Long-term Mechanical Properties and Microstructure of Concrete Utilizing Self-Cementing Fly Ash as A Sole Binder Material

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Abstract: Self-cementing fly ash, containing calcium oxide (CaO) of about 20%, has successfully become a sole binder material in concrete through hydration. A very low water-to-fly ash ratio, e.g., below 0.20, is the main key to achieving the high compressive strength of concrete. This study explores the strength evolution, long-term compressive strength, and mechanical properties of concrete that utilize self-cementing fly ash as a sole binder material. Remarkably, the long-term compressive strength continues to develop over a year, reaching nearly 50 MPa at 365 days, a 34% increase from the 28-day strength. While the other mechanical properties are slightly lower than predictions from empirical formulas for Portland cement concrete, promising results are observed. Scanning Electron Microscope (SEM) images highlight surface-restricted hydration products in self-cementing fly ash, rather than full dissolution of fly ash particles.

Keywords: Fly ash; self-cementing; CaO; very low water-to-fly ash ratio; long-term; SEM.

Introduction

Fly ash that contains a higher amount of calcium oxide (CaO) will have cementitious properties in addition to its pozzolanic properties [1]. According to ASTM C618, Class C fly ash is expected to exhibit cementitious properties when the CaO content in the fly ash exceeds 18%. Additionally, the Canadian Standard outlined in CSA A-3001 categorizes fly ash based on its CaO content. Specifically, Type F designates fly ash with a CaO content below 8%, Type CI for fly ash with intermediate CaO content (ranging from 8% to 20%), and Type CH for fly ash with high CaO content (above 20%) [2].

The self-cementing property of fly ash has been extensively studied and applied for decades, particularly in terms of soil stabilization [3–7]. Conversely, research and utilization of self-cementing fly ash in concrete, especially as a sole binder material, remain quite limited. There are only a few studies and reports available on the utilization of self-cementing fly ash in concrete [8–12]. Moreover, research has demonstrated that a high calcium oxide content in fly ash does not necessarily ensure the expression of selfcementing properties. Some fly ashes with high CaO content exhibit notably low compressive strength when utilized as a sole binder material in concrete [11]. Our previous study has successfully demonstrated the self-cementing behavior of fly ash obtained from PLTU Paiton in Indonesia [13]. A very low water-tofly ash ratio has become the main key to achieving high compressive strength. The lower amount of water content in this concrete, compared with ordinary Portland cement concrete, is achievable because of the spherical shape of the fly ash and its particle size distribution. A 28-day compressive strength of up to 37 MPa was reached from a mixture with a waterto-fly ash ratio (w/fa) of 0.143 (0.600 in volume).

The correlation between concrete compressive strength and its other mechanical strengths, such as splitting tensile strength and flexural strength, is a crucial aspect in the design and analysis of concrete structures. It has been observed, in Portland cement concrete, that there exists a strong positive relationship between compressive strength and those mechanical properties [14]. However, different binders used in concrete can result in different behavior in terms of mechanical properties.

This study focuses on examining the long-term compressive strength and strength development of concrete utilizing self-cementing fly ash as the sole binder material. Furthermore, an analysis of various mechanical properties will be conducted. The investigation involves conducting a series of tests (i.e., compressive test, splitting tensile test, flexural test, modulus of elasticity, and rebar pull-out test) and experiments to assess the compressive strength over an extended period and to understand the overall mechanical behavior of the concrete mixture.

By evaluating the performance of concrete with selfcementing fly ash, this research aims to provide

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insights into its suitability as a standalone binder in construction applications, especially as reinforced concrete material. The concrete microstructure is also observed using Scanning Electron Microscope (SEM) to better understand the reaction product of selfcementing fly ash hydration. The findings of this study will significantly enhance our comprehension of both the potential benefits and limitations of using self-cementing fly ash as a substitute for traditional binders in concrete production.

