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Effect of Curing Time and Variations in Na_2SiO_3: NaOH Ratio on the Mechanical Properties of Fly Ash-based Geopolymer Artificial Aggregates using the Crushing Method

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ABSTRACT

Burning coal to produce electrical energy in PLTU produces waste in the form of fly ash. This waste must be managed properly so that it does not cause detrimental environmental effects. Fly ash has potential as a building material because it contains silica, aluminum, and other oxides that can react with alkaline solutions to form geopolymer compounds. Geopolymer has good mechanical properties and is resistant to acidic and alkaline environments. The development of the crushing method for aggregate materials, artificial geopolymers, and fly ash is an interesting work direction because it can utilize fly ash waste and reduce the dependence on aggregates. Moreover, this material can be used to help reduce the effects of development construction on the environment. The crushing method is utilized to obtain geopolymer artificial aggregate with the desired size. Aggregates can be destroyed to become small particles by using destruction techniques and mechanical crushing tools, such as hammer mills, jaw crushers, and ball mills. The aggregate impact value (AIV) percentage reaches 13.20% at a Na_2SiO_3/NaOH ratio of 2, but it decreases to 12.64% at a Na_2SiO_3/NaOH ratio of 3.5. The AIV percentage reaches 12.82% without the addition of sand, but it decreases to 12.64% when the added amount of sand is 20%. The AIV percentage increases to 12.76% when the added sand is 40%. Moreover, the AIV percentage reaches 12.64% after curing for 24 hours, decreases to 12.46% after 48 hours of curing, and increases to 12.94% after 72 hours of curing.

1. Introduction

In industry construction, aggregates are used in the making of concrete, asphalt, and other construction materials. Aggregates, which are acquired from natural sources, such as crushed stone, gravel, and sand, have become an important source of power in infrastructure development and project construction. However, the increasing global demand for fine aggregates has reduced the availability and quality of aggregates.

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In recent decades, the demand for development infrastructure and project construction has increased considerably in various countries. Population growth, urbanization, and economic development have resulted in the need for aggregates to fulfill construction material requirements. This increasing demand puts pressure on the availability of aggregates.

The use of artificial aggregates has its merits. First, artificial aggregates can reduce the dependence on natural aggregate and the effects of mining on the environment. Second, the use of cycled materials as the material standard for artificial aggregates can reduce waste (including industrial waste) generation. Third, artificial aggregates have characteristics that can be controlled by physics and mechanics, which increases the quality and consistency of construction materials [1,2].

The crushing method is used to obtain geopolymer artificial aggregates of the desired size. In this method, fly ash can be crushed into small particles by using various crushing techniques, such as mechanical crushers or crushing tools, including hammer mills, jaw crushers, and ball mills.

According to Rafiza, the demand for aggregates is increasing and has reduced the supply or source of natural aggregates in the environment [3]. Several developed countries, such as America, England, and Poland, have succeeded in establishing effective and efficient aggregate manufacturing. Alternatively, researchers have developed lightweight geopolymer aggregates. Geopolymers are made from natural ingredients containing large amounts of silicon and aluminum [4]. Using waste as a basic material to replace non-renewable natural materials is increasingly gaining significant momentum in waste management practices [5]. One of the wastes that can be used to make artificial coarse aggregate is fly ash. Fly ash can be used as a material compiler artificial aggregate geopolymer. The combination of fly ash and sand can enhance the characteristics of aggregates. Fly ash is formed from burning coal and contains silica and lime. Fly ash, one of the disposable wastes from combustion, can be mixed and reacted with an activator to form aggregates. The aggregate impact value (AIV) reveals the strength of aggregates against collision and is a basic testing process in the manufacturing of aggregates because it determines the destruction extent of aggregates after impact. According to BS 812 Part 112: 1990, the limit value of aggregate destruction (AIV) is 30% [6].

The durability of geopolymer aggregates can be influenced by the ratio of fly ash to alkaline activators. Fly ash with sodium (Na) and aluminum (Al) elements is known for its quick responsiveness [7-9]. Curing temperature is another crucial factor that can enhance geopolymer strength. An increase in curing temperature increases compressive strength [10].

2. Methodology

The experimental method was implemented with a fly ash-to-alkaline activator mass ratio of 2, NaOH molarity of 15 mol, and sand addition of 0%, 20%, and 40%. The mass ratio of Na_2SiO_3 to NaOH was varied to 2.5, 3, and 3.5. This study included testing of the filter, heavy type, specific gravity, capability to absorb water, and capability to withstand degradation caused by abrasion and impact. This testing was done sequentially from the beginning to the end.

