

Effect of Activator Composition on the Setting Time and Compressive Strength Of Fly Ash-Lime-Based Geopolymer Paste

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Abstract: This study aims to investigate the optimal composition of geopolymer paste based on fly ash and lime to achieve ambient temperature hardening, ideal setting time, and high compressive strength. The primary materials used include Class F fly ash from the Mpanau coal-fired power plant and hydrated lime (calcium hydroxide) at 5% of the fly ash weight. The activators employed were Sodium Silicate (Na_2SiO_3) and Sodium Hydroxide (NaOH), with Na_2O dosage variations of 7.5%, 10%, and 12.5%, and activator modulus ($\text{SiO}_2/\text{Na}_2\text{O}$ ratio) variations of 0.75, 1.00, and 1.25. The specimens were cylindrical with a diameter of 25 mm and a height of 50 mm, and compressive strength tests were conducted at 3, 7, 14, and 28 days. The results showed that the optimum setting time was achieved at a Na_2O dosage of 7.5% with an activator modulus of 0.75 and 1.00, yielding setting times of 88.67 and 60.94 minutes, respectively. The highest compressive strength was recorded at a Na_2O dosage of 10% with an activator modulus of 1.25, reaching 29.76 MPa at 28 days. These findings suggest that the composition of the alkaline activator significantly influences the early-age properties and mechanical performance of fly ash-lime-based geopolymer paste.

Keywords: fly ash, lime, activator, dosage, geopolymer, setting time, compressive strength

Abstrak: Penelitian ini dimaksudkan untuk mengkaji komposisi ideal pada pasta geopolimer berbahan dasar abu terbang dan kapur, guna menghasilkan pengerasan pada suhu ruang, waktu pengikatan optimal, serta kekuatan tekan yang tinggi. Material utama yang digunakan adalah abu terbang tipe F dari PLTU Mpanau dan kapur padam (kalsium hidroksida) sebanyak 5% dari berat abu terbang. Sebagai aktivator digunakan Sodium Silikat (Na_2SiO_3) dan Sodium Hidroksida (NaOH) dengan variasi dosis Na_2O sebesar 7,5%, 10%, dan 12,5%, serta variasi modulus aktivator (rasio $\text{SiO}_2/\text{Na}_2\text{O}$) 0,75; 1,00; dan 1,25. Benda uji berbentuk silinder (diameter 25 mm dan tinggi 50 mm) kemudian diuji kekuatan tekan pada umur 3, 7, 14, dan 28 hari. Hasil penelitian menunjukkan bahwa waktu pengikatan optimal diperoleh pada komposisi Na_2O 7,5% dengan modulus aktivator 0,75 dan 1,00, masing-masing sebesar 88,67 dan 60,94 menit. Sementara itu, sampel dengan dosis Na_2O 10% dan modulus aktivator 1,25 menghasilkan kekuatan tekan tertinggi yaitu 29,76 MPa pada umur 28 hari. Temuan ini diharapkan dapat memberikan kontribusi signifikan dalam pengembangan bahan geopolimer yang ramah lingkungan dan memiliki performa mekanik unggul.

Kata kunci: abu terbang, kapur, aktivator, dosis, geopolimer, waktu pengikatan, kekuatan tekan

1. Introduction

Sustainable infrastructure development requires the use of environmentally friendly materials that can reduce dependence on Portland cement. One promising alternative is geopolymer, an inorganic binder produced through the reaction of silica- and alumina-rich materials with alkaline solutions. Geopolymers present significant potential as a substitute for conventional cement, offering a lower carbon footprint and the ability to utilize industrial by-products as raw materials, such as fly ash [1]. Fly ash is a solid by-product generated from coal combustion in coal-fired power plants (CFPP), and its volume continues to increase in line with the growing national demand for fossil energy. In Indonesia, Central Sulawesi Province is among the regions with the highest utilization of CFPPs, particularly due to the presence of large-scale industrial zones such as the Indonesia Morowali Industrial Park (IMIP), which relies on CFPPs as its primary energy source [2]. The production of fly ash in this region is abundant; however, its utilization remains very limited, with the majority being disposed of in landfills, thereby posing potential environmental risks. [2].

