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EVALUATION OF THE EFFECT OF FINENESS OF FLY ASH IN TYPE I AND TYPE II PORTLAND CEMENT BLENDED MORTARS

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Abstract. This study evaluates the effects of fineness-modified Class F fly ash (FA) on the performance of Type I and Type II Ordinary Portland Cement (OPC) mortars. FA was mechanically ground to achieve 90% (GFA1) and 98% (GFA2) passing through a 45-µm sieve, replacing OPC at 10%, 30%, and 50% by weight. Comprehensive testing included compressive strength, water demand, setting time, heat of hydration, acid resistance, and microstructural analysis. Results demonstrated that increasing FA fineness reduced water demand by up to 6.3% (Type I) and 5.9% (Type II) at 30% replacement, while enhancing long-term strength. Mortars with 10% GFA2 achieved 58.6 MPa at 56 days. Type II OPC blends exhibited superior acid resistance, with mass loss reductions of 43% (50% GFA2) compared to Type I, attributed to lower C₃A content and denser microstructures. Hydration heat decreased by 10.9% in Type II OPC, further reduced by 40% with 50% FA replacement. The microstructural analysis confirmed reduced ettringite formation and enhanced compactness in FA-modified mortars, correlating with improved durability. These findings highlight the viability of finely ground FA as a sustainable supplementary material, enabling high-volume FA utilization (up to 50%). The study provides critical insights for optimizing greener cement formulations, particularly in regions prioritizing Type II OPC for sulfate resistance and moderate heat applications, advancing Malaysia's net-zero carbon goals through reduced clinker reliance.

Keywords: fineness of fly ash, type I & II ordinary Portland cement, setting time, soundness, heat of hydration.

List of Abbreviations

FA - Fly Ash

OPC - Ordinary Portland Cement

XRD - X-ray Diffraction

XRF - X-ray Fluorescence

SEM – Scanning Electron Microscope

C-S-H - Calcium Silicate Hydrate

C₃A - Tricalcium Aluminate

C₃S – Tricalcium Silicate

FCaO – Free calcium oxide

cao Tree calciam oxia

LOI - Loss on Ignition

SAF – Total Oxides of Silica, Aluminate, and Ferrite

1. Introduction

The concrete industry's reliance on ordinary Portland cement (OPC) persists globally, but regional material practices demand localized solutions. In Malaysia, type II OPC

is prioritized for sulfate-resistant infrastructure under MS EN 197-1 standards (Department of Standards Malaysia, 2019); further, based on the Malaysian government's target of net-zero emissions by 2050, optimizing the usage of fly ash (FA) in cement blends is critical. While this study focuses on Malaysia's industrial context, the methodologies offer insights for tropical regions with similar material constraints.

Portland cement is made by heating raw materials in a kiln to produce clinker, which is then mixed with additives and ground into powder. The clinker consists mainly of CaO, SiO₂, Al₂O₃, and Fe₂O₃. Type I OPC is a widely used classification of cement that does not require special properties (American Society for Testing and Material [ASTM], 2017). The C₃A content distinguishes Type I OPC, with a higher C₃A content resulting in higher heat evolution. Lower C₃A cements are preferred in sulfate environments to prevent the formation of ettringite and thaumasite (Hossack & Thomas, 2015). Type II OPC is used when

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moderate sulfate resistance is required, with a lower C_3A limitation at 8.0% maximum and a maximum alumina content of 6.0% (ASTM, 2017). Special raw materials are required to produce type-II OPC. Its moderate heat of hydration makes it suitable for use in structures with considerable mass.

FA, a by-product of Malaysia's coal-fired power plants (Algahtani et al., 2021), enhances sustainability through its pozzolanic and filler effects (Golewski, 2022). The management of FA is a concern because of its large disposal area and potential environmental pollution. FA's physical and chemical features are important for its use and disposal (Mironyuk et al., 2019; Parra-Huertas et al., 2023). It can be used as a concrete cement substitute or a constituent in blended cement (Hsu et al., 2018). FA is used in construction because of its durability and environmental advantages. FA has a uniform particle size, sphericity, and smooth surface, which improve the workability of concrete and increase its strength. FA improves the durability of concrete by reducing the expansion caused by water and sodium sulfate and rapid chloride penetration (Pok et al., 2021). It can also replace cement to enhance corrosion and heat resistance (Bondar & Coakley, 2017; Hongbo et al., 2020). FA is categorized as Class C or Class F and typically contains silicon dioxide, aluminium oxide, iron oxides, and calcium oxides (ASTM, 2021). Generally, the effects of FA in cementitious materials are described by two mechanisms: the filler effect at early ages and the pozzolanic reaction of FA (Park & Choi, 2022). When combined with OPC, the pozzolanic reaction occurs, forming a calcium-silicate-hydrate gel and improving the durability of the mortar or concrete (Hemalatha et al., 2016; Hsu et al., 2018; Nawaz et al., 2016; Nedunuri et al., 2020). Previous studies have indicated that the C-S-H gel phase in cement paste can be observed after 30 min of curing. The subsequent reaction in the cement composite consisting of FA was observed through a scanning electron microscope (SEM) assessment after 7 days (Golewski, 2022) - the denser microstructure of concrete results from the pozzolanic reaction and the formation of additional C-S-H gels. A denser microstructure improves the mechanical behaviour of concrete on a macro scale, and it makes concrete stronger, more durable, and less permeable to water and other substances (Ghais Abadi, 2021; Schackow et al., 2020; Tkaczewska, 2014a). The pozzolanic properties promote enhanced long-term strength development and self-healing behaviour (Kan et al., 2019).

The water absorption of the cement composite in concrete decreased with the addition of FA and an increase in the FA ratio (Golewski, 2023a, 2023b). This characteristic is explained by the pozzolanic reactivity of fly ash (FA), which exhibits considerable advancement, leading to the substantial consumption of calcium hydroxide while simultaneously generating supplementary secondary C-S-H gels. This phenomenon contributes to a more densely packed and compact microstructure, thereby resulting in a reduction in water absorption (WA) through the process of immersion (Golewski, 2023a).

FA can replace cement at levels ranging from 1% to 100%, with a maximum permissible replacement of 55% under Malaysian cement standards (Department of Standards Malaysia, 2019). EN 197-1 classifies four categories of cement for using FA: Portland fly ash cement, Portland composite cement, pozzolanic cement, and composite cement (Kara De Maeijer et al., 2020; Department of Standards Malaysia, 2019). Studies on high-volume fly ash (HVFA) have focused on concrete sections, with the addition of geopolymers enabling replacement ratios of up to 100% (Alqahtani et al., 2021; Hongbo et al., 2020; Liu et al., 2020). One study showed that adding 5% limestone powder and FA improved strength development. The effect of binary additives is influenced by the physical and chemical characteristics of the individual additives (Kim et al., 2015; Nedunuri et al., 2020; Sun et al., 2013).

