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The Effect of the Ratio of Fly Ash to Alkaline Activator and the Molarity of NaOH on the Mechanical Properties of Fly Ash-Based Aggregates

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ABSTRACT

The global demand for aggregates has escalated, leading to a decline in the availability of quality natural aggregates. In response, the cold bond pelletization (CBP) process emerged in the early 2000s as a novel approach to artificial aggregate production, utilizing dry powdered fly ash. This method involves agglomerating fly ash particles at room temperature in an inclined rotating pan to form pellets. However, the abstract lacks clarity in elucidating the significance of this approach. Our study investigates the mechanical properties of aggregates produced through the CBP method via experimental laboratory methods, focusing on variations in the ratio of fly ash to alkaline activator (FA/AA) and the molarity of Sodium Hydroxide (NaOH). Notably, we found that an effective molarity of 15 resulted in the lowest Aggregate Impact Value (AIV) percentage, indicating improved mechanical properties. Furthermore, we observed fluctuations in AIV values across different FA/AA ratios and NaOH molarities, suggesting nuanced effects on aggregate quality. Our findings underscore the importance of optimizing parameters in the CBP process for enhanced aggregate performance.

1. Introduction

In the construction industry, aggregates serve as a fundamental component in the production of concrete, asphalt, and various other construction materials. Natural aggregates, sourced from materials such as crushed stone, gravel, and sand, have historically played a vital role in infrastructure development and construction projects. However, the escalating global demand for aggregates, both locally and internationally, has led to a decline in the availability of high-quality natural resources [1].

The past few decades have witnessed a substantial surge in infrastructure development and construction activities across numerous countries. Factors such as population growth, urbanization, and economic advancement have spurred this heightened demand for aggregates to meet the growing needs of construction projects. Consequently, this surge in demand has exerted immense pressure on the supply of natural aggregates [2,3].

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The utilization of artificial aggregates presents several potential advantages. Firstly, artificial aggregates offer a means to mitigate reliance on finite natural resources, thereby reducing the environmental impact associated with mining activities. Secondly, by utilizing recycled materials as raw ingredients for artificial aggregates, it becomes possible to diminish the volume of construction and industrial waste destined for landfills. Thirdly, artificial aggregates can exhibit superior control over their physical and mechanical properties compared to their natural counterparts, thereby enhancing the quality and uniformity of construction materials.

In the early 2000s, a novel approach to producing artificial lightweight aggregates emerged, known as the pelletization process, which is applied to dry powdered fly ash. This innovative method involves agglomerating fly ash particles in an inclined rotating pan at ambient temperature to yield fly ash pellets [2]. During the agglomeration process, water serves as a wetting agent, while Portland cement and/or lime act as binders. Notably, this method boasts significantly lower energy consumption to produce lightweight aggregates. Furthermore, given that the resulting artificial lightweight aggregates consist of 90% fly ash by weight, this process presents another environmentally favorable aspect [1].

According to pervious study [3], the demand for aggregates is increasing, causing a decrease in the supply of natural aggregate resources around the environment. Several developed countries such as America, Britain, and Poland have succeeded in creating effective and efficient artificial aggregates. As an alternative, the researchers developed lightweight geopolymer aggregates. Geopolymers are made from natural materials that contain large amounts of silicon and aluminum [2]. Natural materials containing a lot of silicon and aluminum, such as fly ash, can be used as a building block for artificial geopolymer aggregates. The combination of fly ash and sand can improve the characteristics of the aggregate. Fly ash is formed from burning coal and has a high content of silica and lime [3]. When reacted with water, the fine particles interact with silica oxide and calcium hydroxide, producing substances that can bind and exhibit cement hydration processes.

Assessing aggregate strength against impact constitutes a fundamental aspect of aggregate production, with the aggregate impact value (AIV) serving as a critical parameter. As per (British Standard) BS 812 part 112:1990, the permissible limit for aggregate destruction value (AIV) stands at 30% [4]. The strength of geopolymer aggregates can be influenced by factors such as the ratio of fly ash to alkaline activators (FA/AA) and the molarity of Sodium Hydroxide (NaOH). Fly ash, containing sodium (Na) and aluminum (Al) components, is renowned for its rapid reactivity [5-10].