2.1 Preparation

The first stage was the preparation of materials and tools to be used in mixing the geopolymer artificial aggregate. The prepared materials were sand, fly ash, and an alkaline activator solution consisting of NaOH, Na_2SiO_3, and distilled water. The prepared equipment included mixers, beakers, measuring glasses, and an oven.

For geopolymer artificial aggregate, the alkaline activator solution was the main material used for making the polymer bond with fly ash. The Na_2SiO_3-shaped gel and NaOH solution formed a solid chunk. NaOH in congested form needed to be dissolved in advance with distilled water; after that, it was mixed with Na_2SiO_3. The following shows the stages of preparing the alkaline activator solution (Figure 1-3):

i. The necessary NaOH was prepared. The required molarity was 1 mol of NaOH (40 g). We used molarity values of 15 M, which are equivalent to 600 gr.
ii. NaOH was dissolved using distilled water until the solution reached 1,000 mL in a beaker and stirred with a stick stirrer until the NaOH was completely dissolved. Afterward, the solution reacted and became hot. Then, the solution was allowed to stand for 24 h until the solution reached room temperature.
iii. Na_2SiO_3 and NaOH were mixed.

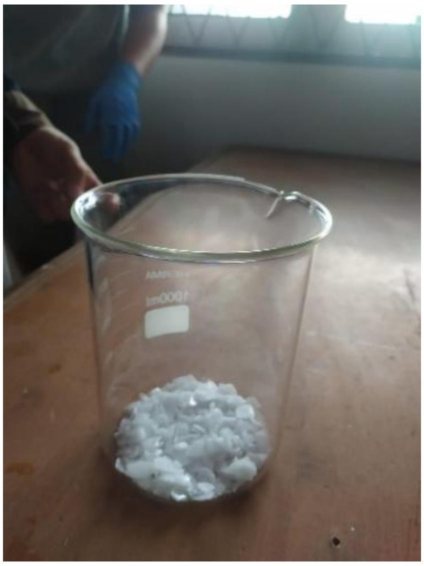

Fig. 1. NaOH in a measuring cup

Fig. 2. Mixing NaOH with Aquadest

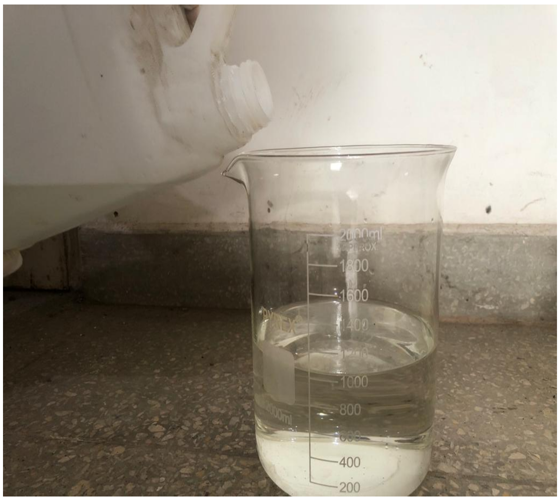

Fig. 3. Mixing the NaOH solution with Na_2SiO_3

2.2 Testing of Sand and Fly Ash

The stage second was for testing sand and fly ash. This process was implemented to determine the characteristics of sand and the composition of fly ash and sand. Sand testing was conducted in accordance with the following ASTM standards:

i. Testing and analysis of the sieve by using ASTM C136 [11],
ii. Testing rate of organics in sand by using ASTM C40 [12],
iii. Testing rate of mud on sand by using ASTM C142 [13],
iv. Testing the volume weight of sand by using ASTM C29/C29M [14],
v. Testing of the specific gravity and water absorption by using ASTM C128 [15].

The fly ash used in this study was obtained from PT. Pupuk Sriwidjaja Palembang. This material was tested using X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF), and Scanning Electron Microscope (SEM) to determine its characteristics and class.

2.3 Planning

The third stage was the planning and preparation of the mixture of geopolymer artificial aggregate. Planning and preparation were performed by collecting source data from a previous study. The planning of the mix design was based on trials conducted in the laboratory.

2.4 Mixing

The fourth stage was the mixing of the materials that made up the artificial geopolymer aggregate. Samples were prepared using different mass ratios of fly ash to the alkaline activator (i.e., ratio of Na_2Sio_3 and NaOH), namely, 2.5, 3, and 3.5. The molarity was 15 mol, and the added sand was 0%, 20%, and 40%. Each variation had three samples, so 45 samples were obtained.