Therefore, the utilization of fly ash as the primary precursor for geopolymer paste represents a strategic solution in terms of both environmental sustainability and material efficiency in construction. Previous studies by Hardjito and Rangan have demonstrated that fly ash can produce geopolymer paste and concrete with high compressive strength, even surpassing that of conventional Portland cement-based concrete [3]. Meanwhile, Chindaprasirt et al [4]. reported that the characteristics of geopolymers are strongly influenced by the type and composition of the activator, including the ratio of sodium silicate to sodium hydroxide as well as the dosage of alkali ions such as Na_2O [5].

Further studies have also shown that the addition of lime (Ca(OH)_2) to fly ash mixtures can accelerate the early setting process by increasing the availability of calcium, which promotes the formation of C-A-S-H structures alongside N-A-S-H in the geopolymer system [6][7]. However, there are still limited studies addressing the specific effects of activator composition—both dosage and modulus—on the setting time and compressive strength of fly ash-lime-based geopolymer paste. Taking into account

the condition of Central Sulawesi as a region with significant coal-fired power plant waste generation, this study aims to evaluate the influence of activator composition variations on the setting time and compressive strength of geopolymer paste, thereby providing a more environmentally friendly and value-added construction alternative utilizing local industrial waste [8]

2. Research Method

This study employs an experimental approach to analyze the influence of activator composition on the setting time and compressive strength of geopolymer paste produced from fly ash and lime. [8]. The research methodology comprises four main stages: material preparation, sample fabrication, testing, and data analysis.

2.1. Material and Mix Design

This study utilized Type F fly ash obtained from the Mpanau coal-fired power plant, with its characteristics presented in Table 1, hydrated lime (calcium hydroxide) as shown in Table 2, sodium silicate (Na_2SiO_3), and sodium hydroxide (NaOH). The activator solution was prepared with variations in Na_2O content of 7.5%, 10%, and 12.5%, as well as different activator moduli, expressed as the SiO_2 -to- Na_2O ratio of 0.75, 1.00, and 1.25, according to the mix design presented in Table 3. The equipment employed in this research included a mixer for material blending, cylindrical molds with a diameter of 25 mm and a height of 50 mm, a compressive strength testing machine for mechanical property evaluation, and a setting time apparatus for analyzing the initial setting characteristics of the mortar.

Table 1. Fly ash composition

No.	Parameter	Fly Ash%
1	SiO_2	56,04
2	Fe_2O_3	21,27
3	Al_2O_3	12,99
4	CaO	5,18
5	K_2O	1,62
6	TiO_2	0,90
7	MnO	0,26
8	P_2O_5	0,25
9	Other Oxide	0,57
10	Loss of Setting Heat	0,9
Total		100

Table 2. Lime composition

No.	Parameter	Lime %
1	CaO	85,04
2	SrO	2,17
3	Cl	1,06
4	K_2O	1,03
5	Fe_2O_3	0,35
6	TiO_2	0,03
7	Other Oxide	0,10
8	Loss of Setting Heat	10.25
Total		100

Table 3. Geopolymer + lime mix design

Mix Code	Dosis Na_2O (%)	Modulus Aktivator	Lime Content (%)	Binder
FC7,5 - 0,75	7,5	0,75	5	Fly Ash + Aktivator
FC7,5 - 1,00	7,5	1	5	Fly Ash + Aktivator
FC7,5 - 1,25	7,5	1,25	5	Fly Ash + Aktivator
FC10 - 0,75	10	0,75	5	Fly Ash + Aktivator
FC10 - 1,00	10	1	5	Fly Ash + Aktivator
FC10 - 1,25	10	1,25	5	Fly Ash + Aktivator
FC12,5-0,75	12,5	0,75	5	Fly Ash + Aktivator
FC12,5-1,00	12,5	1	5	Fly Ash + Aktivator
FC12,5-1,25	12,5	1,25	5	Fly Ash + Aktivator
PPCC	-	-	-	Pcc cement