Geopolymer aggregates, as discussed by Tajra, Elrahman, and Stephan in their 2019 review [11], represent a significant advancement in sustainable construction materials. These aggregates are produced through the cold-bonding process, which involves the chemical activation of aluminosilicate materials. The process not only reduces the energy consumption and carbon footprint associated with traditional aggregate production but also enhances the mechanical and durability properties of the resulting concrete. Geopolymer aggregates synthesized from local fly ash, as detailed by Nergis, Vizureanu, and Corbu in their 2019 study [12], present a promising alternative to conventional construction materials. These geopolymers are created by activating fly ash, an abundant industrial by-product, with an alkaline solution, resulting in a material with excellent binding properties. Geopolymer aggregates, are significantly influenced by the concentration of sodium hydroxide (NaOH) used in their production. Different NaOH concentrations affect the inter-particle gelation in fly ash-based geopolymer aggregates [13,14].

In their 2020 study, Gökçe *et al.,* [15] focus on the development of eco-efficient fly ash-based geopolymer aggregates with a reduced dosage of alkaline activator. By minimizing the amount of alkaline activator, such as sodium hydroxide or sodium silicate, the researchers aimed to lower the overall cost and environmental impact of the geopolymer production process. Their findings reveal that even with reduced activator dosages, the resulting geopolymer composites maintain satisfactory mechanical properties and durability.

This research aims to analyze the impact of the FA/AA ratio and NaOH molarity on both the mechanical and physical properties of artificial geopolymer aggregates.

2. Methodology

With the cold bond pelletization method, fly ash particles are agglomerated in an inclined rotating pan at room temperature to produce fly ash pellets. During agglomeration, water is used as a wetting agent while Portland cement and/or lime are used as a binder. This method requires much lower energy consumption for artificial light aggregate production. Since the artificial light aggregate produced by this method consists of 90% fly ash by weight, this can be considered another environmental benefit of the cold bond pelleting process. It can also be presented as an alternative solution to reduce the environmental impact of the approximately 500 million tonnes of fly ash produced worldwide. This type of aggregate can be used for concrete having various mechanical characteristics. Depending on the lightweight aggregate content in the concrete production, concrete with compressive strengths ranging between 20 and 80 MPa can easily be achieved [16]. If the fly ash contains a high silica content, then it can produce a hard a strong material [17,18].

After the geopolymer aggregates are formed, they remain soft and require curing. The curing process involves heating them at 80°C for 24 hours in an oven. This research phase was carried out sequentially from beginning to end. The several stages to get the results of this study are as follows.

2.1 Preparation

The first stage is the preparation of materials and tools to be used in mixing geopolymer-made aggregates. The materials prepared are sand, fly ash, and alkaline activator solution consisting of NaOH and Na_2SiO_3, and distilled water. The equipment prepared is a mixer, beaker, measuring cup, and oven.

In geopolymer-made aggregates, alkaline activator solution is the main material that is useful for making polymer bonds with fly ash. Na_2SiO_3 is in the form of a gel solution and NaOH is in the form of solid chunks. Solid NaOH needs to be dissolved first with distilled water, after that it is mixed with Na_2SiO_3. The following are the steps for preparing an alkaline activator solution:

i. Prepare the necessary NaOH to make the required molarity, 1 Mol NaOH as much as 40 grams. We're using molarities of 13, 15, and 17. So we need 520 g, 600 g, 720 g (Figure 1).
ii. Dissolve the NaOH using distilled water until the solution reaches 1000 mL in the beaker and stir with a stir stick until the NaOH is completely dissolved. After that, the solution will react and become hot. Then let stand for 24 hours until the solution reaches room temperature (Figure 2).
iii. Mixing Na_2SiO_3 and NaOH that has been prepared (Figure 3).