Material mixing was performed at the Structural Construction Materials Laboratory, Sriwijaya University. Initially, sand and fly ash were mixed in accordance with the mix design at room temperature. Then, the alkaline activator mixture was prepared, and the dry and liquid ingredients were mixed. After all the ingredients were mixed perfectly, the mortar was ready to be shaped manually. The test object was placed in the oven at 80 °C for 24, 48, and 72 h. Afterward, the test object was removed from the oven.

After the test object was removed from the oven, it was crushed using a crusher. Then, the artificial geopolymer aggregate hardened and was ready to be tested in accordance with ASTM.

2.5 Testing of the Aggregate

The final stage was the testing of the aggregate test object, namely, the artificial geopolymer. Several tests were conducted for this stage, as follows:

i. Testing and analysis of the sieve by using ASTM C136 [11],
ii. Testing of the moisture content by using ASTM C566 [16],
iii. Specific gravity and water absorption testing by using ASTM C127 [17],
iv. Testing of the volume weight by using ASTM C29/C29M [14],
v. AIV testing by using BS 812-112: 1990 [18].

3. Analysis and Discussion

Table 1 shows the results of the testing of the aggregate properties. The test results indicate that fine aggregate can be used as a mixture for geopolymer-made aggregates.

Table 1
Fine aggregate test results

No.	Characteristics	Results	Specification
1	Fineness modulus	2.34	ASTM C136
2	Gradation area	Zone 3	ASTM C136
3	SSD specific gravity	2.382	ASTM C128
4	Oven dry specific gravity	2.327	ASTM C128
5	Apparent specific gravity	2.462	ASTM C128
6	Water absorption (%)	2.355	ASTM C128
7	Bulk density solid state (kg/l)	1.181	ASTM C29/C29M
9	Bulk density fine condition (kg/l)	1.331	ASTM C29/C29M
10	Organic impurities	No.4	ASTM C40/40M
11	Clay lumps (%)	2.0	ASTM C142/142M

3.2 Fly Ash Testing

3.2.1 X-ray diffraction testing

On the basis of the results of the X-ray diffraction test (Figure 4), the structure of the fly ash used was classified as amorphous because only a few crystal peaks were formed. The condition of the amorphous structure and the presence of a few crystal peaks in the fly ash indicated that the fly ash had reactive properties and dissolved easily.

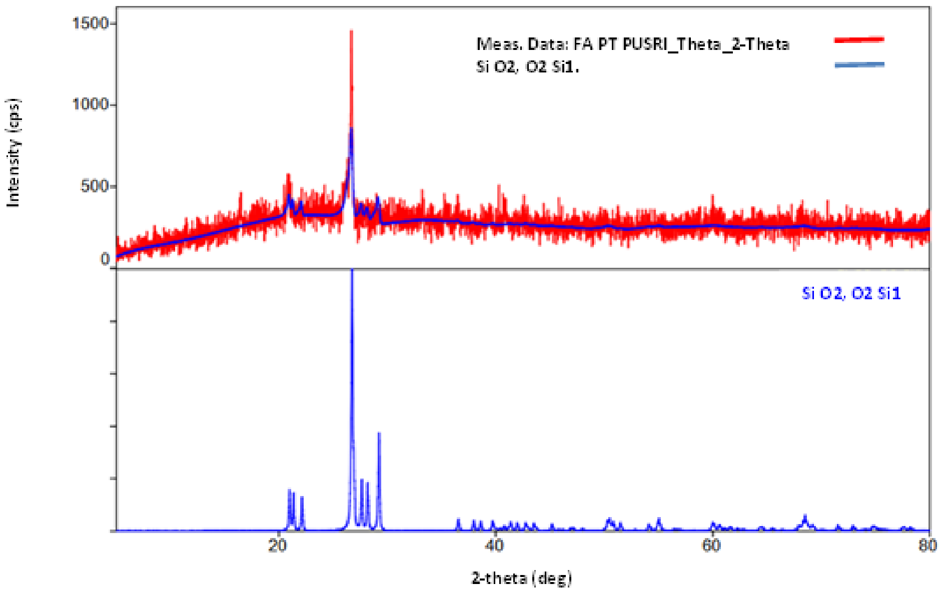

Fig. 4. XRD test results [19]

3.2.2 X-ray fluorescence testing

The X-ray fluorescence test results showed that the fly ash used contained CaO < 10%, indicating that the fly ash belonged to category F according to the ASTM 618 standard (Table 2).