2.2. Geopolymer Paste

The preparation of geopolymer paste began with mixing the raw materials, namely Type F fly ash and hydrated lime (calcium hydroxide), where the lime content was fixed at 5% of the fly ash weight. Both materials were dry-mixed using a mechanical mixer until a homogeneous blend was obtained. The activator solution was prepared by combining sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) in

various proportions. The activator variations included Na_2O contents of 7.5%, 10%, and 12.5%, as well as activator moduli (molar ratio of SiO_2 to Na_2O) of 0.75, 1.00, and 1.25, in accordance with the designated mix compositions. [9]. The activator solution was gradually added to the fly ash–lime mixture while continuously stirred until a homogeneous paste was obtained. The resulting paste was then immediately placed into cylindrical molds with a diameter of 25 mm and a height of 50 mm, followed by

compaction to eliminate entrapped air voids. After casting, the samples were stored under controlled room conditions ($25 \pm 2^\circ\text{C}$) until the designated testing ages, without any additional external heat treatment, in accordance with the adapted methodology from [8]. This procedure was intended to ensure consistency in the formation of the geopolymer structure and to minimize variability during the hardening process.

The setting time of the paste was measured using a Vicat apparatus, following the standard procedure [10]. The tests were conducted until the initial and final setting times were achieved. Subsequently, compressive strength tests were carried out at ages of 3, 7, 14, and 28 days using a compression testing machine with a loading rate of 0.5 MPa/s, in accordance with the standard procedure [11]. The compressive strength values were obtained from the average of three samples for each variation..

The experimental data were statistically analyzed to evaluate the influence of Na_2O dosage and activator modulus variations on the mechanical properties of the geopolymer paste. Analysis of variance (ANOVA) and linear regression were employed to identify significant relationships among the investigated variables [12].

3. Result and Discussion

3.1. Time Setting

The setting time test represents an essential step in the initial characterization of geopolymer materials (Table 3). The primary objective of this test is to determine the duration required for geopolymer paste from the initial mixing stage until reaching specific hardening conditions. In this study, the test was carried out on geopolymer paste samples using a standard Vicat apparatus, which is specifically designed to measure both the initial and final setting times based on the resistance of a standard needle penetrating into the paste. The testing procedure was initiated immediately after all geopolymer components were mixed, including the aluminosilicate source material and the alkaline activator solution.

Table 4. Setting time for each variation

Mix Code	Initial Binding Time (Minute)	Final Binding Time (Minute)
FC7,5 - 0,75	88,67	135,00
FC7,5 - 1,00	60,94	105,00
FC7,5 - 1,25	26,36	75,00
FC10 - 0,75	195,47	315,00
FC10 - 1,00	178,61	270,00
FC10 - 1,25	162,55	255,00
FC12,5-0,75	920,96	990,00
FC12,5-1,00	621,58	810,00
FC12,5-1,25	542,01	705,00
PPCC	110,74	240,00

The mixture variations were determined based on two key parameters: the Na_2O (sodium oxide) dosage, expressed as a percentage of the binder weight, and the activator

modulus, defined as the molar ratio between silica (SiO_2) and sodium oxide (Na_2O) in the activator solution. These two parameters strongly influence the rate of geopolymerization reactions, thereby directly affecting the setting time (Figure 1).

The results of this test provide an overview of how quickly the geopolymer paste begins to harden, as well as the influence of compositional variations on the setting behavior. Such information is highly important in the design of geopolymer materials, particularly for construction applications that require controlled workability and setting time.

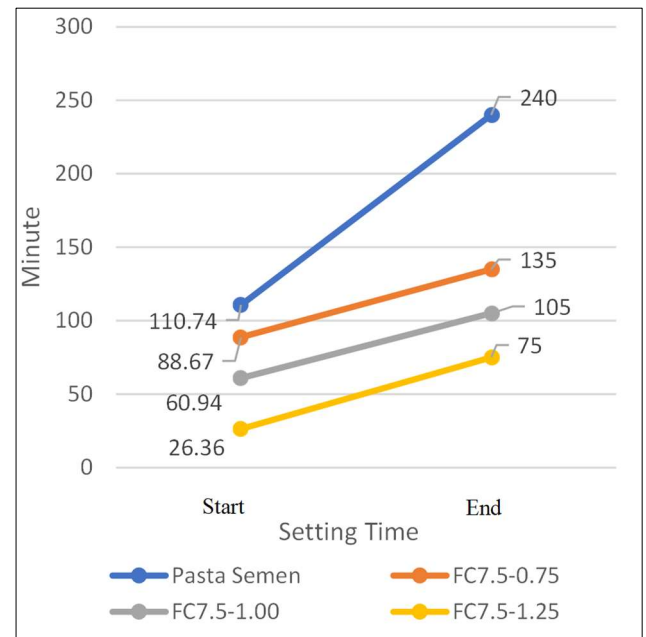


Figure 1. Relationship between activator modulus and setting time at a Na_2O dosage of 7.5%