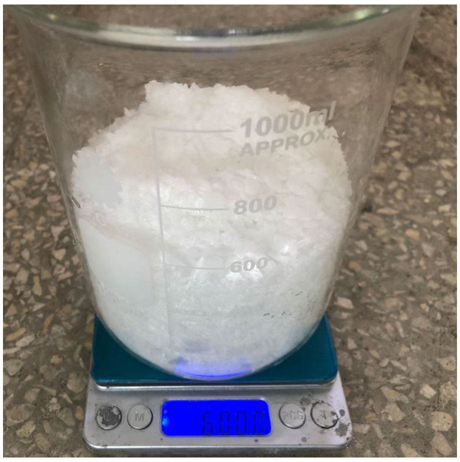

Fig. 1. NaOH in a measuring cup

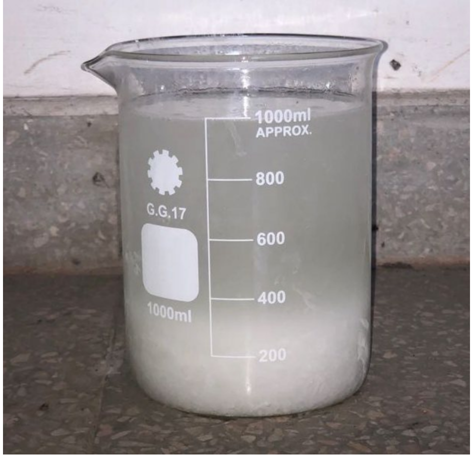

Fig. 2. Mixing NaOH with Aquades

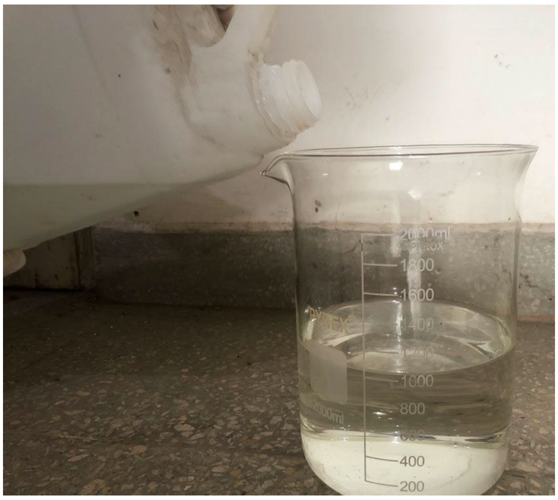

Fig. 3. Mixing the NaOH solution with Na_2SiO_3

2.2 Sand and Fly Ash Testing

The second stage is the stage carried out for testing sand and fly ash. This test was carried out to determine the characteristics of the sand and the composition of the fly ash and sand. The following is a sand test carried out according to (American Society for Testing and Materials) ASTM standards:

i. Sieving analysis test based on ASTM C136 [19]
ii. Testing for organic content in sand based on ASTM C40 [20]
iii. Testing the clay lumps based on ASTM C142 [21]
iv. Test for specific gravity and water absorption based on ASTM C128 [22]

For fly ash test, the following tests are conducted:

i. X-Ray Diffraction (XRD) Testing
ii. X-Ray Fluorescence (XRF) Testing
iii. Scanning Electron Microscope (SEM) Testing

2.3 Planning

The third stage involves planning and preparing the mix composition or mix design for the geopolymer aggregates. This is done by collecting data from previous research and conducting laboratory trials. The planned mix design is informed by these trials, ensuring an optimized composition for the geopolymer aggregates. A summary of the mix design includes the selection of raw materials, the ratio of fly ash to alkaline activator, and the molarity of NaOH, which were all determined based on their impact on the physical and mechanical properties of the final product.

2.4 Mixing

The fourth stage is the mixing of geopolymer-made aggregate constituent materials. Samples using fly ash to alkaline activator ratios, namely ratios 3, 3.25, and 3.5 were combined with samples with molarities of 13, 15, and 17. Each variation had 3 samples, resulting in a total of 45 samples.

The Mix design used are as follows (Table 1):

Table 1
Mix Design

Variation	Sample Size	Fly Ash (kg)	Sand (kg)	Alkaline Activator (kg)	Na_2SiO_3 (kg)	NaOH (kg)	Weight Total (kg)
1	3	3.4	0.68	0.971	0.756	0.216	15.15
2	3	3.4	0.68	1.046	0.814	0.232	15.38
3	3	3.4	0.68	1.133	0.881	0.252	15.64
4	3	3.4	0.68	0.971	0.756	0.216	15.15
5	3	3.4	0.68	1.046	0.814	0.232	15.38
6	3	3.4	0.68	1.133	0.881	0.252	15.64
7	3	3.4	0.68	0.971	0.756	0.216	15.15
8	3	3.4	0.68	1.046	0.814	0.232	15.38
9	3	3.4	0.68	1.133	0.881	0.252	15.64

Material mixing is carried out at the Material Construction Structure Laboratory, Sriwijaya University. Initially, the mixing of sand and fly ash was carried out according to the mix design at room temperature. Then prepare a mixture of alkaline activator. Prepare the pelletization tool and set the tool to 45^o. Also, a sprayer contains an alkaline activator solution so that it is evenly distributed during mixing.

