guideline given in Table 10, this mix can be classified as having a high corrosion rate.

Finally for mix-3, the average resistivity was 2.71 kΩcm at 7 days, then increased to 7.86 kΩcm at 14 days, and at the age of 28 days, the resistivity reached 14.42 kΩcm. Based on guideline given in Table 10, mix-3 can be categorized into low to moderate corrosion rate concrete. Therefore, mix-3 has the best performance based on resistivity test results.

2.4.4 The compressive strength tests

Concrete in a chloride environment requires a minimum of 35 MPa concrete strength and a maximum of 0.4 water-cement factors [16]. Geopolymer concrete strength testing was conducted using Universal Testing Machine (UTM) with a

capacity of 200 tons. The result of geopolymer concrete testing at the ages of 7, 14 and 28 days is shown in Figure 10.

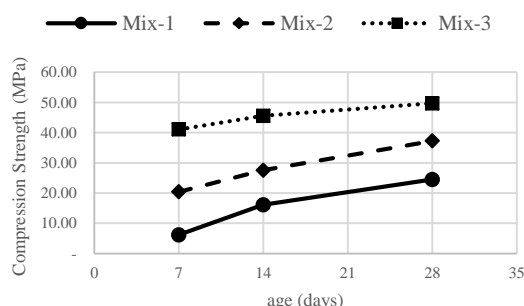


Fig.10 Compressive strength for 7,14, and 28 days

Fig.10 shows the average concrete strength of mix-1 for 7 days was 6.16 MPa, for 14 days was 16.08 MPa and for 28 days was 24.5 MPa, respectively. The figure also shows that the average concrete strength of mix-2 at the age of 7 days was 20.39 MPa, at the age of 14 days was 27.53 MPa and at the age of 28 days was 37.25 MPa, respectively. For mix-3, the average concrete compressive strength of mix-3 for 7 days was 41.07 MPa, for 14 days was 45.53 MPa and for 28 days was 49.70 MPa, respectively. Based on compressive strength, mix-3 has the highest strength at all ages.

2.4.5 pH test

pH test is a measurement of hydrogen concentration in concrete which indicates the level of acid or alkaline in concrete with a scale of 1-14. GPCC with its higher alkalinity promotes passive layer formation at the range of 12.5-13.8pH. This passive layer protects concrete rebar from corrosion [5].

The results of GPC pH for mix-1, mix-2, and mix-3 are presented in Table 11 below.

Table 11 GPC pH

Mix	PH of BG at 28 days
Mix-1 (1)	11.70
Mix-1 (2)	11.50
Mix-2 (1)	12.50
Mix-2 (2)	12.50
Mix-3 (1)	12.50
Mix-3 (2)	12.30

The pH test results in Table 11 indicates that the three GPC mixes are in the alkaline condition. pH of mix-2 and mix-3 were relatively close to one another and relatively higher than pH of mix-1.

3. CONCLUSION

This study shows that mix-3 delivers the best GPC

performance for concrete used in a chloride environment. Mix-3 has the highest compressive strength and the highest concrete resistivity at 28 days. In term of workability, the three mixes had a relatively similar slump value. Based on pH test, mix-3 has the average second highest pH compared with the other two mixes. However, based on permeability test using Torrent Permeability Test, all the three mixes had a similar concrete quality at 28 days. This research will be continued to obtain mix-3 performance when it is used in a chloride environment.

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