Despite all these benefits, the utilization of FA has not reached the desired levels because of issues with consistency in quality, particle size, and slower early hydration activity (Bondar & Coakley, 2017; Coppola et al., 2018; Cui et al., 2022; Hongbo et al., 2020; Krishnaraj & Ravichandran, 2019; Liu et al., 2020). Various modifications, such as mechanical activation via fineness modification, have been studied to improve the use of FA in cement and concrete manufacturing (Bondar & Coakley, 2017; Cui et al., 2022; Ghais Abadi, 2021; Krishnaraj & Ravichandran, 2019; Mezhov et al., 2019).

Mechanical activation through fineness modification is common for studying and applying FA replacements. This method can be divided into two techniques: separation and mill grinding (Bondar & Coakley, 2017; Kan et al., 2019; Kara De Maeijer et al., 2020; Liu et al., 2020). The fineness of FA affects the properties of cement-based materials, such as their strength and durability, making it an important factor in the cement industry (Sun et al., 2021). In blended cement production, FA is introduced via blending or inter-grinding. Previous research has shown that finer fractions of FA increase compressive strength. The importance of grading ash particle size for compressive strength gain has been highlighted (Bondar & Coakley, 2017; Golewski, 2022; Hsu et al., 2019; Hu, 2014; Tkaczewska, 2014a; Wang et al., 2023). The influence of the surface area (Blaine value) on the compressive strength was also investigated, as a finer portion of FA accelerated both OPC hydration and the pozzolanic reactions of FA (Park & Choi, 2022). One study showed an optimum grinding time of 120 min for producing ground fly ash with an improved particle size distribution (PSD) (Tkaczewska, 2014b).

A previous study used an air classifier to sieve and separate FA portions finer than 75 and 45 microns. The separation resulted in three portions with different ash contents. Mortars with 40% FA samples required less water than Portland cement mortar. Water demand was higher for FA, which was less than 45 microns, due to the increased surface area. The compressive strengths of mortars made with FA less than 75-micron and 45-micron increased by 35% and 74%, respectively. Mortars made with

different water contents showed varying 3-day strengths. Fineness was measured for all portions, and the FA less than 45-micron had similar fineness to the medium-separated portion. FA-graded less than 45-micron ash produced a higher compressive strength, emphasizing the importance of ash particle size grading (Bondar & Coakley, 2017). Another similar study suggested that by controlling the fineness of fly ash, its performance in cement and concrete applications can be significantly improved. This study revealed that finer fractions of fly ash (< 20 µm) exhibited significantly higher reactivity than coarser fractions (Wang et al., 2023). Studies have utilized wet grinding of FA (Liu et al., 2020). Fineness modification of FA allows for higher percentages with better mortar or concrete performance compared with RFA. Many studies have demonstrated the benefits of this modification (Kara De Maeijer et al., 2020; Lin, 2020). An increase in fineness contributes to better workability owing to the increase in finer spherical particles, which increases the lubricant effect in the mix (Lin, 2020; Moghaddam et al., 2019).

The Malaysian government has committed to environmental conservation. With a focus on reducing CO2 emissions, Malaysia has set a target to achieve a "net zero carbon" status by 2050. Attention is now directed towards all significant stakeholders. Cement, one of the largest contributors to CO₂ emissions, is under scrutiny. The substitution of conventional OPC with FA directly reduces CO₂ emissions. However, this substitution must be implemented while maintaining the desired performance of cement mortar and concrete (Golewski, 2022). Fineness modification is an improved and practical method, particularly for the cement industry, for utilizing and increasing the FA ratio as a cementitious material replacement. Durability and heat evolution are critical parameters of cement that determine its performance and that of concrete in the construction industry.

Most studies on FA replacement have primarily focused on Type I OPC, presumably because of its widespread availability as a common Portland cement. In contrast, Type II OPC is less commonly available in many regions, resulting in limited research on its behaviour when replaced with FA. This gap underscores the need for further studies to understand the characteristics of FA replacement in Type II OPC, particularly in Southeast Asian regions, particularly Malaysia, where this type of cement is available. Consequently, the use of Type II OPC, which is classified as having a moderate heat of hydration and moderate sulfate resistance, was considered for FA replacement in this study. This study aims to evaluate the effect of FA replacement, specifically for Type II OPC, with comparisons made using Type I OPC. A comparison of the FA ratio of different fineness between Type I and Type II OPC was also evaluated. This research will assist the industry in re-examining the methodology to optimize FA usage, particularly in green cement manufacturing. It also aims to bridge the gap between lab-scale studies and real-world adoption (e.g., 30% FA replacement meets Class 42.5N strength, reducing clinker use without performance loss). The developing nations in Asia and Southeast Asia, including Malaysia, must explore all available avenues for a greater push towards greener and more durable construction materials. Hence, this study is expected to benefit the Asian construction industry by providing an alternative approach to this agenda.

2. Materials and methods

This study used two types of OPC: Type I OPC (identified as OPC I) and Type II OPC (identified as OPC II). Both cement samples were obtained from Negeri Sembilan Cement Industries (Malaysia). OPC I is classified as CEM I 52.5N under MS EN 197-1 (Department of Standards Malaysia, 2019) and Type I OPC according to ASTM C 150. OPC II is classified according to CEM I 52.5N under MS EN 197-1 (Department of Standards Malaysia, 2019) and Type II OPC according to ASTM C 150 (ASTM, 2017). Class-F FA (FA) was obtained from the Jimah Energy Ventures Power Plant, Negeri Sembilan, Malaysia, and is classified as class F under ASTM C 618. The fineness of the RFA was 3096 cm²/g, and the residue retained for the RFA on a 45-micron sieve was 27.3%. The block diagram in Figure 1 shows the overall experimental procedure. It highlights the steps involved in material preparation, characterization, mix design, and various tests performed to assess the performance of the FA-modified cement. The compositions and properties of Type I OPC, Type II OPC, and FA are given in Table 1 (chemical properties) and Table 2 (physical properties). Type II OPC has a specific requirement for C₃A, which needs to be less than 8%, as per ASTM standards (ASTM, 2017). Thus, it significantly differentiates Type II OPC from Type I OPC. Raw FA (RFA) was ground into two fineness targets with different durations in a laboratory ball mill. Ground FA 1 (GFA1) had particle sizes 98% less than 45 µm, and Ground FA 2 (GFA2) had particles 90% less than 45 µm. These two sizes of fineness were chosen because they are common fineness targets for FA-blended cement and align with achievable cement-plant processes.