Turn on the pelletization tool and then enter the dry mixture, and then turn on the machine. After that, spray the alkaline activator onto the dry mixture slowly and steadily for 10 minutes to produce a graded aggregate. Then after finishing spraying the alkaline activator, we wait another 10 minutes until the artificial aggregate is formed.

After the geopolymer-made aggregates are formed, the aggregates are still in a soft state, so it is time for curing. Curing was carried out at 80^oC for 24 hours in the oven. After that, the geopolymer-made aggregate has hardened and is ready to be tested according to ASTM.

2.5 Geopolymer Aggregate Testing

The last stage is the testing stage of the geopolymer-made aggregate test object. For this stage, several tests were carried out, as follows:

i. Sieving analysis test based on ASTM C136 [19]
ii. Moisture content testing based on ASTM C566 [23]
iii. Specific gravity and water absorption testing based on ASTM C127 [24]
iv. Testing Aggregate Impact Value based on BS 812-112: 1990 [25]

3. Results

3.1 Fine Aggregate Testing

Table 2 is the result of testing the aggregate. The test results show that the properties of fine aggregate can be used as a mixture of geopolymer-made aggregates.

Table 2
Fine Aggregate Test Results

No.	Characteristics	Results	Specification
1.	Fineness modulus	2.34	ASTM C136
2.	Gradation Area	Zone 3	ASTM C136
3.	(Saturated, Surface Dry) 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 (XRD) testing

Based on the results of the XRD 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 few crystal peaks in the fly ash indicate that the fly ash has reactive properties and dissolves easily.

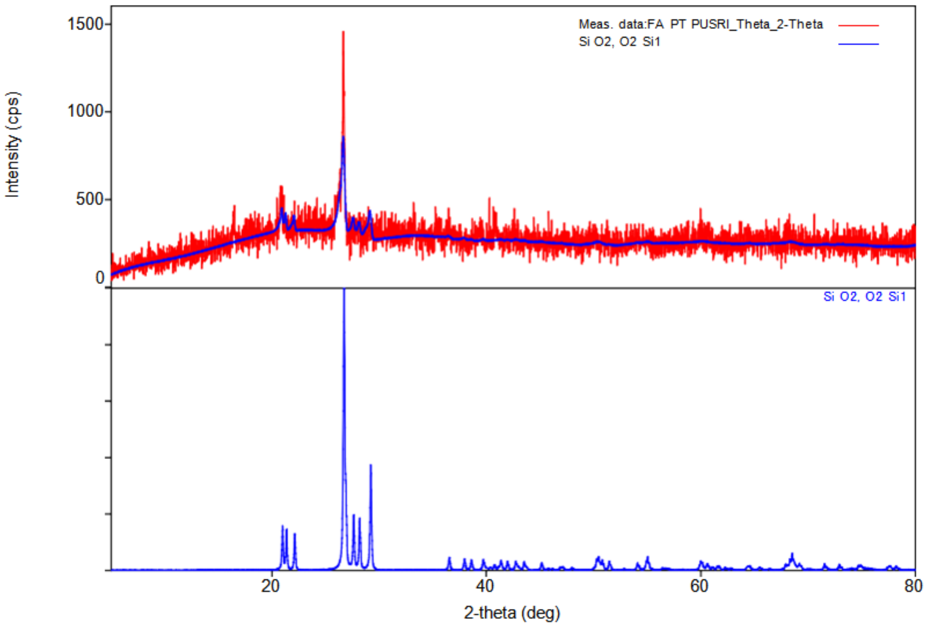

Fig. 2. XRD Test Results [26]

3.2.2 X-Ray Fluorescence (XRF) testing

A From the test results (Table 3), it can be seen that the fly ash used contains CaO < 10%. This signifies that the fly ash falls into category class F.

Table 3
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 Microscope (SEM) testing

From the SEM test (Figure 5), it was obtained that the dominant fly ash grains were round in shape and had an average particle size of 2 µm. According to research [27] the particle size of fly ash also influences the geopolymer mortar. The finer the fly ash is the more increased workability and compressive strength of mortars geopolymer.