An increase in the activator modulus exhibited a linear relationship with the reduction in the setting time of the geopolymer paste. Higher modulus values resulted in a faster setting process and a thicker paste consistency (Figure 2). The average difference in initial setting time among the modulus variations was recorded at 31.16 minutes, while the average duration from initial to final setting reached 46.34 minutes. This phenomenon is consistent with previous findings, which reported that increasing the silicate-to-alkali ratio accelerates the polymerization reaction due to the formation of a denser and faster-developing geopolymer network [13].

The test results indicated that increasing the activator modulus at each Na_2O dosage consistently accelerated the setting time of the geopolymer paste. At a Na_2O dosage of 7.5%, the setting time was faster than that of conventional cement paste. However, samples with moduli of 0.75 and 1.00 remained within the initial setting time range specified by SNI 03-6825-2002, namely 60–120 minutes. In contrast, a modulus of 1.25 produced an initial setting time outside this range, making it less ideal as it led to rapid hardening and difficulties in compaction. At a Na_2O dosage of 10%, the trend of reduced setting time was still evident. The

average difference in initial setting time among the moduli became smaller (16.46 minutes), while the duration from initial to final setting increased (averaging 101.12 minutes). This provided a longer workable time, thereby facilitating the casting process (Figure 3).



Figure 2. Relationship between activator modulus and setting time at a Na

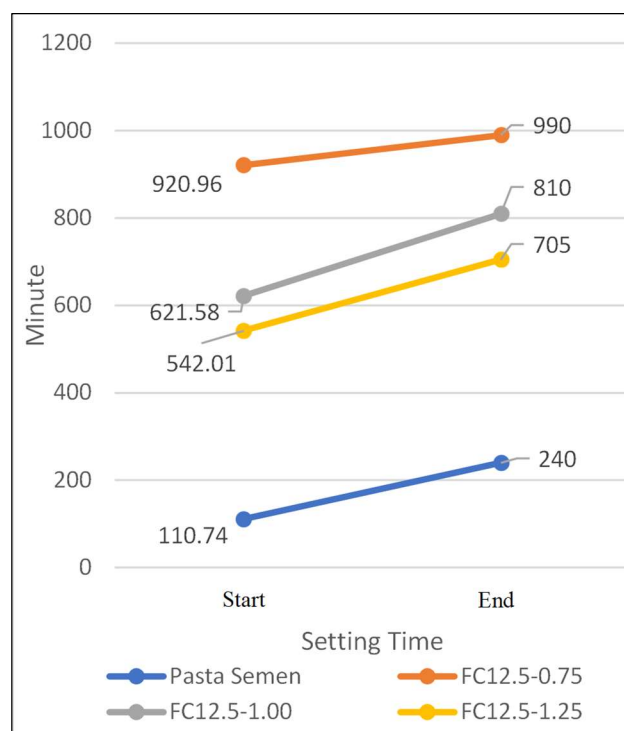


Figure 3. Relationship between Activator Modulus and Setting Time at a Na₂O Dosage of 10%

Conversely, a Na₂O dosage of 12.5% resulted in a much larger difference in initial setting times among the modulus variations (up to 299.38 minutes), with an average duration

from initial to final setting reaching 140.15 minutes. Samples at this dosage tended to be overly fluid and difficult to shape optimally. Overall, the fastest setting times—both initial and final—were observed in samples with a modulus of 1.25, while the longest setting times were consistently recorded in samples with a modulus of 0.75 across all Na₂O dosages tested. These findings confirm that increasing the activator modulus accelerates the formation of geopolymer structures through the enhanced rate of silicate polycondensation [14][15].

