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THE EFFECT OF STEAM CURING METHOD TO THE COMPRESSIVE STRENGTH OF GEOPOLYMER CONCRETE WITH DIFFERENT MOLARITY

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Graphical abstract

Abstract

Cement, an important part of concrete mixture, determine the mechanical properties of concrete. The mixture of cement and water will stimulate the hydration reaction which will produce CSH gel and heat of hydration. The high level of CO_2 pollution produced in the cement manufacturing process requires alternative efforts to replace cement with other pozzolanic materials. One of the materials that can replace the role of cement in concrete mixture is fly ash. The replacement of cement 100% with fly ash will produce a geopolymer concrete, which requires an alkaline solution as a reagent from fly ash. This research was conducted with the aim to explore the effect of steam curing to the compressive strength of geopolymer concrete with different molarities. As the test object in this study was a geopolymer concrete cylinder with a standard size i.e. 150 mm × 300 mm. The molarity of NaOH solution varying from 6, 8, 10, 12, 14 to 16 M. The temperature of steam curing was carried out varied from 60°C, 80°C, up to 90°C, with duration from 1 until 4 hours. The compressive strength test conducted for 28 days old of concrete cylinder specimens. The results show an indication of an increase in the compressive strength of the concrete along with the increase in the molarity of the NaOH solution. In addition, it can also be shown that the steam curing process at a temperature of 60°C with 4 hours in duration shows optimal compressive strength results.

Keywords: Geopolymer, alkaline solution, molarity, steam curing, temperature

1.0 INTRODUCTION

Concrete structure, the most popular construction material, used for the manufacture of some infrastructure facilities such as building, bridge, road, dam etc.. Normal concrete mixture, usually contain cement, filler material (gravel and sand), and water. The combination of these materials with the right amount, as well as the correct maintenance process will produce concrete with good compressive strength. Concrete, which has more than 55 MPa of compressive strength, categorized as high strength concrete [1]. Cement, which is the basic ingredient to produce concrete, has an important contributions in terms of the compressive strength. However, cement as a basic ingredient of concrete, has a negative impact on environmental sustainability. This is an important issue because of the high level of environmental pollution contributed by the cement manufacturing process. According to Winter *et al.* [2], cement manufacturing process is contribute up to 8% of global CO_2 emissions. Chen *et al.* [3] said that for almost 30 years (from 1990 to 2019) there was an 186% increase in CO_2 emissions produced by the world cement industry. From 0.86 billion tons of CO_2 in 1990, it increased to 2.46 billion tons of CO_2 in 2019. In Indonesia itself, through the Instruction of the Ministry of Public Works, Republic of Indonesia, declare the need of sustainable construction materials, which meets security, safety, health and sustainability standards, efforts are needed to reduce greenhouse gas emissions. This is achieved by limiting the use of OPC in construction works in Indonesia [4].

The replacement of cement with other cementitious materials is a breakthrough innovation in concrete technology. A promising materials, that potential to substitute cement is called fly ash [5]. Fly ash is the most popular substitution material for cement in concrete manufacture. Geopolymer concrete utilize fly ash until 100% as a cementitious materials in concrete to replace cement. Geopolymer concrete basically consists of the same materials as normal concrete, including sand and gravel as a filler material, water and a mixture of cement and fly ash. However, the geopolymer concrete must be added with an activator solution, i.e. NaOH and Na_2SiO_3 solutions.

Davidovits from France in 1978 was the inventor of geopolymer concrete [6]. According to Davidovits, alkaline liquids work as an activator to perform reaction between Silica (Si) and Aluminum (Al), materials that can be found in fly ash. This material acts as a binder as well as cement in normal concrete mixes. The reaction in geopolymer concrete mixtures is often referred to as polymerization. Geopolymer is an inorganic polymer which is the result of the synthesis of the reaction between solid aluminosilicate material and alkali hydroxide, which is consisting of repeating 3-dimensional units of sialate monomer (-Si-O-Al-O-) [7], in the form:

$$M_n[-(SiO_2)z - AlO_2]n.\ wH_2O \quad (1)$$

The concrete treatment process is normally done by immersing the concrete in water for a certain period. Hardjito and Rangan [11] found that the mechanical properties of geopolymer concrete, i.e. compressive strength, increased after undergo a steam-curing process with temperature from 30°C to 90°C. In addition, it was also mentioned that the duration of curing time, from 4 until 96 hours, said 4 days, would be able increase the compressive strength of geopolymer concrete. However, curing more than 24 hours doesn't have a significant effect to the compressive strength.