Table 2
XRF test results

Composition Chemistry	Amount Content (%)
MgO	0.43
Al_2O_3	6.76
SiO_2	15.0
P_2O_5	0.121
SO_3	0.951
Cl	0.0344
K_2O	0.494
CaO	2.29
TiO_2	0.528
MnO	0.0678
Fe_2O_3	4.01
ZnO	0.0072
As_2O_3	0.0077
SrO	0.0643
Y_2O_3	0.005
ZrO_2	0.0361
Ag_2O	0.0745
Balance	69.1

3.2.3 Scanning electron microscopy

Scanning electron microscopy revealed that the dominant fly ash grains were round in shape and had an average particle size of 2 µm (Figure 5). Extant research shows that the particle size of fly ash influences the geopolymer mortar. The finer fly ash is, the higher the workability and compressive strength of the geopolymer mortar are [20].

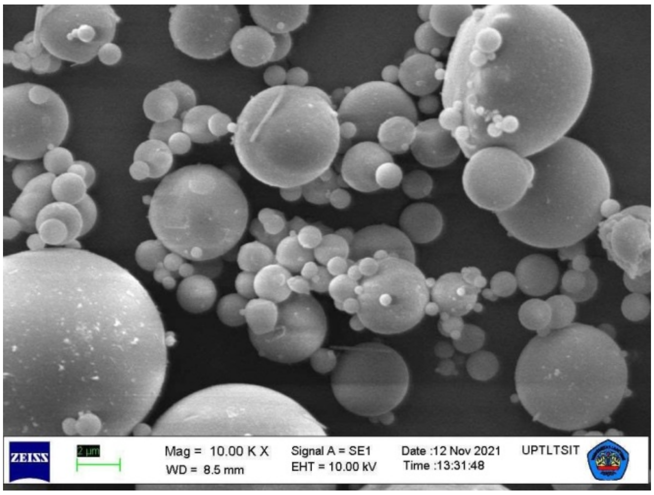

Fig. 5. SEM test results [19]

3.3 Natural Coarse Aggregate Testing

The natural coarse aggregate material used in this experiment was crushed rock obtained from PT. Sinar Musi. The size of the coarse aggregate ranged from 5 mm to 20 mm. The purpose of testing the coarse aggregate was to compare its results with those of tests on artificial aggregates (Table 3-6, Figure 6). A series of tests, including testing for moisture content, sieving, specific gravity, water absorption, unit weight, and AIV, was conducted on the coarse aggregate.

Table 3
Natural coarse aggregate test results

No.	Characteristics	Results	Specification
1	Water content (%)	1.668	ASTM C566
2	Gradation area	Size number 67	ASTM C136
3	Fineness modulus	2.272	ASTM C33
4	SSD specific gravity	2.539	ASTM C127
5	Water absorption (%)	3.94	ASTM C127
6	AIV (%)	1.46	BS 812-112: 1990

3.4 Testing of the Artificial Aggregate Crushing Method

Table 4
Variation in the Ratio of Na_2SiO_3/NaOH

Data	Information
Sand	Use sand 20% of heavy fly ash
Molarity NaOH	15 mol
FA/AA	2
Variation 2 .5	Ratio of Na_2SiO_3 to NaOH = 2,5
Variation 3	Ratio of Na_2SiO_3 to NaOH = 3
Variation 3 .5	Ratio of $Na_2SiO_3$to NaOH = 3,5
Curing	Oven with a temperature of 80℃ for 24 hours
Testing time	Testing was done 7 days after test specimens were made and oven-cured for 24 hours
Treatment testing	Every testing variation was treated the same, that is, curing was done before testing and testing was done in accordance with the standard ones used
Testing standards	Testing of the physical and mechanical properties of the aggregate used ASTM and SNI standards

Table 5
Variation in sand addition

Data	Information
Na_2SiO_3 /NaOH	3.5
Molarity NaOH	15 mol
FA/AA	2
0% Variation	Use sand 0% of heavy fly ash
20% Variation	Use sand 20% of heavy fly ash
40% Variation	Use sand 40% of heavy fly ash
Curing	Oven with a temperature of 80℃ for 24 hours
Testing time	Testing was done 7 days after the test specimens were made and oven-cured for 24 hours
Treatment testing	Every testing variation was treated the same, that is, curing must be done before testing and testing must be done in accordance with the standard onesused
Testing standards	Testing of the physical and mechanical properties of the aggregate used ASTM and SNI standards