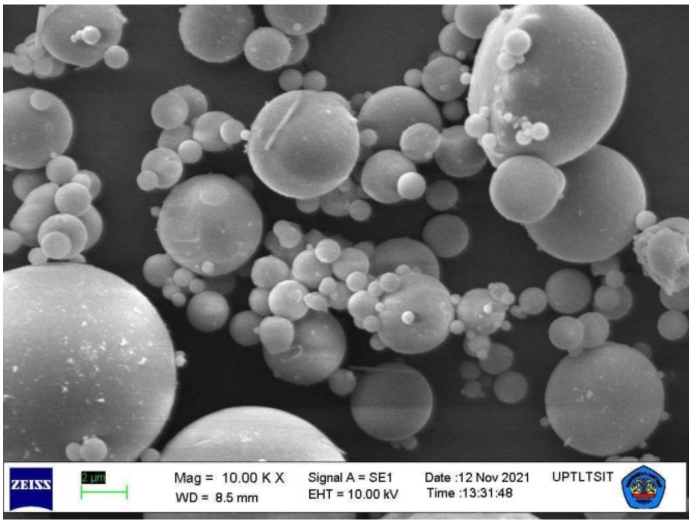

Fig. 3. SEM test results

3.3 Natural Coarse Aggregate Testing

The coarse aggregate material used in this experiment is crushed rock obtained from Sinar Musi Limited Liability Company (LLC). The size of the coarse aggregate used ranges from 5 mm to 20 mm. The purpose of testing on coarse aggregate is to compare the results of tests on artificial aggregates. A series of tests will be carried out on this coarse aggregate, including testing for moisture content, sieving, specific gravity and water absorption, unit weight, and AIV (Table 4).

Table 4
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 Artificial Aggregates Pelletization Method and Cold Bond Pelletization Method

Table 5
Research data

Data	Information	Number of Samples
Sand	The weight of the sand uses 20% of the weight of *the fly ash*	
Na_2SiO_3 /NaOH	3,5	
curing	Baked at 80 °C for 24 hours	
13 Mol; 3 FA/AA Variation	NaOH 13 mol and the ratio of *fly ash* to alkali activator 3	3
15 Mol; 3 FA/AA Variation	NaOH 15 mol and the ratio of *fly ash* to alkali activator 3	3
17 Mol; 3 FA/AA	NaOH 17 mol and the ratio of *fly ash* to alkali activator 3	3

Variation		
13 Mol; 3.25 FA/AA Variation	NaOH 13 mol and the ratio of *fly ash* to alkali activator 3,25	3
15 Mol; 3.25 FA/AA Variation	NaOH 15 mol and the ratio of *fly ash* to alkali activator 3,25	3
17 Mol; 3.25 FA/AA Variation	NaOH 17 mol and the ratio of *fly ash* to alkaline activator 3,25	3
13 Mol; 3.5 FA/AA Variation	NaOH 13 mol and the ratio of *fly ash* to alkali activator 3,5	3
15 Mol; 3.5 FA/AA Variation	NaOH 15 mol and the ratio of *fly ash* to alkali activator 3,5	3
17 Mol; 3.5 FA/AA Variation	NaOH 17 mol and the ratio of *fly ash* to alkali activator 3,5	3
Testing time	The test is carried out at least 7 days after the specimen is made and *cured* for 24 hours	
Test treatment	Two types of tests were carried out, namely physical and mechanical testing of the aggregate according to the standards used	
Test standard	The standards used are ASTM and BS	