Similar phenomenon were shown by Voraa *et al.* [12], which stated that the geopolymer concrete specimens, heated in an oven at a temperature of 75°C for 24 hours until 48 hours, has a higher compressive strength. The best results achieved, when the treatment temperature is between 60°C to 90°C with a duration of up to 24 hours. Patankar *et al.* [13] evaluated the effect of degree of heat curing at high temperatures at 40°C, 60°C, 90°C, and 120°C respectively for 24 hours. The compressive strength test was conducted after the specimens reach 3 days old. Patankar *et al*. claimed that increasing the degree of heating until 60°C, were able to accelerated early strength of concrete. Meanwhile, the results of different studies on geopolymer concrete treatment methods were carried out by Supraja *et al.* [14] which revealed that oven drying did not produce a better strength after 3 days, but the rate of increase in strength appeared to occur in the drying process with sun exposure.

Another study was conducted by Ahmed [15] analyzed the effect of oven curing process to the compressive strength of geopolymer concrete. He used different molarities of NaOH, from 8M, 12M, and 16M. Oven treated specimens produce better compressive strength, but sun treatment can also be carried out for practical purposes. Hardjasaputra *et al.* [16], reveal that the steam curing process of geopolymer concrete at a temperature of 85 – 87°C for 4 hours was able to produce a higher compressive strength of geopolymer concrete, up to 8 - 23% than the same specimens treated at normal room temperature. The same result was stated by Yewale *et al.* [17], they stated that geopolymer concrete treated by steam curing method at 80°C had better compressive strength than geopolymer concrete treated by water immersion method. This can be explained due to the slow development of the compressive strength of geopolymer concrete at low temperatures. Several similar conclusions were found by Nurruddin *et al*. [18] and Corominas *et al*. [19].

This research has an objective to evaluate the effect of steam treatment on the compressive strength of geopolymer concrete, with different molarities. The novelty of this research is the selection of 3 types of hot steam temperature conditions, i.e low temperature (60°C), medium temperature (80°C) and high temperature (90-100°C). This study combines the curing temperature range of Patankar *et al.* [13] and the curing time carried out by Hardjasaputra *et al*. [16].

Curing at low temperature is carried out with a longer duration of up to 4 hours, while curing at high temperature is carried out with a shorter duration of time, with the aim of maintaining energy use from the excessive heating of the water.

2.0 METHODOLOGY

To carry out this research, we need to cast some cylinder specimens. Standard cylindrical specimens of concrete cylinders with dimension 15 cm × 30 cm were used in this research project. The specimens are tested after each specimens reach 28 days old. After the cylindrical specimens has been casted, all specimens are treated with hot steam.

Materials

Geopolymer concrete consists of some basic materials, i.e. sand as a fine aggregate, gravel as a coarse aggregate, fly ash as a cementitious material, water, and alkaline solution (combination of NaOH solid, Na_2SiO_3 and water). The sand and gravel used in this experimental program are came from Sudamanik, Banten, Indonesia. The physical properties of aggregates are tested according to ASTM C33/C33M – 18 [17]. Table 1 summarized the physical properties of the aggregates used in this study.

Table 1 Physical properties of sand and gravel

Properties	Sand	Gravel
Unit weight	2,53	2,52
Specific Gravity	1,44	1,41
Materials Finer than 75-m	0,91	3,66
Absorption	2,61	2,78
Abrasion Test	19,89	-
Fineness Modulus	7,81	3,15

Fly ash is basically a waste arising from the coal combustion process in power plants. Fly ash is visually and chemically similar to cement, which is rich in silica (Si), iron (Fe), alumina and calcium (Ca). In 2022, Indonesia national coal production is estimated at 618 million tons. A year later, it rose again to 625 million tons. Then it will be 628 million tons in 2024 [8]. The specifications for fly ash can be found in ASTM C618 – 19 [9]. Researches on geopolymers mostly were used of class F fly ash [10]. Fly ash supplied from Suralaya, Banten, Indonesia. Table 2 shows the chemical composition of fly ash obtained from the XRF test results. Based on its chemical composition, we classified the fly ash as class F, based on ASTM C618 [16].