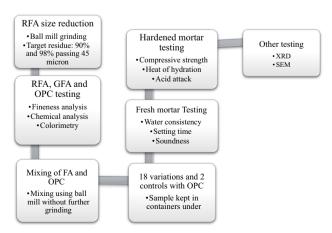


Figure 1. Block diagram for experimental studies

Table 1. Chemical properties of materials

Oxides	Cementitious materials' oxide composition (%)						
Oxides	OPC I	OPC II	RFA	GFA1	GFA2		
SiO ₂	19.81	20.58	62.61	62.10	61.87		
Al ₂ O ₃	5.59	4.92	15.25	15.17	15.04		
Fe ₂ O ₃	3.45	3.63	8.87	8.81	9.12		
CaO	63.55	63.99	7.80	7.94	8.60		
MgO	1.00	0.48	1.60	1.57	1.61		
Na ₂ O	0.11	0.12	0.35	0.33	0.35		
K ₂ O	0.94	0.56	0.95	0.97	0.96		
SO ₃	3.16	2.81	0.56	0.58	0.54		
FCaO	1.24	2.14	_	-	-		
C ₃ S	56.68	57.8	-	_	-		
C ₃ A	8.95	6.90	_	_	-		
SAF	_	_	86.73	86.08	86.03		
LOI	3.2	2.31	3.18	3.26	3.12		

Table 2. Physical properties of materials

Physical properties	OPC I	OPC II	RFA	GFA1	GFA2
Residue retained on 45 µm sieve, %	5.5	7.9	27.3	8.4	2.0
Specific surface area, cm ² /g Specific gravity, g/cm ³	3885 3.14	3500 3.15	3096 2.21	4801 2.28	5381 2.37

Cement plants typically control cement fineness by measuring the percentage retained on a 45-micron sieve and the specific surface area. The 45-micron sieve method was selected for its industrial practicality.

The fineness of the FA and cement was tested using two methods. The first method, known as the Blaine method, measures the fineness by observing the time it takes for a fixed amount of air to flow through a compacted cement bed using a Blaine permeability apparatus. The second method involved wet sieving the sample through

a 45 μ m sieve and reporting the residue retained as a percentage. Additionally, the specific gravity of the samples was tested using a pycnometer according to the MS EN 196-6 (Department of Standards Malaysia, 2021a). Particle size analysis was conducted using a Cilas analyzer to evaluate the materials comprehensively.

Table 2 shows the fineness and specific gravity results for RFA, GFA 1, and GFA 2. The results show that the FA surface area and specific gravity increase with the decreasing amount of FA retained in the 45-micron residue sieve. This is because of the extended grinding time in a laboratory ball mill (Rajak et al., 2017), as reported by researchers (Krishnaraj & Ravichandran, 2019; Moghaddam et al., 2019; Patil & Anandhan, 2015).

The particle size evaluation shown in Figure 2 shows that OPC I and OPC II have relatively similar particle size distribution curves, with OPC II having slightly larger particle sizes. RFA had a broader PSD curve, indicating a wider range of particle sizes. GFA1 and GFA2 show a narrower PSD curve than RFA, indicating more uniform particle sizes and reflecting the grinding process.

2.1. Material mixing

For the cementitious material preparation, the RFA and GFA with two different finenesses (GFA1 and GFA2) were used to replace both OPC types I and II at 10%, 30%, and 50%. A total of 20 mixes with six variations were prepared, as listed in Table 3, along with the two control specimens. The mix proportions of OPC and FA composites are listed in Table 3.

2.2. Colorimetric analysis

In this study, the reflected colour of RFA and GFA was evaluated using a HunterLab ColorFlex EZ spectrophotometer. It is measured based on three parameters: L, a, and b. The details of these parameters are presented in Table 4.

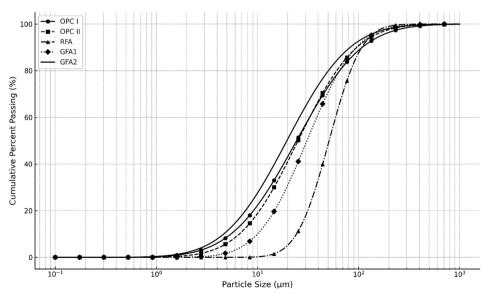


Figure 2. Particle size analysis of OPC and GFA

Table 3. Sample variations and identifications

Mix Designation	Description	Target Fineness (% < 45µm)
S1	Type I OPC + 10% RFA	80
S2	Type I OPC + 30% RFA	80
S3	Type I OPC + 50% RFA	80
S4	Type I OPC + 10% GFA1	90
S5	Type I OPC + 30% GFA1	90
S6	Type I OPC + 50% GFA1	90
S7	Type I OPC + 10% GFA2	98
S8	Type I OPC + 30% GFA2	98
S9	Type I OPC + 50% GFA2	98
S10	Type II OPC + 10% RFA	80
S11	Type II OPC + 30% RFA	80
S12	Type II OPC + 50% RFA	80
S13	Type II OPC + 10% GFA1	90
S14	Type II OPC + 30% GFA1	90
S15	Type II OPC + 50% GFA1	90
S16	Type II OPC + 10% GFA2	98
S17	Type II OPC + 30% GFA2	98
S18	Type II OPC + 50% GFA2	98
OPC I	Type I OPC	-
OPC II	Type II OPC	-

Table 4. Colorimetry index

L	0 (dark)	100 (bright)
a	Negative (green)	Positive (red)
b	Negative (blue)	Positive (yellow)

2.3. Evaluation of cement paste and mortar

Tests on the consistency and setting time of the cement and blended cement mixes were conducted using the method outlined in MS EN 196-3 (Department of Standards Malaysia, 2022). For the consistency and setting time tests, 500 g of cement, 250 g of water (maintaining a water-cement ratio of 0.5), and FA replacing cement at 10%, 30%, and 50% by weight were used. The consistency of

the OPC and FA blended cement mixes was determined using the Vicat apparatus plunger, and the initial and final setting times of the cement paste were determined thereafter.

2.4. Soundness or expansion

The expansion or soundness test was performed according to the MS EN 196-3 (Department of Standards Malaysia, 2022). Part of the cement paste prepared in the standard consistency testing in part 2.3 was used for this test. The paste was filled in a Le Chatelier mould, covered with glass, and cured for 24 h in a humidity cabinet at 20 °C temperature and a relative humidity of more than 90%. The hardened paste, along with the mould, was heated to its boiling point in a water bath, and subsequently, the distance expansion was measured.

2.5. Compressive strength test

The compressive strength test of the specimens was conducted on mortar prism specimens according to the MS EN 196-1 (Department of Standards Malaysia, 2021b). For each mixture, moulds consisting of three horizontal compartments for three prismatic $40 \times 40 \times 160$ mm specimens were prepared using 450 g of cement, 1350 g of sand (sand-to-cement ratio of 3:1), 225 g of water (water-cement ratio of 0.5), and FA replacing cement at 10%, 30%, and 50% by weight. The prisms were cured at 20 °C and >90% relative humidity for 24 h and tested after 1, 2, 7, 14, 28, and 56 days. The three-point loading method was used to break the prisms, and the compressive strength of the broken halves was measured. Figure 3 shows the sample preparation and compressive strength testing of the mortar.

2.6. Heat of hydration

The heat of evolution of the cement and the mixed paste was determined using the ToniCAL Trio, a computer-controlled isotherm heat flow calorimeter (differential scanning calorimeter), to continuously determine the total hydration heat of binding agents, particularly cement.



Figure 3. Sample preparation and testing method for compressive strength

The cement paste was prepared with a water-cement ratio of 0.5 and FA, replacing cement at 10%, 30%, and 50% by weight. With online computer operation, the device allows direct determination of the rate of heat development (J/g) depending on time. This test measured the total heat evolution for up to 7 or 168 h.