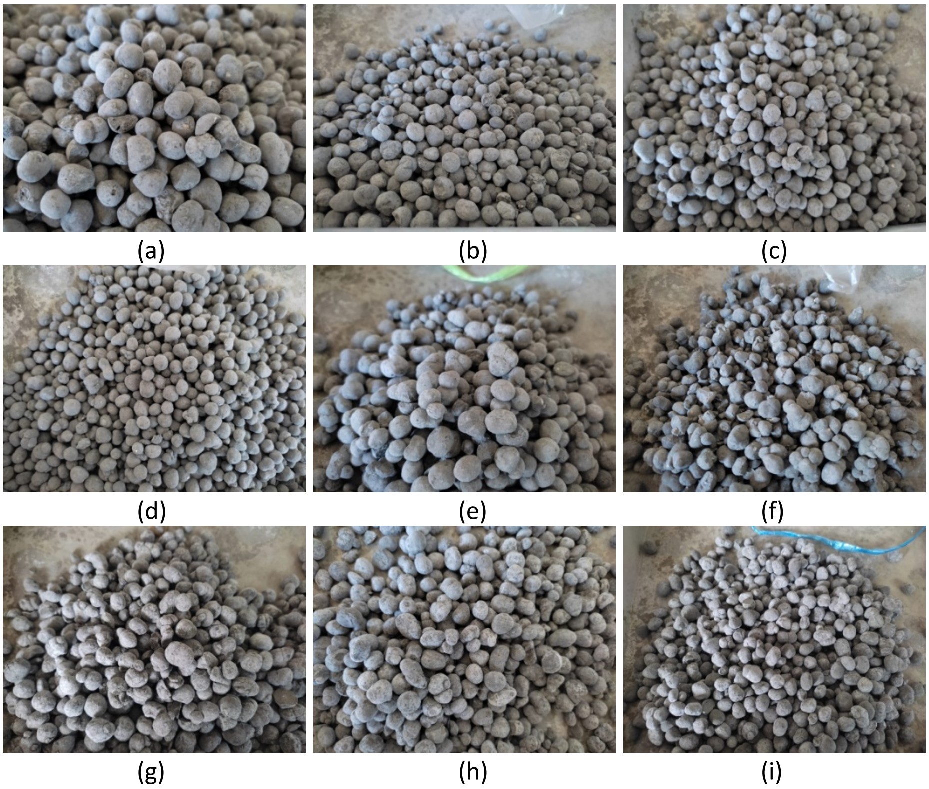

Fig. 4. Pelletizing Aggregate Variation (a) 13 Mol; 3 FA/AA (b) 15 Mol; 3 FA/AA (c) 17 Mol; 3 FA/AA (d) 13 Mol; 3.25 FA/AA (e) 15 Mol; 3.25 FA/AA (f) 17 Mol; 3.25 FA/AA (g) 13 Mol; 3.5 FA/AA (h) 15 Mol; 3.5 FA/AA (i) 17 Mol; 3,5 FA/AA

3.4.1 Moisture content testing

Moisture content (Figure 7) testing is carried out to determine the water content contained in pelletizing and crushing aggregates. This test is carried out according to ASTM C566 [23].

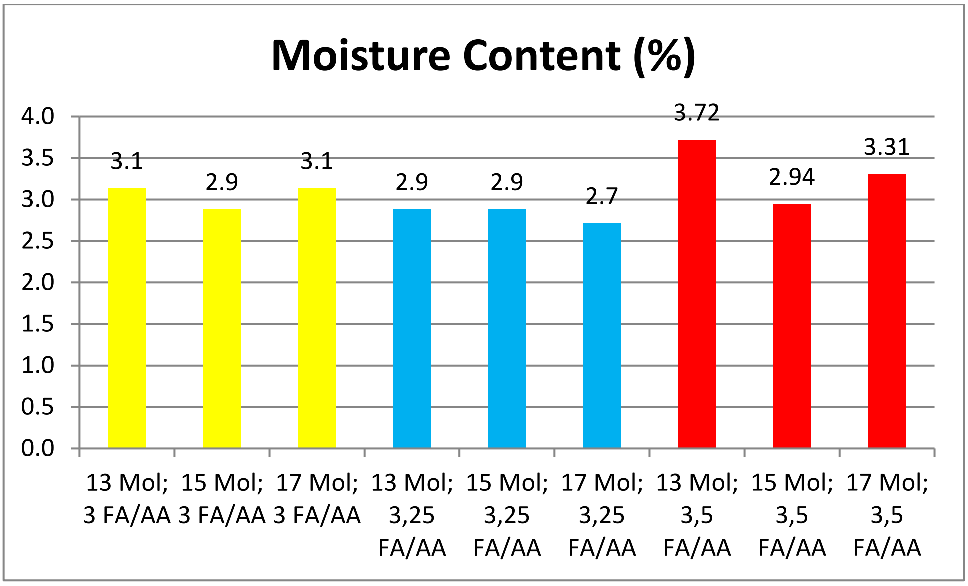

Fig. 5. Moisture content graph of aggregate geopolymer

3.4.2 Sieve analysis testing

Sieve analysis (Table 6) was carried out to see the modulus fineness and gradation graphs contained in the artificial pelletizing and crushing aggregates that were made based on ASTM C136 [19].

Table 6
The value of fineness modulus and area of pelletizing aggregate gradation

Variation	Fineness Modulus	Gradation Area
13 Mol; 3 FA/AA Variation	2.36	Size Number 7
15 Mol; 3 FA/AA Variation	2.39	Size Number 7
17 Mol; 3 FA/AA Variation	2.47	Size Number 7
13 Mol; 3.25 FA/AA Variation	2,42	Size Number 7
15 Mol; 3.25 FA/AA Variation	2.45	Size Number 7
17 Mol; 3.25 FA/AA Variation	2.48	Size Number 7
13 Mol; 3.5 FA/AA Variation	2.49	Size Number 7
15 Mol; 3.5 FA/AA Variation	2.49	Size Number 7
17 Mol; 3.5 FA/AA Variation	2.51	Size Number 7

All of the variation are in gradation area size number 7 based on ASTM C33/33M [28].