Table 2 Result of X-Ray Flourecence (XRF) of Fly Ash Material

Compound Name	(%)	Compound Name	(%)
SiO_2	37,385	BaO	0,349
Fe_2O_3	25,223	SrO	0,275
CaO	14,084	Na_2O	0,173
Al_2SiO_3	12,543	ZrO_2	0,144
K_2O	3,474	ZnO	0,121
TiO_2	2,757	Cl	0,048
P_2O_5	1,638	Rb_2O	0,045
MgO	0,855	Br	0,016
SO_3	0,853	Y_2O_3	0,015

To activate fly ash in the mixture of geopolymer, we need an alkaline solution, which is a combination of SiO_2/Na_2O and NaOH. According to Hardjito and Rangan (2005), the higher the molarity of the NaOH solution will be able to produce a higher compressive strength, besides that the higher the mass ratio of Na_2SiO_3 to NaOH, it will also produce higher compressive strength [11].

When mixing a geopolymer concrete mixing, alkaline solution has a function as a trigger for the reaction between Silica and Alumina contained in fly ash. This reaction known as polymerization reaction. A commonly used alkaline solution is a solution of NaOH and Na_2SiO_3. The higher Na_2SiO_3 content provide a better strength of geopolymer concrete to be higher than the higher NaOH content. In addition, variations in the ratio of Na_2SiO_3 with NaOH can also affected the workability of the concrete mixture. Proportion of Na_2SiO_3 to NaOH in the range of 1.5-2.0, can produce higher strength of geopolymer concrete [10]. Figure 1(a) shows NaOH in solid form before being diluted with water to obtain the desired molarity. Figure 1(b) shows an alkaline solution, which was obtained from mixing a solution of NaOH and Na_2SiO_3.

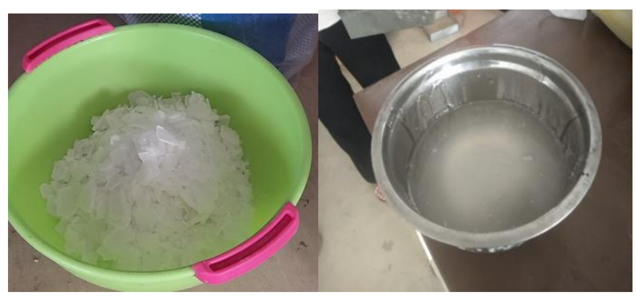

Figure 1 (a) Sodium Hydroxide, (b) Sodium Silicate Liquid

Mix Design

The targeted of concrete compressive strength is f'_c= 20 MPa. The geopolymer concrete mixture in this study was designed according to Indonesian Standard SNI 7656:2012 [18]. Six different molarity of NaOH were used in the mixed design, namely 6M, 8M, 10M, 12M, 14M and 16M NaOH. The material proportions required per m^3 including the weight of Fly Ash (FA), Na_2SiO_3 (SS), NaOH (SH), water (W), coarse aggregate (CA), fine aggregate/sand (S) are shown in the Table 3. The table also provide the information of the ratio for alkaline solution to fly ash (AS/FA) and for Na_2SiO_3 to NaOH (SS/SH).

Table 3 Composition of Concrete Mixture

Mix Code	Quantity (kg/m³)						Molarity	AS/FA ratio	SS/SH ratio
	FA	SS	SH	W	CA	S			
GC6	315.14	118.18	7.88	31.51	1201.59	705.7	6	0.400	15.00
GC8	315.14	118.18	10.05	29.35	1201.59	705.7	8	0.407	11.76
GC10	315.14	118.18	12.05	27.34	1201.59	705.7	10	0.413	9.81
GC12	315.14	118.18	13.94	25.45	1201.59	705.7	12	0.419	8.48
GC14	315.14	118.18	15.76	23.64	1201.59	705.7	14	0.425	7.50

Specimens

The specimens were made based on the standard ASTM C39/C39M-09a [20]. The specimens used in this research is a cylindrical concrete with dimension 150 mm in diameter and 300 mm in height. The 28 days old specimens were tested for its compressive strength using the Universal Testing Machine (UTM). With 6 different molarity of NaOH, i.e. 6, 8, 10, 12, 14 and 16M, the number of specimens made was 60 cylinders to be tested with 3 different curing temperature conditions. Cylinders that have reached the age of 28 days will undergo a compressive strength test, with a capping of sulfur mortar caps. This sulfur capping is needed so that the load can be distributed uniformly to the surface of the cylindrical specimens.