Experimental Methods

Materials and Mixtures Composition

Fly ash used in this experiment was obtained from the steam power plant in Paiton, East Java Province, Indonesia. The combustion process used in this power plant is Pulverized Coal Combustion (PCC). Fly ash was captured by an electrostatic precipitator (ESP) and directly transferred into silos in dry conditions. X-Ray Fluorescence (XRF) analysis was done to identify the major chemical composition of the fly ash obtained as can be seen in Table 1. According to ASTM C618 [1], this fly ash is classified as class C fly ash because the calcium oxide (CaO) content is above 18%. While Canadian Standard CSA A-3001 [2] classifies this fly ash into CH type, as the CaO content is above 20%. However, the amount of CaO in this fly ash is much lower than the self-cementing fly ash used in most existing literature, where the amount of CaO ranged between 25 - 30% [8]–[12]. The specific gravity of fly ash used in this research was 2.80 and the pH value was as high as 12.6. The high value of pH confirmed the high content of calcium oxide since the alkalinity of fly ash is strongly affected by the calcium compound [15].

Table 1. Chemical Composition of Fly Ash Used (%)

SiO_2	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO_3	LoI
29.50	15.02	17.71	22.34	8.37	1.90	0.36

The concrete mixture used local silica sand and crushed stone as aggregates. The properties of aggregates in saturated-surface-dry (SSD) conditions can be seen in Table 2. Tap water, with a pH value of \pm 7, was used and added to the mixture in stages according to the mix design. Some amount of superplasticizers, with polycarboxylate-ether (PCE) based, were used to enhance the workability of the concrete mixture to achieve the targeted slump (10 ±2 cm). Utilizing superplasticizers is also a key factor in minimizing the water-to-fly ash ratio (w/fa) and reducing the water content in concrete mixtures. Therefore, a lower amount of voids (due to reduced water content) is expected to increase the concrete compactness, resulting in higher compressive strength.

Tabl	e 2. /	Aggregates	Properties
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	Eine e	Commo
	rine	Coarse
	Aggregate	Aggregate
Density (kg/m ³)	1604	1459
Specific Gravity (unitless)	2.66	2.61
Absorption (%)	0.44	3.32
Fineness Modulus	2.65	7.30

Five concrete mix compositions with five different water-to-fly ash ratios, as shown in Table 3, were designed to analyze and establish the correlation between concrete compressive strength up to one year of age and the water-to-fly ash ratio in a mixture. In all mix designs, the volume of the paste remains constant at 30%, emphasizing that the strength of the concrete is solely reliant on the quality of the selfcementing fly ash paste. The term w/fa (by volume) is introduced due to the variation in specific gravity between fly ashes and Portland cement. Considering the difference in specific gravity between fly ashes and Portland cement, it is important to acknowledge that for a given w/fa (by mass) value, the mass or weight of binder material in a 1 m³ mixture would differ depending on the specific gravity of the binder material used. By providing information in terms of w/fa (by volume), the comparison of different binders with varying specific gravity can still be made since they have the same volume. For the mechanical characterization of concrete, three concrete mixtures were prepared with different water-to-fly ash ratios as shown in Table 4.

Table 3. Concrete Mix Designs for Compressive Test

w/f	à	Flu Ach	Waton	Fine	Coarse
by volume	by mass	(kg)	(kg)	Agg. (kg)	Agg. (kg)
0.80	0.286	466.7	133.3	745.1	1155.0
0.70	0.250	494.1	123.5	745.1	1155.0
0.60	0.214	525.0	112.5	745.1	1155.0
0.50	0.179	560.0	100.0	745.1	1155.0
0.40	0.143	600.0	85.7	745.1	1155.0

 Table 4. Concrete Mix Designs for Mechanical Characterization

w/f	a	Elv Ach	Watan	Fine	Coarse
by volume	by mass	(kg)	(kg)	Agg. (kg)	Agg. (kg)
0.50	0.179	560.0	100.0	745.1	1155.0
0.45	0.161	579.3	93.1	745.1	1155.0
0.40	0.143	600.0	85.7	745.1	1155.0

Testing Procedures

Concrete specimens were dry-cured and immediately wrapped using plastic wrap to prevent evaporation right after the concrete hardened and the mold release process was completed. These wrapped specimens were stored at room temperature (approximately 28 °C) until the testing day. Bath curing was avoided because it could lead to the leaching of calcium oxide from the fly ash into the water, compromising its strength. The compression tests were conducted on a diameter of 10 cm by height of 20 cm concrete cylinders following ASTM C39 guidelines. Before the compressive test, sulfur capping was applied to achieve a smooth concrete top surface. The compressive strength of the concrete was calculated as the average strength of three cylinders.