2.7. Acid resistance test

The acid resistance test was performed using 50mm mortar cube specimens prepared using 450 g of cement, 1350 g of sand (sand-to-cement ratio of 3:1), 225 g of water (water-cement ratio of 0.5), and GFA2 replacing cement at 10%, 30%, and 50% by weight. The cubes were cured for 28 days and then immersed in 0.31 mol/L HCl solution for 4 weeks. After obtaining the initial weight of the specimens, they were examined for the next 28 days for changes in their visual appearance and mass (Kwasny et al., 2018). The mass loss of the immersed cubes was recorded and assessed.

2.8. X-ray Diffraction (XRD)

The mineral composition of the blended cement mortar paste was determined using Panalytical Xpert Powder XRD. For this investigation, mortar cubes of the mixes with 30% FA and reference OPC samples were evaluated according to the sample identification of Table 5. The portlandite, Ca(OH)₂ content of the blended cement paste powder samples was measured for the cement mortar prism samples (from Section 2.5) at 28 days (Rao & Kumar, 2021; Snellings et al., 2014).

2.9. Scanning electron microscopy (SEM)

For the SEM investigation, mixes with 30% FA and reference OPC samples were evaluated according to the sample identification in Table 5. Cement mortar prism samples (from Section 2.5) were analyzed at 28 days of age. Before SEM analysis, the samples were oven-dried at 105 \pm 5 °C for 24 h. SEM imaging was conducted using a high-performance Phenom ProX desktop SEM with an accelerating voltage of 15 kV and a resolution of less than 8 nm, providing a high depth of field.

Table 5. XRD and SEM reference

Mix Number	XRD and SEM Reference	28 Days Strength, MPa
OPC I	OPC I	55.93
OPC II	OPC II	56.46
S2	OI3R	38.96
S5	OI3G1	42.28
S8	OI3G2	46.45
S11	OII3R	39.92
S14	OII3GI	42.45
S17	OII3G2	45.01

3. Results and discussion

Table 6 shows the colorimetric analysis results of RFA and GFA. Darker shades are usually correlated with unburned carbon in FA, which may affect water demand or air entrainment in concrete. The results showed that the darkness of the FA increased with increasing fineness. It is because of the increase in finer particles and the increase in the surface area of FA. GFA2 was the darkest, followed by GFA1 and then RFA. All FA samples showed a positive red index, with RFA showing the highest value. RFA also showed the highest positive result for the yellow colour index, followed by GFA1 and GFA2. It indicates that the raw FA was more yellowish than the ground FA. Based on the assessment and findings, the Colorimetric analysis could be a rapid, low-cost method to assess FA consistency in industrial settings. Visual observations of raw and ground FA are shown in Figure 4.

Table 6. Colorimetric analysis of raw and ground FA

Item	Rang	RFA	GFA1	GFA2	
L	0 (dark)	100 (bright)	55.09	51.07	48.19
а	Negative (green)	Positive (red)	0.81	0.51	0.40
b	Negative (blue)	Positive (yellow)	7.83	5.48	4.30



Raw Fly Ash (RFA)



Ground Fly Ash (GFA2)



Ground Fly Ash (GFA1)

Figure 4. Visual observation of RFA, GFA2, and GFA1

3.1. Effect on fresh cement paste

3.1.1. Consistency, initial and final setting time

The consistency of the binders was used to determine the amount of water required for the initial setting time and other mixes. Figure 5 shows the consistency of the two types of OPC and the blended cement pastes with different FA fineness and ratios. A previous study showed that mixes with GFA demand more water owing to their higher fineness and surface area (Krishnaraj & Ravichandran, 2019). The water consistency of the samples with FA varies from 22.8% to 29.1%. The values of the consistency tests for the OPC I and OPC II pastes were found to be 27.9% and 28.3%, respectively. The consistency of FA mixes was generally lower than that of both OPC I and OPC II, except for S4, S7, S13, and S16 mixes. Generally, these batches are from the replacement of 10% GFA. It shows

that Consistency increased with an increase in FA fineness. A higher surface area requires more water for the reaction. The results also indicate that FA replacement reduces water demand, and this trend decreases with an increase in the FA ratio. This finding concurs with previous researchers' studies on replacement with high-volume FA (Bondar & Coakley, 2017).

For mixes with similar fineness, the consistency of the cement paste decreased with an increase in the FA ratio. The results show an increase in water demand with an increase in fineness for a similar FA ratio. The lowest range of water consistency was found in the mixes of S3, S6, S12, and S15. Samples S3 and S12 are from the replacement of RFA 50%, showing the lowest results.

The initial and final setting times of the cement and the mixes are shown in Figure 6. The initial and final setting times increased with the addition of the FA ratio (Kocak &

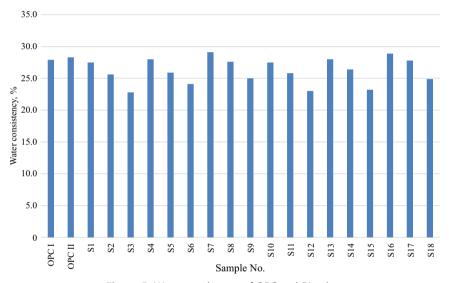


Figure 5. Water consistency of OPC and FA mixes

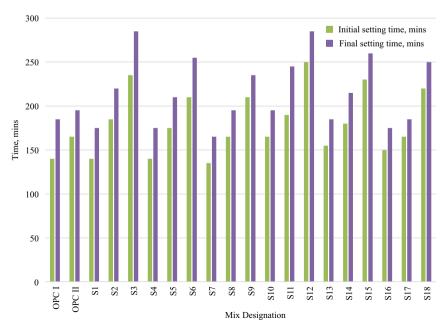


Figure 6. Initial and final setting times of the samples

Nas, 2014). An increase in FA fineness decreases the initial and final setting of the cement paste (Krishnaraj & Ravichandran, 2019). This increase can be seen in the mixes with GFA1 and GFA2 compared with the replacement with RFA FA samples. The OPC I setting time was lower than that of OPC II. The initial and final setting times of S7 are lower than those of OPC I. Thus, it indicates that replacing 10% finer FA (GFA2) has reduced the setting time. This reduction could be explained by the more reactive nature of finer FA particles, which accelerates the pozzolanic reaction and shortens the setting time (Krishnaraj & Ravichandran, 2019). However, it was noticed that the setting time increased when FA increased, although GFA2 was used. The mixes explained this increase with a higher replacement ratio of the cement component to the cementitious

component. The trends were similar for mixes with both OPC 1 and OPC II (Bondar & Coakley, 2017; Krishnaraj & Ravichandran, 2019).

3.2. Soundness or expansion

The expansion of the cement pastes is illustrated in Figure 7, and the results are shown in Figure 8. The expansion of the cement paste is referred to as volume expansion, and excessive amounts of components such as free lime, free magnesia, and sulfate usually cause it. A higher expansion leads to cracks and subsequent damage to the concrete structure. All values obtained were lower than 10 mm, the maximum specification for the MS EN 197-1 standard (Department of Standards Malaysia, 2019).