Curing Method

After the specimen is molded and aged for 1 day, the specimen is removed from the mold for further treatment by the steam curing method. The steam curing process is carried out using an acrylic box that is connected to a water heater that can blow hot steam into the acrylic box. At the top of the box there is a thermometer to measure the temperature. The specimens were given hot evaporation with temperature variations of 60°C (4 hours), 80°C (2 hours), and 90°C (1 hour). The selection of these three types of curing process aims to represent curing at low temperatures (60°C), medium temperatures (80oC) to high temperatures (90°C). Treatment at low temperatures is carried out with a longer duration of up to 4 hours, while the higher the curing temperature, the curing duration is also reduced to 1 hour at 90°C. Figure 2(a) shows the acrylic box and the water boiler used for steam curing process.

After the steam curing process is complete, the test object is removed from the acrylic box and cooling down at ambient temperature for 24 hours, then proceed with conventional treatment by immersing it in a water bath until it reaches the specified test age. Figure 2(b) shows the process of treating the test object by immersing it in a water bath.

Figure 2 a. Steam Curing Process, b. Water Curing Process

3.0 RESULTS AND DISCUSSION

Slump Result

The slump measurements obtained from the 6 types of concrete mixtures are shown in Figure 3. From the figure it appears that the 6M mixture shows the highest slump value. The slump value tends to decrease, in line with the increase in the molarity of the NaOH solution. The largest decrease was in geopolymer concrete with a molarity of 16M, which experienced a decrease in slump value of 10.72%. This phenomenon is in line with the results presented by Hardjasaputra [16], Memon *et al.* [21], and Fang *et al.* [22]. The decrease in slump value accompanied by an increase in NaOH molarity is quite understandable because the higher molarity of NaOH, the higher the viscosity of the solution and the cohesiveness of the concrete [21], the water volume decreases, resulting lower workability [16].

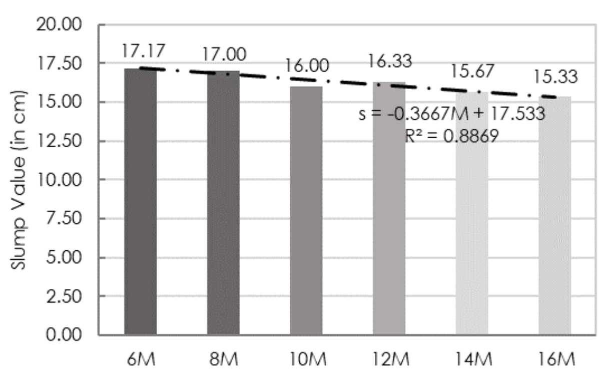

Figure 3 Slump test results

Relationship between the ratio AS/FA to the slump value, is shown in Figure 4. From the graph it is very clear that the slump value of the concrete mixture tends to continue to decrease if the ratio between the alkali solution to fly ash increases. This also supports the conclusions drawn by several other studies regarding the slump value of geopolymer concrete [22-24].

In contrast to the relationship between the slump value to the ratio of alkaline solutions and fly ash, the opposite relationship is shown between the slump value to the ratio of Na_2SiO_3 and NaOH (SS/SH). Figure 5 shows the relationship between the slump value to the ratio of Na_2SiO_3 and NaOH (SS/SH). Ratio of SS/SH influenced the slump value, the higher the SS/SH ratio, will produce higher slump value.