Table 6
Variations in curing time

Data	Information
Sand	Use sand 20% of heavy fly ash
Molarity NaOH	15 mol
FA/AA	2
Na_2SiO_3 /NaOH	3.5
Curing 24 hours	Oven curing for 24 hours at 80℃
Curing 48 hours	Oven curing for 48 hours with a temperature of 80℃
Curing 72 hours	Oven curing for 72 hours at 80℃
Treatment testing	Every testing variation was treated the same, that is, curing was done before testing and testing was done in accordance with the standard ones used
Testing standards	Testing of physical and mechanical properties of the aggregate used ASTM and SNI standards

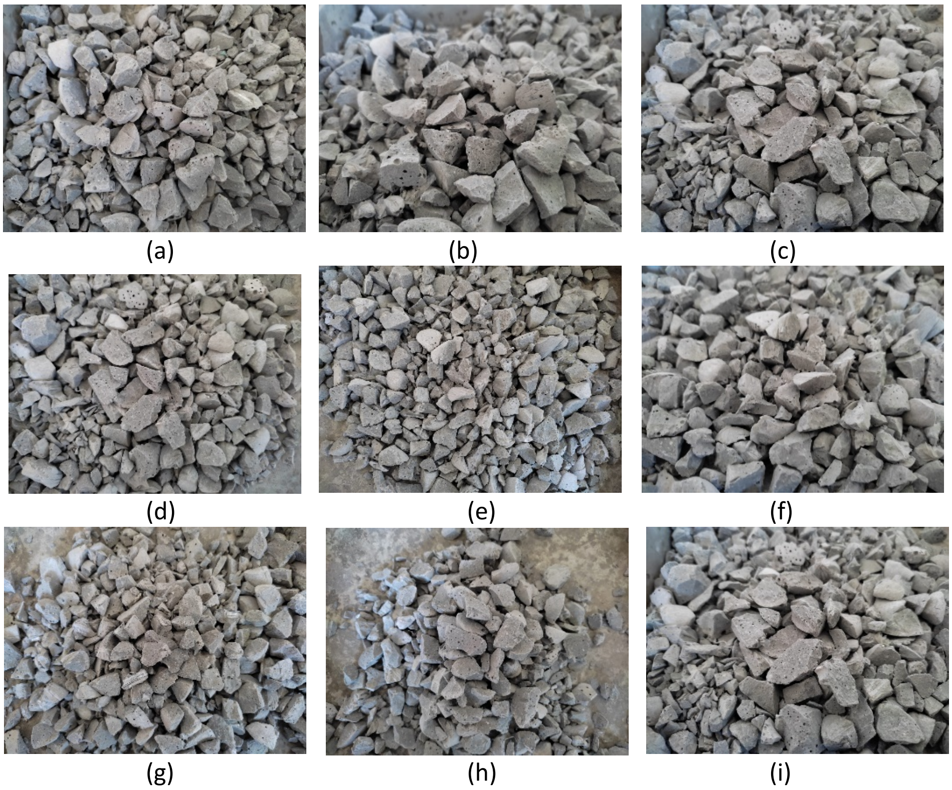

Fig. 6. Crushing aggregate variation: (a) the ratio of Na_2SiO_3/NaOH is 2.5, (b) the ratio of Na_2SiO_3/NaOH is 3, (c) the ratio of Na_2SiO_3/NaOH is 3.5, (d) 0% sand, (e) 20% sand, (f) 40% sand, (g) curing for 24 hours, (h) curing for 48 hours, and (i) curing for 72 hours

3.4.1 Testing of moisture content

Testing of moisture content was performed to determine the existing water content in the crushed aggregate (Table 7). This test was done based on ASTM C566 [16].

Table 7
Testing of moisture content

Name	Variation	Moisture Content (%)
Variation 1	Ratio of Na_2SiO_3/NaOH = 2,5	12,309
Variation 2	Ratio of Na_2SiO_3/ NaOH = 3	12,613
Variation 3	Ratio of Na_2SiO_3/ NaOH = 3,5	12,511
Variation 4	0% sand	12,208
Variation 5	20% sand	12,511
Variation 6	40% sand	13,327
Variation 7	Curing for 24 hours	12,511
Variation 8	Curing for 48 hours	12,816
Variation 9	Curing for 72 hours	13,225

3.4.2 Sieve analysis and testing

Sieve analysis was conducted to determine the fineness modulus and graph gradation of the crushed artificial aggregates (Table 8). The results of the sieve analysis indicated that met the condition range of fineness modulus from 2.96 to 3.55 in ASTM C33 [21]. The lowest fineness modulus was found in variation Ratio of Na_2SiO_3/NaOH = 3; the highest was found in variation 0% sand.