3.4.3 Specific gravity testing and water absorption

Specific gravity (Figure 8) and water absorption (Figure 9) tests were carried out based on ASTM C127 standards [24].

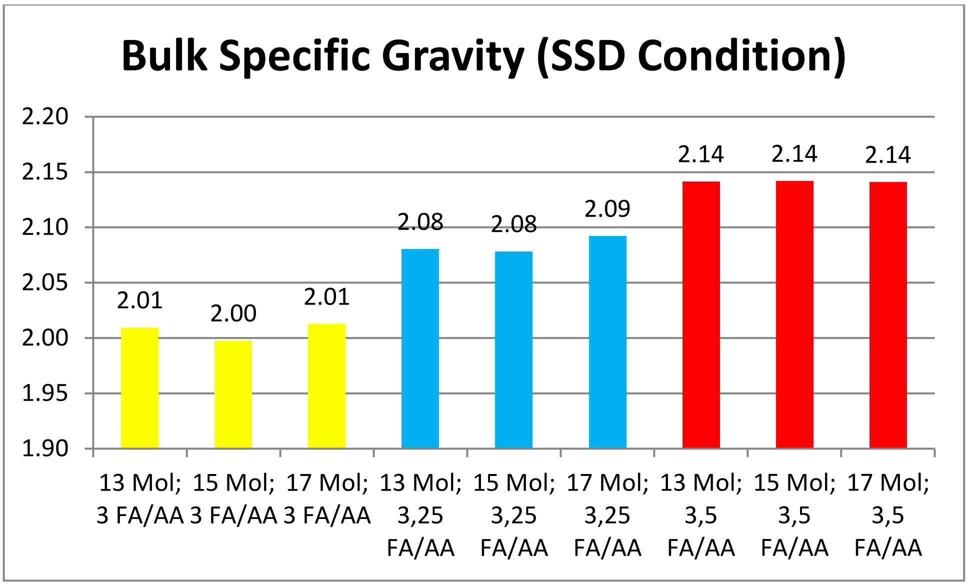

Fig. 6. Bulk specific gravity (SSD condition) graph of aggregate geopolymer

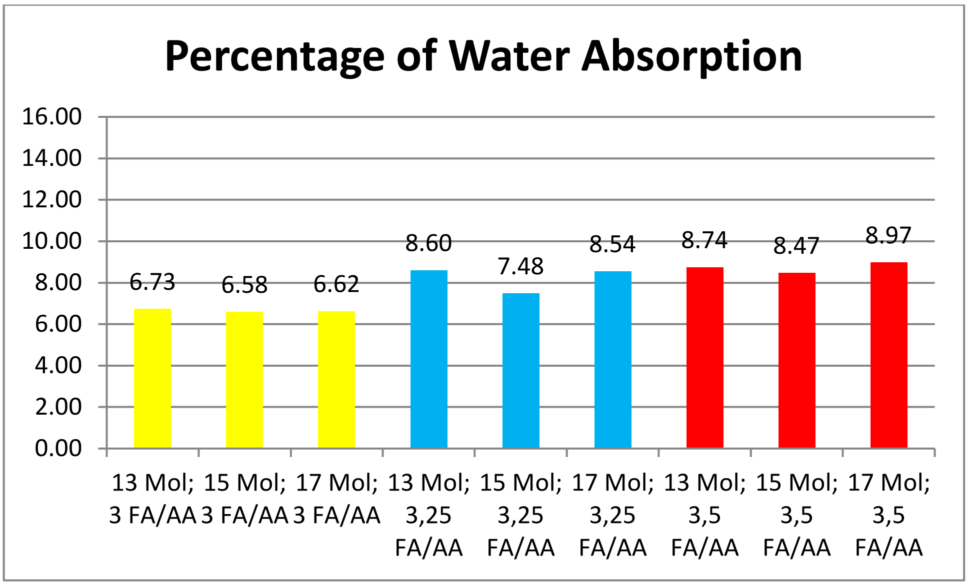

Fig. 7. Water absorption percentage graph of aggregate geopolymer

3.4.4 AIV testing

AIV or Aggregate Impact Value is an aggregate test (Figure 10) to determine the strength of the aggregate based on BS 812-112: 1990. The lower the percentage of AIV value, the better the aggregate toughness.