In addition to compressive strength, other concrete mechanical properties were also tested, including tensile strength, flexural strength, modulus of elasticity, and bond behavior between concrete and steel reinforcement. The tensile behavior of concrete was assessed through two indirect tensile tests: the splitting tensile strength test according to ASTM C496 and the flexural strength test according to ASTM C78. The concrete modulus of elasticity was determined using the stress-strain test based on ASTM C469 with some adjustments. The bond strength between concrete and reinforcing steel, also known as rebar bond strength, is widely acknowledged as a crucial factor in ensuring the satisfactory performance of reinforced concrete structures. Deformed bars are the preferred choice due to their ability to enhance bond strength through the presence of ribs along the surface of the reinforcing steel. In ribbed reinforcing steel, the bond strength mainly arises from three key mechanisms:

(1) the chemical adhesion between the binding material in concrete and the reinforcing steel; (2) the frictional resistance that occurs between the reinforcing steel and the surrounding concrete; and (3) the bearing resistance of the ribbed surface of the reinforcing steel against the concrete surface surrounding the steel [16]. The bond strength of concrete towards the reinforcement bar was tested using pull-out specimens with the help of steel jigs as shown in Figure 1.

Results and Discussions

Strength Development

The results of the average compressive strength tests for the five different water-to-cementitious ratios are presented in Table 5. The highest concrete compressive strength is 37.05 MPa at 28 days, obtained from the lowest w/fa (by volume) value of 0.40 (equivalent to w/fa (by mass) 0.143). This compressive strength continues to develop and reaches 41.89 MPa at 56 days and further increases to 49.78 MPa at 365 days. In general, concrete with a w/fa (by volume) of 0.60 or lower demonstrates the ability to achieve compressive strengths exceeding 20 MPa at 28 days. Conversely, concrete mixtures with w/fa (by volume) values of 0.70 and 0.80 exhibit compressive strengths of less than 15 MPa at 28 days, and even extending to a 365-day age, the compressive strength of these concrete mixtures remains below the 20 MPa.





Figure 1. Steel Jig Sketch and Specimen Set Up for Pull-Out Test

Table 5. Concrete Compressive Strength

w/f	la			Concret	e Compress	sive Strengt	h (MPa)		
by volume	by mass	3 days	7 days	14 days	28 days	56 days	90 days	180 days	365 days
0.80	0.286	3.69	5.41	5.92	7.77	8.59	9.23	9.93	10.12
0.70	0.250	7.96	10.12	11.78	14.07	15.41	16.30	17.06	17.38
0.60	0.214	13.43	16.17	19.93	21.96	22.98	23.43	24.26	25.15
0.50	0.179	19.74	21.39	25.15	28.84	33.74	35.97	37.43	38.64
0.40	0.143	29.92	33.10	34.50	37.05	41.89	46.22	48.51	49.78



Figure 2. Compressive Strength Development (as a Percentage of 28-day Strength)

In concrete mixtures with w/fa (by volume) of 0.60, 0.70, and 0.80, a significant development in compressive strength was observed up to 28 days of age. Meanwhile, for mixtures with w/fa (by volume) values of 0.50 and 0.60, the increase in compressive strength was still notable, extending up to 365 days of age. Across all variations of water-to-fly ash ratios, the concrete's compressive strength continued to improve for up to 365 days. The most substantial strength development occurred in the concrete with the lowest w/fa value, approximately 134% higher than its 28-day compressive strength.

The strength development of the concrete utilizing self-cementing fly ash also suggests the presence of hydration reactions over time. This behavior is similar to the hydration process in Portland cement, where concrete compressive strength increases with the progress of hydration, commonly known as the degree of hydration or rate of hydration [14]. However, further research is required to conduct a thorough analysis to ascertain if the hydration process and product in self-cementing fly ash are similar to those in Portland cement.

Mechanical Properties

Apart from compressive strength, other mechanical properties, such as tensile strength, flexural strength, modulus of elasticity, and bond strength between concrete and steel reinforcement, will be discussed in this section. These mechanical properties were tested at an age of more than one year to assess the longterm strength of the concrete utilizing self-cementing fly ash as a sole binder material. Each mechanical properties test was accompanied by a compressive test to generate the correlation between compressive strength to other mechanical strengths.