A review of the effect of Na₂O dosage revealed that increasing Na₂O concentration generally extended the setting time of geopolymer paste across all activator modulus variations. The increase in Na₂O dosage also influenced the differences in setting time among the modulus values. This difference increased from the 7.5% to the 10% dosage but decreased again at the 12.5% dosage. Such a pattern indicates the existence of an optimum threshold in the effect of alkali dosage on the dynamics of the polycondensation reaction and the formation of the geopolymer structure [16][17].

3.2. Compressive Strength of Geopolymer Paste

The compressive strength test was carried out on geopolymer paste samples using a compression testing machine in accordance with the standard procedure [18]. Cubic molds with dimensions of 50 mm were used for specimen preparation. The tests were conducted after the samples had reached their final setting time, covering all variations of Na₂O dosage and activator modulus applied in this study.

The compressive strength tests revealed that increasing the activator modulus consistently enhanced the strength of the geopolymer paste, as shown in Figure 4. At a Na₂O dosage of 7.5%, the highest compressive strength was achieved at a modulus of 1.25 with a value of 23.64 MPa, while the lowest was recorded at a modulus of 0.75 with 22.01 MPa. The most significant strength gain occurred between 3 and 14 days, after which the rate of increase slowed. Over the 3–28 day period, the strength improvement ranged from 3 to 10 times, depending on the modulus value.

A similar trend was observed at a Na₂O dosage of 10%, as shown in Figure 5, where a modulus of 1.25 yielded the highest compressive strength of 39.95 MPa, and a modulus of 0.75 produced the lowest value of 24.46 MPa. However, the significant strength gain occurred earlier, between 3 and 7 days, and tended to plateau thereafter. Compressive strength from 3 to 28 days increased approximately 2 to 3 times (Figure 4, Figure 5, and Figure 6).

Conversely, at a Na₂O dosage of 12.5%, the increase in compressive strength did not continue. The maximum value was still observed at a modulus of 1.25 (28.13 MPa), but overall the compressive strength decreased compared to the 10% dosage. Although significant gains still occurred between 3 and 14 days, the growth rate slowed again by day 28, with a total strength increase of only 3 to 5 times, as shown in Figure 6. These results are consistent with [19][20].