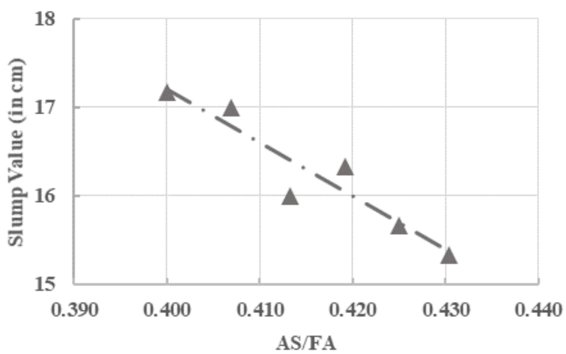

Figure 4 Relationship Between Slump Value & AS/FA

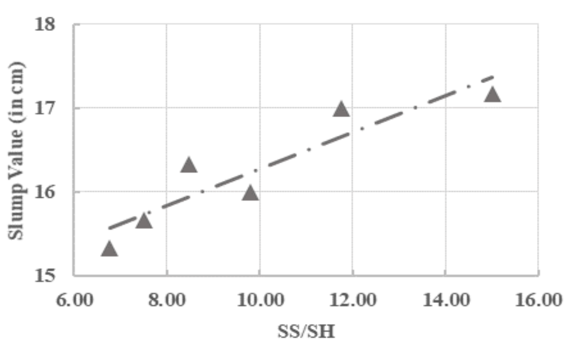

Figure 5 Relationship Between Slump Value and SS/SH

Density of Concrete

Figure 6 shows the density of the geopolymer concrete. Geopolymer concrete with a molarity of 6M alkaline solution has a specific gravity of 2,083.25 kg/m^3, while in a 16M solution, the specific gravity reaches 2,248.28 kg/m^3, an increase of about 7.82%. The result shows that geopolymer concrete has density between 2,000 to 2,250 kg/m^3. As the molarity of alkaline solution was increased the density has also a tendency to increase. This results inline with the work presented by Wazien *et al.* [25].

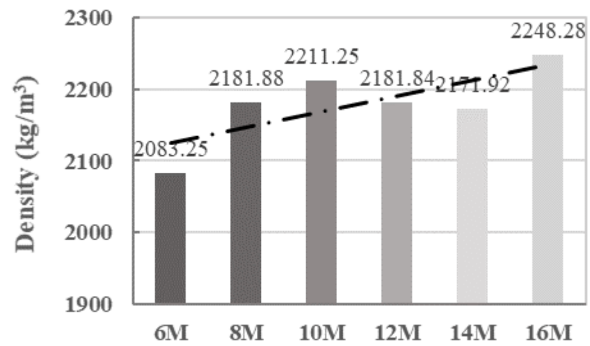

Figure 6 Density of Geopolymer Concrete

Compressive Strength

The concrete compressive strength test was carried out in accordance with the ASTM C39/C39M – 21 standard [20]. The value of the compressive strength of concrete is:

$$f'_c = P/A \quad (1)$$

With f'_c is the compressive strength of concrete (MPa), *P* is the maximum test load in Newton and A is the cross-sectional area of the concrete cylinder in mm^2. Figure 7 shows the relationship between the molarity of sodium hydroxide solution (NaOH) to the compressive strength of the concrete, for several different steam curing temperatures. The targeted concrete compressive strength is 20 MPa. From Figure 7, the 6M molarity of NaOH has not reached the targeted compressive strength. Meanwhile for the 8M molarity value, with 60°C steam curing for 4 hours is able to reach design compressive strength. For molarity values above 10M, the design compressive strength value can be achieved by all test specimens. The greatest compressive strength value was achieved by 14M mixture, i.e. 23.56 MPa which was treated with hot steam curing of 60°C for 4 hours. This result inline with the work done by Patankar *et al.* [26], who recommend the suitable molarity of sodium hydroxide solution is 13-molar, and cured 8 hours at 90°C in an oven.

Refer to the steam curing temperature, compressive strength of concrete cured with a temperature of 60°C for 4 hours showed relatively better results than temperatures of 80°C for 2 hours and 90°C for 1 hours. It looks that the duration of curing determines the compressive strength of the concrete specimens. It can be understood that in contrast to normal concrete, geopolymer concrete has a geopolymerization reaction between fly ash and activator solution. According to Hardjasaputra *et al.* [16], this reaction is slow, so it affects the compressive strength of young concrete. The steam curing process is able to provide acceleration for the ongoing geopolymerization process in geopolymer concrete. The duration of steam curing with a longer period of time is able to have a positive impact on the continuity of the geopolymerization reaction in concrete. Steam curing method is an effective way to produce a good quality of geopolymer concrete. The research done by Hardjasaputra *et al.* [16] shows that heating with steam for 4 hours can be achieved increasing in compressive strength up to 20%, compared to room temperature curing.