Tensile strength is crucial in understanding concrete's resistance to cracking and its ability to withstand tensile stresses. Flexural strength, on the other hand, reflects concrete's behavior under bending or flexure, which is essential for evaluating its performance in beams and other structural elements subject to bending loads. The modulus of elasticity provides insights into concrete's stiffness and deformation characteristics, influencing its behavior under various loads. Lastly, the bond strength between concrete and steel reinforcement is a critical factor in reinforced concrete structures, as it ensures the effectiveness of the bond between the concrete and the reinforcing steel.

Splitting Tensile Strength

Concrete splitting tensile strength (ft), calculated from the splitting tensile test, for three mixtures with different w/fa, is shown in Figure 3 by the continuous line with the diamond mark. While the column chart shows the concrete compressive strength (fc'). The results show that lower w/fa results in higher compressive strength as well as higher splitting tensile strength, following the correlation between splitting tensile strength and compressive strength as in Portland cement concrete. Tensile strength of concrete as well as its compressive strength mainly determined by the quality of the paste or the binder. Therefore, both mechanical properties, i.e., tensile and compressive strengths, have a positive correlation.

The relationship between concrete tensile and compressive strengths is further explored through the empirical formula proposed in ACI 318 [17] and Oluokun [18]. It is worth noting that the prediction formula was generated from Portland cement concrete tests. Then the predicted values are compared with the experimental results that use self-cementing fly ash as a sole binder material. The comparison between concrete experimental splitting tensile strength and the predicted splitting tensile strength, from two formulas established for Portland cement concrete, can be seen in Figure 3.



Figure 3. Comparison of Concrete Compressive Strength and Splitting Tensile Strength

It can be concluded that the tensile strength of the concrete utilizing self-cementing fly ash is slightly lower than the Portland cement concrete. The lower tensile strength in self-cementing fly ash paste could be due to the hydration product that only occurred at the surface as a result of the lower reactivity of the self-cementing fly ash compared to Portland cement. Splitting tensile strength of the concrete utilizing self-cementing fly ash as a sole binder material is about 30% and 10% lower than the predicted value calculated from the Portland cement concrete proposed formula by ACI 318 [17] and Oluokun [18] consecutively.

Flexural Strength

Flexural strength (fr) is the theoretical maximum tensile stress that occurs at the bottom fiber of a beam when subjected to bending loads from above. It is also commonly referred to as the modulus of rupture (R). The results of the flexural strength tests for three different mix designs with varying water-to-cementitious ratios in Figure 4 show that lower w/fa ratios are associated with higher values of modulus of rupture or flexural strength. The flexural strength test yielded higher tensile strength values compared to the splitting tensile strength test. This difference can be attributed to the distinct shape and testing methods of the specimens, which also impact the tensile strength results for conventional concrete[14].

ACI 318 [17] also proposed an empirical formula to express the relationship between flexural strength and compressive strength of Portland cement concrete. Dash-dot line in Figure 4 represents the value calculated from the empirical formula. While the continuous line with a square mark represents the value of concrete flexural strength utilizing selfcementing fly ash as a sole binder material in this experiment. The flexural strength tests yielded results that were quite close to the predicted values calculated using equations for conventional concrete and, in general, even higher (except for the mix with w/fa 0.50). The differences observed were approximately 5% lower for the self-cementing fly ash concrete mix with w/fa (by volume) of 0.50, but about 6 - 15%higher for w/fa (by volume) of 0.40 and 0.45.



Figure 4. Comparison of Concrete Compressive Strength and Flexural Strength

As the water-to-cementitious ratio in the mix increased, the ratio between predicted and tested values also increased, even reaching a point where the predicted value was higher than the tested value for w/fa (by volume) of 0.50. Similar findings were observed in a study conducted by Cross et al. (2005b), where the w/fa (by mass) used was 0.23 or w/fa (by volume) was 0.63, resulting in a prediction-to-test ratio of 1.203. However, further investigation is needed to understand the reasons behind the increasing ratio between predicted and tested flexural strength values as the water-to-cementitious ratio increases.