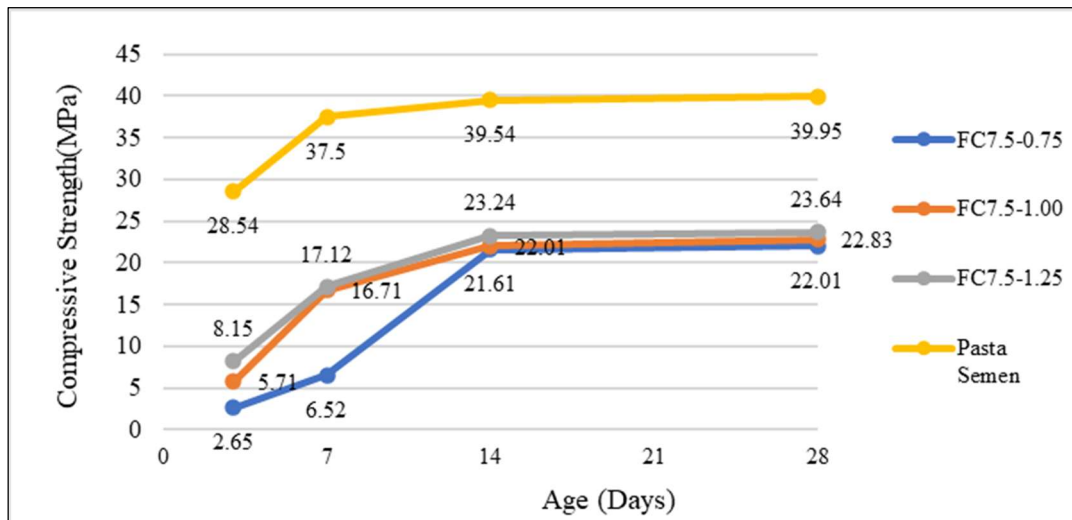


Figure 4. Compressive strength of geopolymer paste at a Na_2O dosage of 7.5%

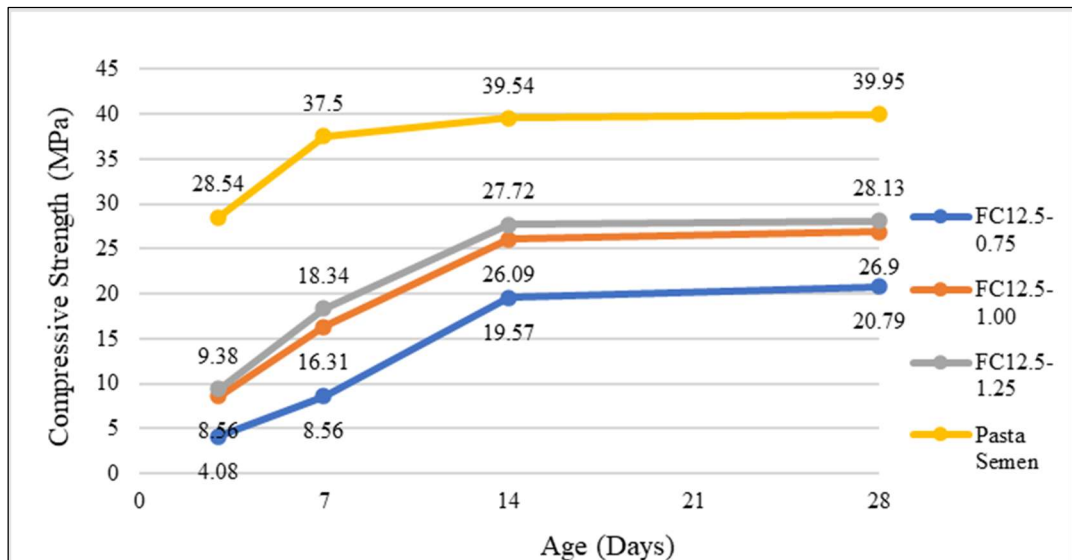


Figure 5. Compressive strength of geopolymer paste at a Na_2O dosage of 7.5%

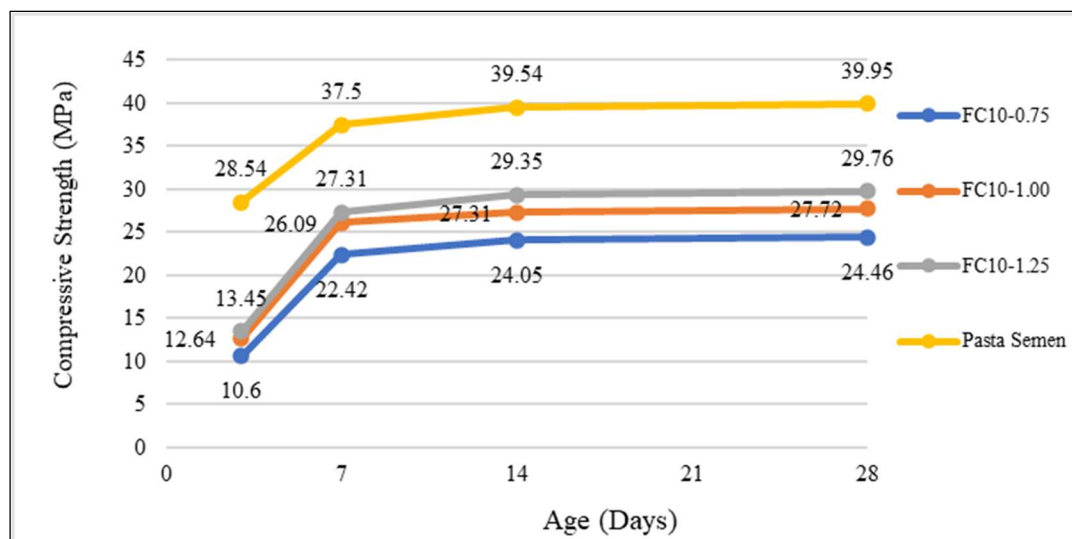


Figure 6. Compressive strength of geopolymer paste at a Na_2O dosage of 7.5%

3.3. Visual Observation

The geopolymer paste with a Na₂O dosage of 12.5% exhibited visual signs of efflorescence, characterized by

white crystallization on the sample surface starting at 14 days, as shown in Figure 7. This phenomenon indicates an excess of alkali activator that did not participate in the polycondensation reaction [19].