Figure 7. Soundness test samples

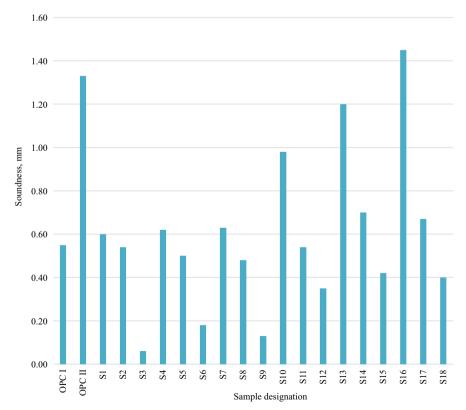


Figure 8. Soundness/expansion of cement paste

The expansion rates were lower in the mixes with FA and decreased with an increase in the FA ratio (Kocak & Nas, 2014). This trend was observed for both types of cement-OPC I and OPC II. OPC II had higher soundness than OPC I. Higher amounts of free lime in OPC II can explain it. In OPC I, replacement with 10% RFA, GFA1, and GFA2 increased soundness. In OPC II, only replacement with 10% GFA2 has shown an increase in the expansion. The 50% FA ratio expansion was the lowest for OPC replacement. Mix S3 was found to decrease by 89% compared to OPC I, while mix S12 was shown to drop by 74% compared to OPC II. The expansion rates increased with increasing FA fineness. This expansion could be attributed to the higher reactivity of finer FA particles.

3.3. Compressive strength rest

The compressive strengths of the mortars for OPC I and OPC II with different FA ratios and fineness are listed in Table 7. Figure 9 shows the compressive development of mortars with 10% FA replacement in both OPC I and OPC II with different fineness of FA. OPC I and II, with their relatively fine particles, also contribute to the early strength owing to their high reactivity (Udebunu et al., 2025). The strength of the development of OPC I was higher than that of OPC II at ages up to 14 days. However, it was noticed that OPC II was on the higher side for 28 and 56 days of strength. It is explained by the higher fineness and C₃A content of OPC I compared to those of OPC II. Higher fineness and C₃A will accelerate hydration and increase strength development at early ages. For the development

of later strength, the content of C₃S plays a vital role, and it is higher in OPC II.

For the comparison of blended mixes with 10% FA, OPC I with GFA2 10% show the highest 1D strength, while OPC II with RFA 10% show the lowest strength. Finer particles, such as those in GFA2, provide a larger surface area for hydration reactions, which can enhance the early strength development in cement mortar (Wang et al., 2022). A previous researcher noticed that an FA replacement of 10% has given an equal strength performance to conventional OPC (Bicer, 2018). The compressive strength of blended mixes of 10% FA was lower than that of pure OPC I and OPC II mixes until 28D. However, it is noticed that the 56-day strength of OPC I with 10% GFA2 and OPC II with 10% GFA2 have surpassed their 100% OPC 56-day strength, respectively. This phenomenon is explained by the pozzolanic reaction of FA, especially in fineness-modified mixes. GFA1 and GFA2, with their controlled particle sizes, can optimize particle packing and reduce porosity, leading to better long-term strength and durability. Both mix with 10% GFA1 and GFA2 fulfilled the strength requirement of class 42.5N of MS EN 197-1 (Department of Standards Malaysia, 2019).

Figures 10 and 11 show the compressive development of mortars with 30% and 50% FA replacement in both OPC I and OPC II with different fineness of FA. Both replacement ratios show lower strength than OPC I and OPC II at all ages. For mixes with 30% GFA1 and GFA2 replacement, although the early strength development was lower than that of their respective OPC, it was noticed that the gap became closer at 28 days and 56 days. If this trend

Table 7.	Compressive	strength	of OPC	and mixes
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Mix No.	Compressive strength (with standard deviations) at age, MPa						
IVIIX INO.	1D	2D	7D	14D	28D	56D	
OPC I	17.12 (0.31)	30.21 (0.54)	44.95 (0.47)	51.05 (0.77)	55.93 (1.38)	58.12 (0.84)	
OPC II	16.85 (0.49)	27.85 (0.36)	43.82 (0.70)	50.80 (0.69)	56.46 (1.16)	59.65 (1.56)	
S1	15.29 (0.42)	26.59 (0.42)	40.26 (0.42)	46.51 (1.08)	49.62 (0.90)	51.59 (1.18)	
S2	12.07 (0.38)	21.88 (0.45)	31.66 (0.95)	35.11 (0.75)	38.96 (0.66)	40.49 (1.41)	
S3	8.63 (0.25)	14.79 (0.48)	23.18 (0.56)	26.06 (0.70)	28.54 (0.88)	29.63 (1.89)	
S4	15.36 (0.25)	27.38 (0.61)	40.71 (0.80)	46.94 (0.88)	51.39 (0.89)	53.66 (1.10)	
S5	11.95 (0.22)	21.71 (0.38)	32.37 (0.59)	37.28 (0.60)	42.28 (1.26)	44.71 (1.29)	
S6	8.61 (0.46)	15.03 (0.51)	23.43 (0.71)	28.95 (1.06)	32.62 (1.17)	35.21 (1.71)	
S7	15.46 (0.38)	27.90 (0.54)	41.01 (0.73)	47.66 (0.55)	55.34 (1.40)	58.61 (1.07)	
S8	11.97 (0.41)	21.57 (0.32)	32.59 (0.51)	38.62 (1.19)	46.45 (1.02)	50.28 (0.89)	
S9	8.60 (0.21)	14.59 (0.54)	25.02 (0.98)	30.61 (1.04)	37.87 (0.71)	42.96 (1.15)	
S10	14.57 (0.49)	24.62 (0.37)	38.58 (0.87)	46.32 (0.64)	50.21 (1.24)	53.09 (0.99)	
S11	12.20 (0.45)	20.03 (0.33)	31.07 (0.96)	35.16 (0.50)	39.92 (1.28)	42.16 (1.92)	
S12	7.62 (0.26)	13.21 (0.68)	21.30 (0.94)	26.21 (1.07)	27.42 (1.11)	29.02 (1.77)	
S13	14.71 (0.25)	24.43 (0.69)	40.70 (0.76)	46.88 (0.99)	52.17 (1.29)	55.35 (1.56)	
S14	12.03 (0.26)	20.17 (0.62)	32.00 (0.95)	37.43 (1.01)	42.45 (1.04)	45.59 (1.85)	
S15	7.76 (0.29)	13.55 (0.42)	23.28 (0.45)	29.57 (1.04)	33.40 (1.07)	36.50 (1.76)	
S16	15.33 (0.36)	25.21 (0.34)	41.25 (0.52)	47.23 (0.55)	54.22 (0.98)	60.09 (1.02)	
S17	11.71 (0.33)	19.69 (0.57)	32.96 (0.43)	39.25 (0.75)	45.01 (0.62)	51.25 (1.87)	
S18	8.97 (0.29)	14.47 (0.48)	24.75 (0.60)	30.36 (0.58)	37.97 (0.70)	43.87 (1.45)	

continues, the gap could be closed or surpassed at later ages, that is, at 90 days. In addition, the mixes with 30% GFA have fulfilled the strength requirement of class 42.5N of MS EN 197-1 (Department of Standards Malaysia, 2019).