Modulus of Elasticity

The modulus of elasticity can also be defined as the tangent modulus since its value is obtained from the stress-strain relationship within a specific range or interval. In this study, the range for determining the tangent value was slightly modified from the ASTM C469 [19], a standard used in the testing procedure. The modification was applied to the lower limit of the range because the initial part of the stress-strain curve did not exhibit a clear linear relationship. This observation might be attributed to adjustments in the testing equipment and capping of the specimens during the early stages of loading, where the applied stress was relatively small. Despite the modification in calculating the modulus of elasticity, it still adhered to the fundamental concept of the tangent modulus or the slope of the linear tangent line formed by the stress-strain relationship under elastic conditions. The stress-strain relationship of the tested specimens under compression can be observed in Figure 5.



Figure 5. Concrete Stress-Strain Relation

The relationship between stress and strain becomes linear when the stress reaches around 10 MPa. The final range for calculating the modulus remains consistent with the procedure recommended in ASTM C469 [19]. While there is a difference in the final range between ASTM C469 [19] (40% of ultimate compressive strength, fc') and ACI 318 [20] (45% of fc' ultimate), it does not significantly affect the modulus results. The testing of two cylindrical specimens yielded an average modulus of elasticity (Ec) of 23.5 GPa. This value is approximately 7% lower than the findings of the study conducted by Cross et al. [8]. This difference is likely due to the variation in ultimate compressive strength, where their study reported 31 MPa, while this experiment obtained 41 MPa. However, this finding contradicts the principle and prediction of the modulus of elasticity recommended in ACI 318 (2022), which is expressed as:

$$E_c = 4700(f_c')^{0.5} \tag{1}$$

Based on Equation 1, as the compressive strength of concrete increases, the modulus of elasticity will also increase. The modulus of elasticity obtained from the test results (23.5 GPa) indicates a value approximately 23% lower than the predicted value (30.4 GPa). To further investigate and justify the potential differences in the relationship between compressive strength and modulus of elasticity in Portland cement concrete and concrete utilizing self-cementing fly ash as a sole binder material, more extensive research with a larger number of specimens is necessary.

Ta	ble 6.	Resul	ts of l	Pull-0	Dut '	Γest

Rebar Bond Strength

The results of the pull-out tests are presented in Table 4, where it can be observed that some of the specimens experienced failure at the bond interface between the reinforcing steel and the concrete (indicated by the pulling or detachment of the reinforcing steel from the concrete cylinder). Other specimens exhibited steel failure above the surface of the concrete cylinder before detachment occurred. The values in the relative bond strength column were obtained by dividing the maximum measured tensile force by the total surface area of the embedded reinforcing steel, which was calculated as the embedded surface area of the cylinder curve face. In cases where the reinforcing steel fractured before detachment, the relative bond strength value represents the exact value at the point of steel fracture, indicating that the actual bond strength value is likely greater than the reported value (indicated by the symbol ">").

Based on the analysis of the conducted tests, it is evident that concrete utilizing self-cementing fly ash as the sole binder material demonstrates satisfactory bond behavior with reinforcing steel, with an average bond length of 16 - 19 times the diameter of the reinforcing steel. The bond strength between reinforcing steel and concrete of different grades has not yielded sufficiently conclusive results. Similar findings were observed in a study conducted by Cross et al. [8], where splitting failure occurred in all specimens with two variations of embedment length. Nevertheless, in this research, when considering the average values of relative bond strength, concrete with a higher grade (w/fa ratio of 0.40 by volume) exhibited higher bond strength. This justification is further supported by the failure mode data, where in the case of higher-grade concrete (w/fa ratio of 0.40 by volume), the majority of failures occurred at the steel reinforcement, despite having a relatively shorter average embedment length compared to lower-grade concrete. This can be understood as concrete compressive strength being a crucial factor influencing the bond strength of reinforcing steel according to ACI 408 [16].

Scanning Electron Microscope (SEM)

Observation of the microstructure was conducted on the fly ash material itself before its reaction with

w/fa (by volume)	Sample No.	Tensile Force (kN)	Anchorage Depth (mm)	Failure Mode	Relative Bond Strength (MPa)	Tensile Strength of Reinforcement (MPa)
	1	51.35	165.00	Reinforcement	> 9.91	653.81
0.40	2	48.82	167.00	Concrete Bond	9.31	-
	3	51.52	161.00	Reinforcement	> 10.19	655.97
0.45	1	48.43	165.00	Concrete Bond	9.34	-
	2	48.43	208.00	Concrete Bond	7.41	-
	3	51.18	187.00	Reinforcement	> 8.71	651.64

water to examine the particle morphology. At a magnification of 5000 times, Figure 7 confirms that fly ash, particularly that derived from the combustion of pulverized coal using the Pulverized Coal Combustion (PCC) system, exhibits a spherical physical shape with non-uniform gradation. This supports the analysis regarding the lower water requirement in fly ash paste, thereby recommending the utilization of very low water-to-fly ash ratios (less than 0.30) when employing fly ash as the sole binder material in concrete.