Figure 7. Visual Observation of specimens at ages 3, 7, 14, and 28 days

As the curing age increased, the samples exhibited a color change to a lighter shade, accompanied by a reduction in bulk density, as shown in Figure 8. This reduction is associated with increased porosity due to water evaporation from the geopolymer matrix during the curing process. This observation is consistent with the findings of [21][22] which stated that water evaporation in geopolymer systems leads to increased porosity and a reduction in bulk density. In contrast, cement paste exhibited relatively minor and

stable weight changes over time, indicating a slower and more controlled hydration process (Figure 7).

Based on Figure 8, the smallest reduction in bulk density was observed in samples with a high activator modulus, whereas the largest reduction occurred in samples with a low modulus. This indicates that a higher activator modulus tends to produce a denser geopolymer matrix that is more stable against mass loss due to evaporation[21]

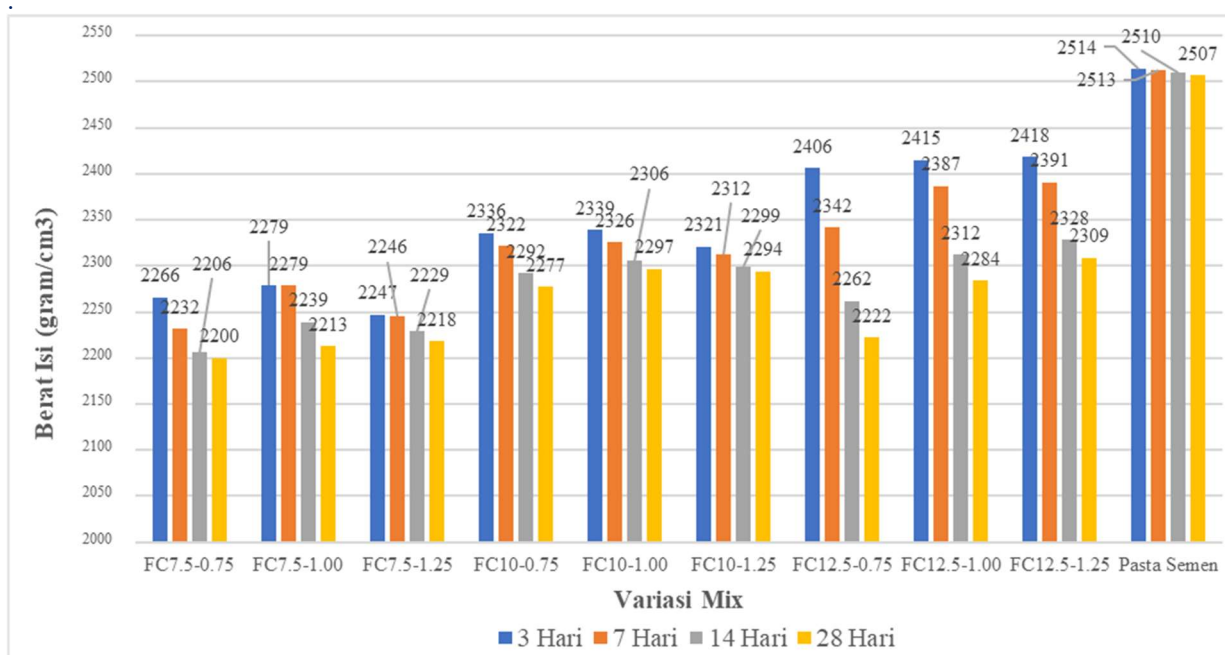


Figure 8. Compressive strength of all mix variations at ages 3, 7, 14, and 28 days

3.4. Relationship between Setting Time and Compressive Strength of Geopolymer Paste

Based on Figures 9 and Figures 10, an increase in activator modulus contributed to faster setting times, followed by an increase in compressive strength, particularly at early ages (≤ 3 days). This effect is primarily due to the high sodium silicate (Na_2SiO_3) content, which plays a key role in accelerating the polymerization reaction and the formation of a three-dimensional network structure in the geopolymer system [23][24]. However, this acceleration of the reaction is also influenced by other

factors, such as the presence of calcium hydroxide ($\text{Ca}(\text{OH})_2$), the type and concentration of the activator, and the thermal curing conditions. [25]. Increasing the Na_2O dosage from 7.5% to 10% resulted in a longer setting time while still achieving higher compressive strength, indicating an optimized geopolymerization reaction. Conversely, further increasing the dosage to 12.5% extended the setting time without providing a significant increase in compressive strength, suggesting an excess of alkali that reduces the efficiency of matrix formation. [26][27].