For 50% replacement, all the mixes have shown lower strength development owing to a slower rate of hydration. The mix OPC II with 50% GFA2 has performed better at 28, and 56-day strength compared to OPC with 50% GFA2. It

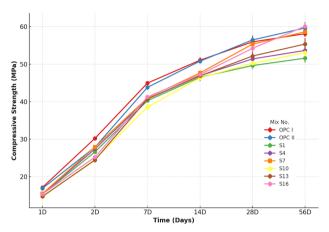


Figure 9. Compressive strength development of OPC and 10% cement FA replacement with different fineness

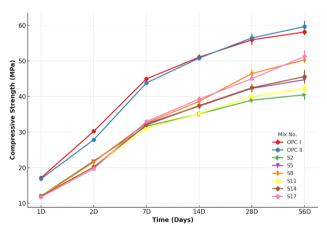


Figure 10. Compressive strength development of OPC and 30% FA replacement with different fineness

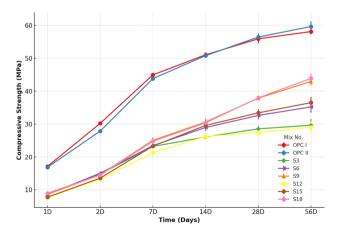


Figure 11. Compressive development of OPC and 50% FA cement replacement with different fineness

shows the effect of fineness on the higher-volume FA replacement. Although lower, the mixes with 50% GFA1 and GFA2 replacements fulfill the strength requirement of class 32.5N of MS EN 197-1 (Department of Standards Malaysia, 2019).

Generally, the strength decreased with the replacement of a higher FA ratio, and it improved when the FA fineness increased (Hsu et al., 2018; Krishnaraj & Ravichandran, 2019; Lin, 2020; Moghaddam et al., 2019). The fineness and PSD of the materials significantly influence the strength development and overall performance of cement mortar. The increased fineness of the ground FA contributed to a denser and more homogeneous microstructure, enhancing the overall strength of the mortar. The pozzolanic reaction between FA and calcium hydroxide resulted in the formation of an additional C-S-H gel, which is responsible for the strength gain (Golewski, 2022; Lin, 2020; Moghaddam et al., 2019; Park & Choi, 2022). Another study also showed that the compressive strength increment is related to the gradual dissolution of FA particles and a positive correlation between FA fineness and compressive strength (Sun et al., 2021).

In addition, the type of OPC used also influenced the compressive strength. Mortar mixes with Type II OPC and FA exhibited a lower heat of hydration and better strength development performance than those with Type I OPC. It could be attributed to the slower rate of hydration of Type II OPC, which is beneficial for long-term strength development.

3.4. Heat of hydration

Figure 12 shows the heat evolution characteristics of the OPC and blended cement mix pastes for up to 7 or 168 h. OPC II was found to have a 10.9% lower heat of hydration at 7 d compared to OPC I. It was explained by the difference in C₃A content between the two types of OPC. The fineness difference between both types of cement also caused the difference in the heat because the higherfineness cement tends to hydrate rapidly compared to the lowest-fineness cement. As identified in previous research, the heat of hydration of FA-blended cement pastes is lower than that of OPC pastes (Moghaddam et al., 2019; Wang et al., 2018). Another study showed that the hydration of cement-based materials differed when FA with different fineness was blended with cementitious materials. Finer FA generated more hydration heat than coarser FA. It was mainly attributed to the enhanced pozzolanic reaction and physical filling effect of the fine and larger surface area of the FA particles. This study also revealed that the particle size of FA had a lesser effect on the hydration heat release during the initial 10 h. An obvious effect on hydration heat release was observed after 10 h. Then, the hydration heat release during 10-50 hours increased rapidly, and the hydration effect gradually decreased with the increase in hydration time and flattened after 80 h (Cui et al., 2022).

The FA fineness increased the heat of hydration in both cement types. For 10% replacement, mixes with GFA

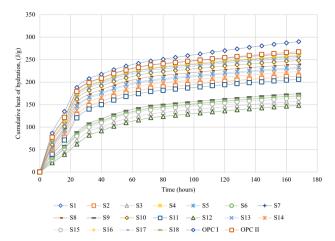


Figure 12. Cumulative heat of hydration for OPC and FA mixes

1 and GFA 2 showed higher heat than those with 10% RFA. This trend was followed by replacing FA with 30% or 50%. Then, for the same fineness of FA, an increase in the FA ratio decreased the heat of hydration in both cement pastes. It was due to the effect of the FA replacement; for the mixes with 50% FA, a decrease in heat evolution was found in seven days by approximately 40% compared to conventional OPC. The results show that mixes of FA with OPC II have lower heat than OPC I in all the respective mix ratios and fineness.

The FA replacement ratio played a significant role in the heat of hydration. Higher FA replacement levels typically result in lower hydration heat due to the reduced amount of OPC, the primary heat source during hydration. The pozzolanic reaction of FA is less exothermic than that of OPC (Hsu et al., 2019; Moghaddam et al., 2019; Wang et al., 2021). Additionally, substituting OPC with FA impacts the early strength development, as the FA reaction rate is slower than that of OPC. Incorporating FA in cement pastes can be particularly beneficial for large concrete structures, where minimizing heat generation is crucial for avoiding thermal cracking. This effect is especially advantageous when using FA with Type II OPC, which already has moderate heat characteristics. This approach could offer a valuable advantage in countries like Malaysia, where Type II OPC is readily available, supporting sustainable and durable concrete applications under tropical conditions.

3.5. Resistance to acid attack

Table 8 displays the mass loss observed in the GFA2 and blank OPC mortar cube mixes when immersed in HCl solution. The appearance of mortar cubes immersed in acid is illustrated in Figures 13–15. The mass loss rate in OPC II was lower than in OPC I. This phenomenon is likely due to the lower C_3A composition of OPC II, which is less susceptible to chemical degradation (ASTM, 2017). The mixes containing FA exhibited lower mass losses than their OPC counterparts. This finding is consistent with previous studies wherein adding 35% FA to cement paste resulted in lower mass loss in acetic acid compared to OPC mixtures

(Dyer, 2017). Moreover, an increase in the FA ratio correlated with a decrease in the mass loss for both cement mixes. These results indicated that adding FA significantly enhanced the durability of the cement mortar. The improved acid resistance can be attributed to the lower free lime content and denser microstructure caused by adding the FA. The pozzolanic reaction between FA and calcium hydroxide produces an additional C-S-H gel, which improves the chemical resistance of the mortar. This property is particularly crucial for structures subjected to harsh environments, such as those in industrial and marine settings (Krishnaraj & Ravichandran, 2019; Kwasny et al., 2018).