Figure 7. Fly Ash Morphology at 5000 Times Magnification

The particle size of fly ash can also be observed physically, revealing a range of sizes varying from the largest at approximately 10-15 μ m to smaller sizes below 1 μ m. This characteristic is advantageous in promoting the density of a material, such as concrete, as a denser structure with reduced void volume contributes to enhanced compressive strength. In addition to improved compressive strength, a wellattained density in concrete can enhance its durability against external conditions.

The SEM analysis was also conducted on samples obtained from concrete utilizing fly ash as the sole binder material. The visual depiction of the hydration products of fly ash at various magnifications can be seen in Figure 7. Generally, the hydration products of fly ash exhibit a dense structure while still retaining the original spherical shape of the particles. This differs from the hydration products of Portland cement, which typically display a completely different physical form distinct from the original cement particles. With the persistence of the fly ash particle shape, it can be inferred that the degree of hydration likely occurs primarily at the particle surface, without fully dissolving the fly ash particles.



Figure 6. Microstructures of Self-cementing Fly Ash Concrete at Four Different Magnifications

Through the examination of the hydration products of fly ash, it can be understood that these products significantly influence the compressive strength of the fly ash paste or concrete. However, since the hydration process in fly ash differs from that in Portland cement, as shown by the SEM, the compressive strength of fly ash paste is predominantly influenced by the particle compactness rather than the bonding strength between particles. This can be attributed to the fact that, in addition to the water-to-fly ash ratio, the strength of concrete is also determined by its density [14]. A well-attained particle density enhances particle interaction, allowing for improved force transfer without generating excessive tensile stresses. The lower splitting tensile strength observed, in comparison to Portland cement concrete, can also be explained by the SEM observations, which suggest a lower adhesion strength between fly ash pastes.

Conclusions

Concrete that utilizes self-cementing fly ash as a sole binder material demonstrated good performance, especially in terms of compressive strength as evidenced by the strength development up to 365 days. Based on the findings of this study, several key conclusions can be drawn as follows:

- 1. The successful utilization of self-cementing fly ash as a sole binder material in concrete relies on the presence of reactive calcium compounds in the fly ash and the use of a significantly low water-to-fly ash ratio in the mixture. When considering the use of alternative sources of fly ash, it is highly recommended to assess their self-cementing properties during the initial stages. In addition to the chemical composition, the morphology of fly ash also plays a crucial role in achieving a very low waterto-fly ash mixture. The spherical shape of fly ash particles contributes to a ball-bearing effect, enhancing concrete workability. Therefore, fly ash derived from a Pulverized Coal Combustion (PCC) burner is preferable.
- 2. The long-term mechanical properties, including compressive strength, of concrete utilizing selfcementing fly ash show a higher rate of development compared to ordinary Portland cement concrete. This observed strength development confirms the occurrence of hydration reactions in the self-cementing fly ash.
- 3. The other mechanical properties of concrete utilizing the self-cementing properties of fly ash as the sole binder also exhibit promising results, although they generally tend to be slightly lower than the predicted values calculated using equations developed for conventional concrete. This reduction in mechanical characteristics can be attributed to the diminished tensile capacity, likely resulting from hydration products that

predominantly form on the surface of fly ash particles, leading to weaker interparticle adhesion.

4. The hydration products of self-cementing fly ash indicate distinct characteristics compared to those of Portland cement. Scanning Electron Microscopy (SEM) analysis reveals the clear presence of initial fly ash particles in a spherical shape. Hydration processes in fly ash, with an approximate CaO content of 20%, are predominantly limited to the particle surface, resulting in weaker interparticle bonding when compared to Portland cement. Additionally, the compressive strength in fly ash paste is derived from the effective transfer of forces between densely packed fly ash particles, as confirmed by SEM observations of the compact particle arrangement.

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