Table 8
Sieve analysis and testing

Name	Variation	Fineness modulus
Variation 1	Variation Ratio of Na_2SiO_3/NaOH = 2,5	3.38
Variation 2	Variation Ratio of Na_2SiO_3/NaOH = 3	2.96
Variation 3	Variation Ratio of Na_2SiO_3/NaOH = 3,5	3.49
Variation 4	Variation 0% sand	3.55
Variation 5	Variation 20% sand	3.49
Variation 6	Variation 40% sand	3.44
Variation 7	24-hour curing variation	3.49
Variation 8	48-hour curing variation	3.33
Variation 9	72-hour curing variation	3.20

3.4.3 Testing of specific gravity and water absorption

The testing of specific gravity and water absorption was conducted based on the ASTM C127 standard (Table 9).

Table 9
Specific gravity and water absorption testing

Variation	Bulk Specific Gravity (SSD)	Absorption Value (%)
Na_2SiO_3/NaOH 2,5	2,042	17,835
Na_2SiO_3/NaOH 3	2,131	18,613
Na_2SiO_3/NaOH 3,5	2,042	17,835
0% sand	2,104	17,127
20% sand	2,042	17,835
40% sand	2,055	17,288
Curing for 24 hours	2,042	17,835
Curing for 48 hours	2,064	17,963
Curing for 72 hours	2,096	17,883

3.4.4 AIV testing

AIV determines the strength of an aggregate. A low AIV percentage increases the toughness of the aggregate (Table 10).

Table 10
Aggregate Impacte Value

Variation	Aggregate Impacte Value (%)
Na_2SiO_3/NaOH 2,5	13,20
Na_2SiO_3/NaOH 3	13,04
Na_2SiO_3/NaOH 3,5	12,64
0% sand	12,82
20% sand	12,64
40% sand	12,76
Curing for 24 hours	12,64
Curing for 48 hours	12,46
Curing for 72 hours	12,94

The results of the 48-hour variation indicated that the lowest AIV percentage was 12.46%. The variation of Na_2SiO_3/NaOH 2.5 produced the highest AIV percentage of 13.20%. The lower the AIV percentage of artificial aggregates is, the higher the strength of the aggregates is. According to the BS 812-112:1990 standard, an AIV value below 30% indicates good quality. However, when the AIV value exceeds 30%, extra care must be exerted during handling [18].

4. Conclusions

The following conclusions were obtained from the testing conducted on crushed aggregates.

i. The AIV test on the geopolymer artificial aggregate by using the crushing method showed that the ratio of Na_2SiO_3/NaOH affected the mechanical characteristic. The AIV percentage reached 13.20% at a Na_2SiO_3/NaOH ratio of 2, but it declined to 12.64% at a Na_2SiO_3/NaOH ratio of 3.5. Therefore, the effective ratio for the geopolymer artificial aggregate crushing method is Na_2SiO_3/NaOH = 3.5.
ii. With the geopolymer artificial aggregate crushing method, the amount of added sand that influenced the mechanical characteristics was tested through AIV. The AIV percentage reached 12.82% without the addition of sand, but it declined to 12.64% when the added sand was 20%. However, the AIV value increased to 12.64% when the added sand was 20% and reached 12.76% when the added sand reached 40%. Therefore, the amount of added sand that is effective for the mechanical characteristics of geopolymer artificial aggregate is 20%. This result proves that a certain proportion of sand must be added to produce a low AIV in geopolymer aggregates.
iii. The effects of curing time on the mechanical characteristics of geopolymer artificial aggregate subjected to the crushing method were determined through AIV. The AIV percentage reached 12.64% at 24 h of curing time but declined to 12.46% when the curing time was extended to 48 h. However, AIV was enhanced to 12.46% when the curing time was 48 h, and it reached 12.94% when the curing time was extended to 72 h. We conclude that the curing process using an oven takes a long time to influence the geopolymer aggregate's AIV. A long curing process affects the percentage increment of AIV. In this study, the 72-hour curing variation experienced an enhancement amounting

to 12.94%, which was previously obtained in the 48-hour curing variation amounting to 12.46%.

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