Table 8. Mass loss (%) of OPC and GFA2 mixes exposed to HCl acid solution

Mass	GFA2							
	Plain OPC		FA (10%)		FA (30%)		FA (50%)	
(%)	OPC I	OPC II	S7	S16	S8	S17	S9	S18
14D	6.30	4.60	5.88	4.05	2.97	2.57	2.34	1.66
28D	8.82	6.48	7.59	5.84	4.20	3.24	3.32	2.35



Figure 13. Appearance of fresh mortar cubes







Figure 14. Appearance on day 14





Figure 15. Appearance on day 28

Additionally, the evaluation indicates that the Type II OPC and its FA blends demonstrate superior acid resistance compared to the Type I OPC. Incorporating FA with Type-II OPC may further improve the acid resistance of cement mortar and concrete, which is crucial for the durability and longevity of structures under acidic conditions.

3.6. X-ray Diffraction (XRD)

3.6.1. XRD plot of OPC (type I)-Fly ash blended cement mortar specimens

Figure 16 shows the XRD patterns of OPC mortar using OPC Type I, OPC I with 30% RFA blended mortar (OI3R), OPC with 30% GFA2 (98% fineness) blended mortar (OI3G2), and OPC with 30% GFA1 (90% fineness) blended mortar (OI3G1). Portlandite (P) or Ca(OH)2 was the second most prevalent hydration product during the hydration process. The major crystalline peaks included portlandite (P), quartz (Q), and the minor compound calcite (CC). Control specimen OPC I obtained the highest 28-day mortar compressive strength value of 55.93 MPa. Peaks that show the intensity of Ca(OH)₂ appeared at 20 values of 18.12°, 34.14°, and 47°. The partial replacement of 30% RFA in the blended OPC specimen (OI3R) shows a significant reduction in portlandite (Ca(OH)2) crystals. Incorporation of 30% ground FA with fineness percentages of 90% (OI3G1) and 98% (OI3G2) in the blended OPC specimens enhanced Ca(OH)₂ consumption, as evidenced by the reduction in portlandite crystal intensity at 20 of 34.14° and 47°. The 28-day compressive strength of these blends was 42.28 MPa (Chindaprasirt et al., 2007; Hsu et al., 2018; Rao & Kumar, 2021). The RFA-blended OPC

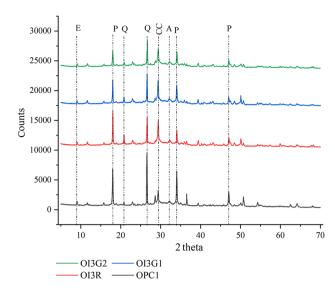


Figure 16. XRD diagram of fly ash blended OPC Type I mortar specimens

mortar specimen (OI3R) exhibited the lowest 28-day compressive strength of 38.96 MPa. Calcite (CC) crystals were observed in the OPC I specimen because of the carbonation effect from the atmosphere. The incorporation of FA enhanced the formation of calcite crystals with a diffraction peak at $2\theta = 29.4^{\circ}$. Alite (A) crystals were observed in their unreacted form at $2\theta = 32^{\circ}$ in all specimens. The effect of FA fineness showed a positive trend in strength enhancement, with OI3G2 achieving a higher compressive strength (46.45 MPa) than OI3G1 (42.28 MPa). It indicates that the pozzolanic reactivity in the FA-based blended cement mortar increases with the fineness of the FA (Cui et al., 2022; Moghaddam et al., 2019).

3.6.2. XRD plot of OPC (type II)-Fly ash blended cement mortar specimens

Figure 17 shows the X-ray diffraction diagram of OPC mortar using Type II cement (OPC II), OPC with 30% RFA blended mortar (OII3R), OPC with 30% ground FA (98% fineness) blended mortar (OII3G2), and OPC with 30% ground FA (90% fineness) blended mortar (OII3G1). The control specimen, OPC II, had the highest 28-day compressive strength value of 56.46 MPa. The RFA-blended OPC mortar specimen showed consumption of portlandite (P) crystals, with a 28-day compressive strength of 39.92 MPa. It indicates that the degree of the pozzolanic reaction of RFA was relatively slow. The effect of FA fineness demonstrated a positive trend in strength enhancement, like OPC I, with OII3G2 achieving a higher compressive strength of 45 MPa than OII3G1 with 42.5 MPa. It indicates that the pozzolanic reactivity of FA increases with its fineness (Cui et al., 2022; Liu et al., 2020; Moghaddam et al., 2019).

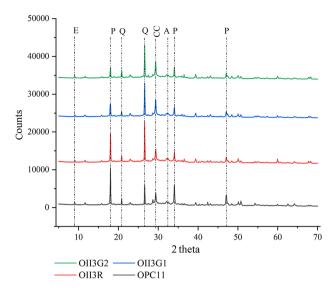


Figure 17. XRD diagram of OPC Type II blended fly ash cement mortar specimens

3.7. Scanning electron microscopy (SEM)

Figures 18–25 show the SEM scanning images of the OPC-FA blended mortar specimens using two OPC types: OPC Type I and Type II. As the material matures, the intensity of the additional phases diminishes while their morphology changes. The structure gradually solidified and became more compact. The porous zones are initially progressively filled and sealed by the C-S-H or ettringite phases (Golewski, 2022, 2023b). As commonly observed, OPC I samples contain needle-shaped and trapezoidal ettringite crystals along with portlandite (Franus et al., 2015). OPC II specimens, as observed in Figure 19, contain less ettringite (De Weerdt et al., 2011). The replacement of OPC with FA leads to a decline in ettringite crystals, as shown in Figures 18–23. This indicates that the SEM micrographs depict the mortar mixes as having compact or dense microstructures. The spherical particles in the SEM images are the unreacted FA particles that act as fillers in the matrix, which can be observed in Figure 18 and Figure 24. A prior study reported that FA particles can persist in an unreacted state and remain detectable after two years (Golewski, 2022). The finer particles enhance particle packing within the sample through the filler effect, which fills the interstitial spaces in the mortar, resulting in a denser microstructure and increased strength. Hydration products, such as Portlandite and Ettringite, can be formed after the hydration process.

3.8. Environmental implications: Preliminary Life Cycle Assessment (LCA) considerations

The sustainability potential of FA-modified cement hinges on balancing clinker replacement benefits against the energy costs of FA grinding. While Type II OPC production and FA fineness modification entail additional embodied energy, our results demonstrate significant long-term environmental advantages:

- 1. CO₂ Reduction: Replacing 30–50% OPC with FA directly reduces clinker demand, mitigating ~0.21–0.35 tons of CO₂ per ton of cement (Alqahtani et al., 2021). This study aligns with Malaysia's net-zero goals by lowering the carbon footprint of construction materials.
- 2. Durability-Driven Savings: Blends with 30% GFA2 achieved 56-day strengths exceeding 46 MPa, complying with MS EN 197-1 Class 42.5N standards (Department of Standards Malaysia, 2019). Such durability minimizes infrastructure repair/replacement cycles, indirectly reducing lifecycle emissions (Golewski, 2022).
- 3. Grinding Energy Trade-off: Mechanical activation of FA (e.g., achieving 98% fineness) consumes ~30–50 kWh/ton (Bondar & Coakley, 2017). However, this is offset by the 40% reduction in hydration heat (Figure 12), which lowers thermal cracking risks in mass concrete applications, extending service life.