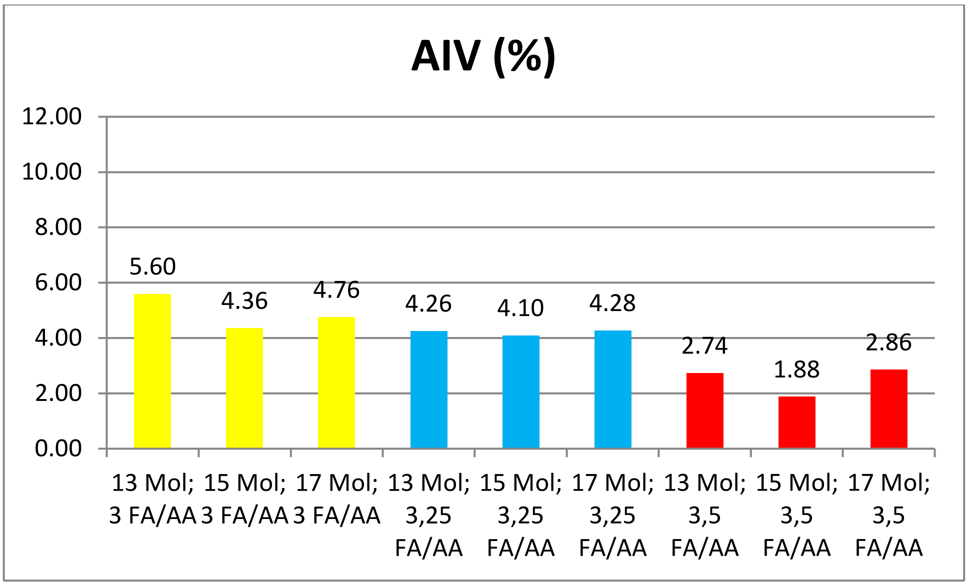

Fig. 8. AIV graph of aggregate geopolymer

Each variation was tested three times and then the average was taken. The lowest value was obtained at the 15 Mol variation; 3.5 FA/AA with a value of 1.88% and the highest value in the 13 Mol variation; 3 FA/AA with a value of 5.60%. The lower the percentage values of AIV, the higher the toughness of the aggregate. Based on BS 812-112: 1990, an AIV value below 30% is considered to be of good quality. If the AIV is above 30% then it is not suitable for use.

4. Conclusions

From the research that has been done, the following are the conclusions obtained from the tests that have been carried out on pelletizing aggregates:

i. The addition of the ratio of fly ash to alkaline activator (FA/AA) affects the AIV percentage value in geopolymer-made aggregates using the cold bond pelletization method. The AIV value of geopolymer aggregates from variations FA/AA 3 to variations FA/AA 3.25 experienced an average decrease of 0.69%. Then it decreased again from the FA/AA variation of 3.25 to the FA/AA variation of 3.5 of 1.72%. The effect of the FA/AA values on the physical properties of aggregates can be seen from the tests that have been carried out, that the higher the FA/AA, the higher the physical values of the aggregates except for the water content test.
ii. Effect of NaOH molarity on the mechanical properties of geopolymer-made aggregate by cold bond pelletization method in aggregate impact value (AIV) testing. The value decreased by an average of 0.75 % at molarity of 13 molar to 15 molar. However, it experienced an increase in the 17 molar variation of 0.52 %. The effective molarity value for geopolymer-made aggregates using the cold bond pelletization method is 15 molar, which has the lowest AIV percentage value. The impact of the FA/AA ratio on the physical characteristics of the aggregate can be observed from the test results. It can be concluded that there was a decrease in the values of the physical properties of the aggregate from a variation of 13 mol to 15 mol, but an increase from 15 mol to 17 mol.

At 17 molar NaOH, the geopolymer aggregate sample experienced an increase in AIV value. This is caused by the speed of geopolymerization which is too high due to the high concentration of NaOH. If the concentration of NaOH is too high, there will be an excessive increase in the number of OH- ions. This excess of OH- ions interferes with the polymerization reaction and inhibits the geopolymerization process from running efficiently.

These findings underscore the importance of fine-tuning both the FA/AA ratio and NaOH molarity to achieve desired mechanical and physical properties in geopolymer aggregates. Future research should further explore the FA/AA ratio, as the current study did not identify the optimal peak for the AIV percentage. Practical applications of these results could lead to the development of more durable and efficient geopolymer-based construction materials.

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