The duration of steam curing for 1 hour at a high temperature is less able to produce optimal compressive strength compared to steam curing at a temperature of 60°C for 4 hours. Some studies use a longer time duration of up to 24 hours [17], but this requires greater heat energy which is not in accordance with the original purpose of using geopolymer concrete, namely reducing CO_2 emissions into the air.

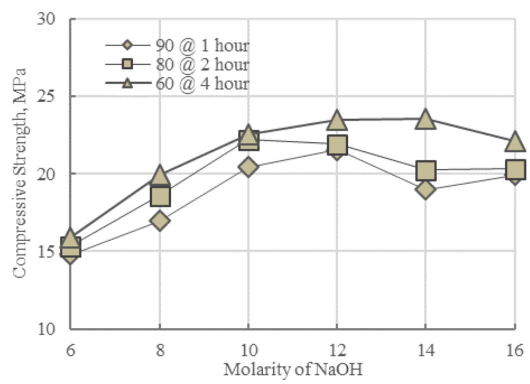

Figure 7 Relationship Between Molarity of NaOH and Compressive Strength

Figure 8 shows the relationship between the ratio of Na_2SiO_3 and NaOH solid, to the compressive strength of concrete. Based on the figure, the optimal ratio between the weight of Na_2SiO_3 to NaOH solid is in the range of 7.5 to 8.5, with steam curing temperature of 60°C with duration of 4 hours. SS/SH ratio valuesabove 12 tend to lower the compressive strength of concrete.

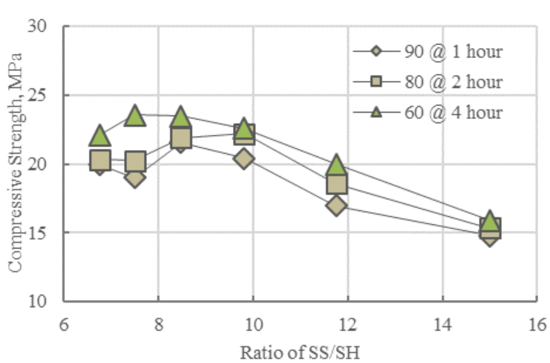

Figure 8 Ratio of SS/SH and Compressive Strength

Figure 9 shows the relationship between the ratio of alkaline activator solution (AL) and fly ash (FA) to the compressive strength of concrete. The geopolymer concrete mixture with an AL/FA ratio of less than 0.41 tends not to reach the targeted compressive strength value. Optimum ratio was obtained for a mixture with a ratio of AL/FA between 0.42 to 0.425, and the steam curing process was carried out at a temperature of 60°C for 4 hours.

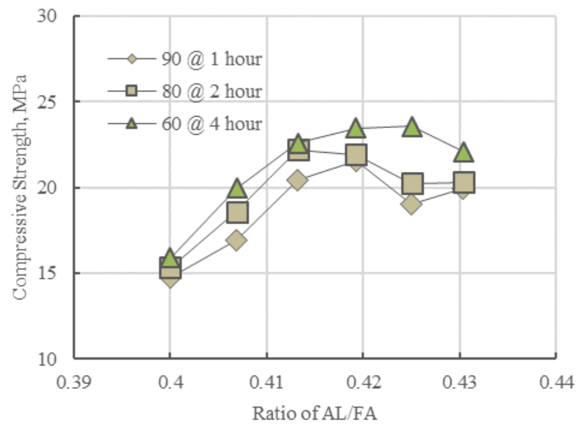

Figure 9 Ratio of AL/FA and Compressive Strength

4.0 CONCLUSION

Geopolymer concrete with NaOH solution molarity of 12M to 14M, which underwent steam treatment at a temperature of 60°C for 4 hours tended to show better compressive strength values, an increase of up to 17.8% from the planned compressive strength value. The optimum ratio between the weight of Na_2SiO_3 to NaOH solid is in the range of 7.5 to 8.5, while the ratio of AL/FA is between 0.42 to 0.425. The ratio of SS/SH above 12 indicate a lower compressive strength of concrete. The optimum steam curing temperature is 60°C with duration of 4 hours. The slump value of geopolymer concrete has a tendency to decrease, when the molarity of the NaOH increases. The density of geopolymer concrete is in the range of 2,000 to 2,250 kg/m^3, the density of geopolymer concrete tends to increase as well as the molarity of NaOH increase.

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