Future work should quantify net emissions via full LCA, including upstream (grinding) and downstream (durability) impacts. Nevertheless, the synergy between FA fineness, performance, and clinker substitution positions this approach as a viable transitional strategy for decarbonizing Southeast Asia's cement sector.

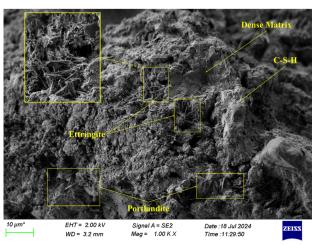


Figure 18. SEM for sample OPC I

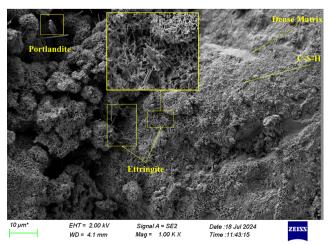


Figure 19. SEM for sample OPC II

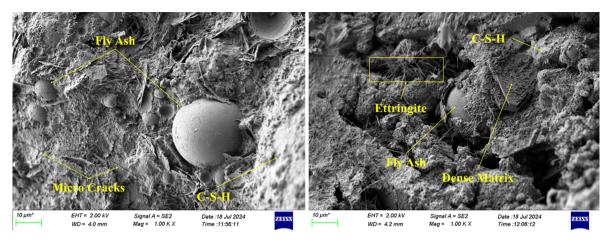


Figure 20. SEM for sample OI3G1

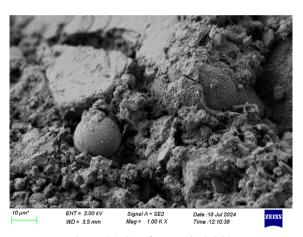


Figure 21. SEM for sample OI3G2

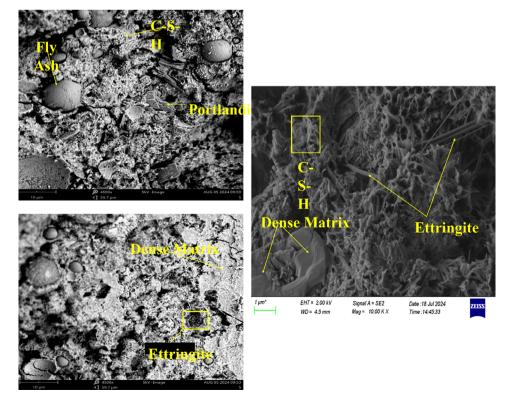


Figure 22. SEM for sample OI3R

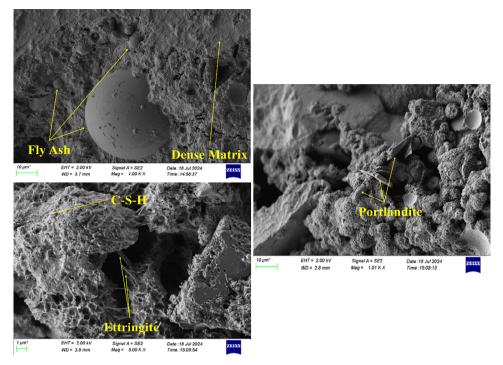


Figure 23. SEM for sample OII3G1

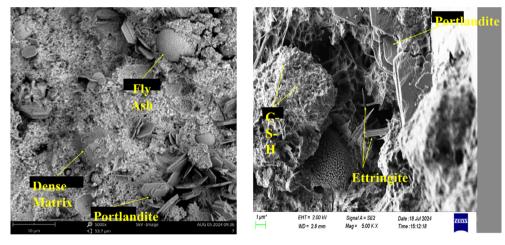


Figure 24. SEM for sample OII3G2

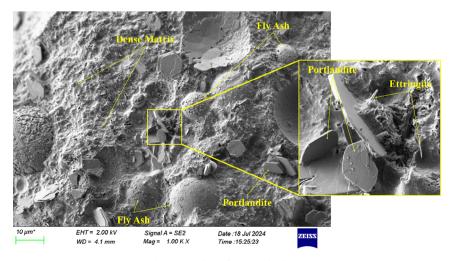


Figure 25. SEM for sample OII3R

4. Conclusions

From the analyses and evaluations conducted, the following conclusions can be summarized:

- Colorimetric analysis revealed that increased FA fineness correlates with darker colouration (L-value reduction), likely due to higher unburned carbon content. This suggests that fineness modification could inadvertently introduce variability in FA quality, necessitating complementary chemical analysis for industrial use.
- 2. Mortar mixes containing ground FA exhibited higher Consistency than mixes containing raw FA. This phenomenon can be attributed to FA's higher fineness of the ground FA, which requires more water for the reaction. Furthermore, water consistency decreased with increasing FA ratio.
- 3. The addition of FA increased the mortar setting time. However, increasing the FA's fineness reduced the mortar paste's setting time. This indicates that finer FA particles facilitate quicker hydration reactions owing to their larger surface areas.
- 4. The expansion or soundness of the mortar decreased with an increasing FA ratio. This suggests that incorporating higher amounts of FA mitigates expansive reactions that can lead to cracking, thereby improving the mortar's dimensional stability.
- 5. FA fineness directly enhances mechanical and durability properties. Mortars with 10% GFA2 (98% fineness) achieved 58.6 MPa at 56 days, surpassing pure Type I OPC (58.1 MPa). At 30% replacement, GFA2 blends met Class 42.5N strength standards MS EN 197-1 (Department of Standards Malaysia, 2019), proving FA's viability without performance tradeoffs.
- **6.** Type II OPC and its mixes with FA exhibited a lower heat of hydration than their respective Type I OPC and its mixes. This is due to the Type II OPC's lower reactivity and slower hydration rate. The heat of hydration of the cement paste decreased with increasing FA ratio. However, mixes with higher fineness FA showed an increase in the heat of hydration. Grinding FA to 98% fineness is offset by 40% lower hydration heat and extended service life in aggressive environments, justifying the energy trade-off.
- 7. Type II OPC blends exhibited superior durability, with 43% lower mass loss in acid exposure than Type I. This is attributed to reduced C₃A content and denser microstructures.
- 8. Despite grinding-related energy costs, FA replacement reduces clinker demand, directly cutting CO₂ emissions by 0.25 tons per ton of cement. The enhanced durability of FA blends further supports long-term emission savings through reduced maintenance.
- **9.** Unlike prior studies on Type I OPC or extreme FA fineness, this work bridges lab-scale research with industrial practicality by tailoring FA grind levels

(90–98%) to regional standards MS EN 197-1 (Department of Standards Malaysia, 2019) and Type II OPC's sulphate resistance.

Finally, several areas are recommended for future research, including incorporating other SCMs, further reducing FA particle size to the nanoscale, analyzing using mercury intrusion porosimetry, assessing sulfate resistance, and long-term evaluations of strength and durability. Future studies should integrate LCA or Life Cycle Inventory (LCI) frameworks to assess net environmental benefits holistically, paving the way for standardized FA utilization in global cement practice.

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Conflict of interests

The author confirms that this article's content has no conflict of interest.

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