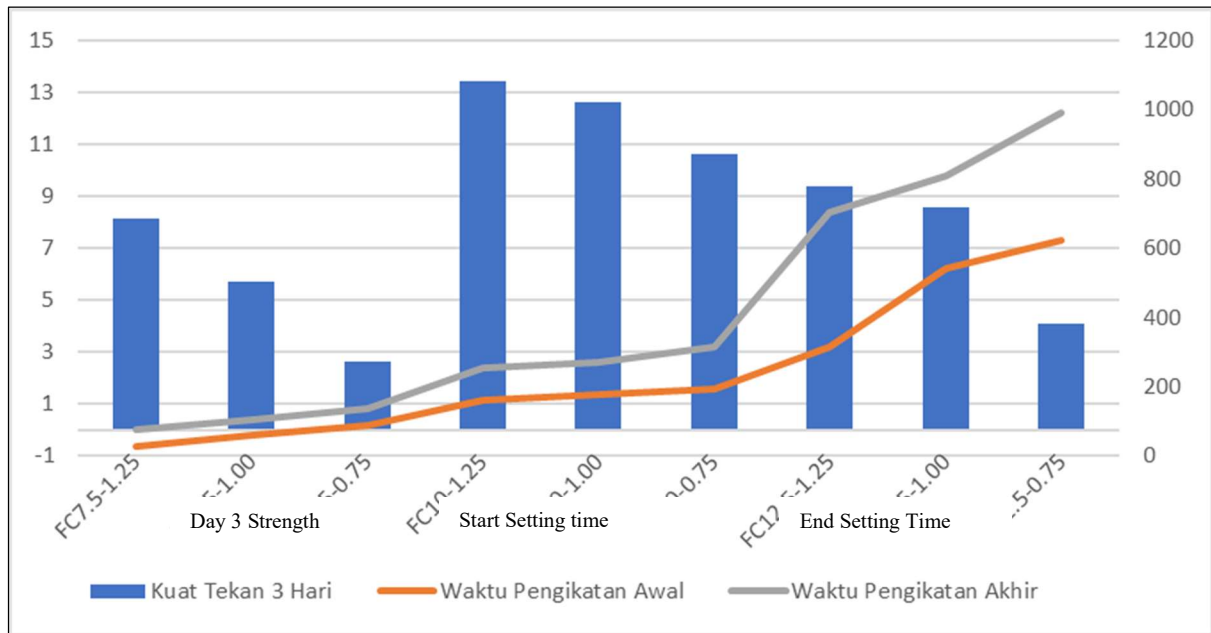


Figure 9. Relationship between setting time and compressive strength of geopolymer paste on day 3



Figure 10. Relationship between setting time and compressive strength of geopolymer paste on day 28

4. Conclusion

Based on the results of this study, it can be concluded that geopolymers paste compositions with Na_2O dosages of 7.5% to 10% and activator moduli between 0.75 and 1.25 exhibited the most optimal performance for curing at room temperature ($\pm 30^\circ\text{C}$). Compositions with a Na_2O dosage of 12.5% are not recommended, as they resulted in final setting times exceeding the ideal limits according to SNI 15-2049-2004 (360–480 minutes). The best initial setting times were achieved by compositions with Na_2O 7.5% and moduli of 0.75 and 1.00, at 88.67 and 60.94 minutes, respectively, remaining within the standard Portland cement range (60–120 minutes). The highest compressive strength was obtained in samples with Na_2O 10% and modulus 1.25 at 29.76 MPa, whereas increasing the dosage to 12.5% decreased compressive strength and caused efflorescence due to unreacted excess activator.

In general, increasing the activator modulus accelerated the setting time due to the high sodium silicate content, which promotes the polymerization reaction. Conversely, increasing the Na_2O dosage slowed the setting time because of the higher activator content, making the system more fluid and slower to harden. Furthermore, geopolymer paste exhibited a lower bulk density compared to cement paste, with a decreasing trend over time due to increased porosity and water evaporation from the matrix.

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