

A Study on the Microstructural and Mechanical Performance of Type F Fly Ash Concrete under Sulfuric Acid and Acid Mine Drainage Exposure

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ABSTRACT

Concrete is the main construction material for infrastructure, but its long-term durability is severely affected by acidic environments, especially in mining regions. This study examines the mechanical and microstructural properties of concrete that uses 15% Type F fly ash and is exposed to sulfuric acid and Acid Mine Drainage (AMD). This research combines analyses of compressive, splitting tensile, and flexural performance with Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD) to determine degradation mechanisms under prolonged acidic curing (28, 56, and 84 days). The results show that using fly ash, significantly enhances strength and durability under tough conditions. Similar improvements were recorded in splitting tensile and flexural strengths, indicating reduced degradation under exposure to both sulfuric acid and AMD. Microstructural analysis revealed pore refinement, reduced calcium hydroxide content, limited microcrack propagation, and suppression of expansive deterioration products. This enhanced performance is governed by secondary pozzolanic reactions, matrix densification, and the increased chemical stability of hydration products. These findings mechanistically explain the role of fly ash in improving acid resistance and support its use in infrastructure in mining and acid-prone environments.

Keywords-fly ash; concrete; microstructure; acidic environment; mechanical performance

I. INTRODUCTION

Concrete exhibits high compressive strength, long-term durability and cost-effectiveness, but its durability depends significantly on environmental exposure, with acidic conditions leading to surface scaling, cracking, and mass loss. Acid Mine Drainage (AMD) is one of the most severe forms of exposure, because hydrogen ions react with calcium hydroxide and calcium silicate hydrate (C-S-H), resulting in leaching,

decalcification, and progressive degradation of structural integrity [1, 2]. In order to prevent this degradation, Supplementary Cementitious Materials (SCMs), particularly fly ash, have been used due to its pozzolanic reactivity. Fly ash improves workability and promotes the formation of additional C-S-H gel, which refines pores and densifies the matrix reducing the penetration of aggressive ions [3]. The enhancement of durability is primarily attributed to fly ash's ability to modify the chemical stability of hydration products

by consuming calcium hydroxide, improving the resistance to formation of expansive products, especially in acidic, sulfate-rich environments [4]. In coal-producing regions, such as East Kalimantan in Indonesia, rebuilding post-mining land for infrastructure poses serious challenges due to acidic mine water. Conventional Portland cement concrete is highly susceptible to acidic attack due to its calcium-rich hydration products, which highlights the need for concrete materials with enhanced resistance and improved long-term mechanical performance [4, 5]. Among the various SCMs, fly ash is preferred, due to its high silica and alumina content, wide availability, and environmental benefits. Through pozzolanic reactions, fly ash produces secondary C-S-H gel, resulting in reduced permeability and a more refined pore structure [6, 7]. Although fly ash concrete may have lower early-age strength due to slower reaction kinetics, it often has superior mechanical performance and durability at later ages limiting the formation of expansive products, such as gypsum and ettringite, under acidic exposure [8-11]. This densification of the microstructure is crucial for maintaining concrete's structural capacity. Previous studies focus on isolated mechanical properties or short-term exposure, leaving a gap in compressive, tensile, and flexural behavior with microstructural analysis. Flexural strength is highly sensitive to microcrack formation and Interfacial Transition Zone (ITZ) degradation, a critical indicator of long-term performance [12, 13]. Additionally, the mechanisms linking microstructural refinement to mechanical resilience in acidic environments remain unclear. This study aims to evaluate the mechanical and microstructural performance of fly ash concrete exposed to sulfuric acid and AMD to clarify degradation mechanisms and contribute to the development of durable, sustainable concrete materials for infrastructure in acidic environments.

II. MATERIALS AND METHODS

A. Materials

This study used Type I Portland cement, exhibiting setting time characteristics and compressive strength suitable for structural concrete applications. The fly ash used was Type F and was sourced from the Balikpapan coal-fired power plant, as shown in Figure 1. It is characterized by high silica and alumina content and low calcium oxide, which makes it ideal for improving concrete's resistance to acidic environments [13]. The fine aggregate in this study consisted of Samboja sand with a fineness modulus that met the relevant standard specifications. The coarse aggregate was crushed stone with a nominal maximum size of 20 mm. All aggregates satisfied the standard requirements for gradation, cleanliness, specific gravity, and water absorption. Clean potable water was used for concrete mixing and standard curing procedures. The media utilized for immersion consisted of freshwater, a sulfuric acid solution, and AMD. The sulfuric acid solution had a pH of 3, and the AMD was collected directly from a coal mining site [14]. Chemical analysis revealed that the AMD had a low pH and high sulfate content, representing realistic field conditions.

B. Mix Design and Specimen Preparation

The concrete mixture was designed to achieve a compressive strength target of 25 MPa with a water-to-cement

ratio of 0.51. Authors in [12] improved durability at this substitution level, using fly ash as a partial replacement for cement. Two types of specimens were prepared: cylindrical specimens (150 mm × 300 mm) for compressive and splitting tensile strength measurements [15], and a beam (150 mm × 150 mm × 600 mm) for flexural strength measurements [16]. All specimens were cast using identical mix proportions, as depicted in Table I.



Fig. 1. Fly ash type F.

TABLE I. CONCRETE MIX DESIGN PARAMETERS FOR NORMAL AND FLY ASH-MODIFIED CONCRETE

Parameter	Value
Cement	396.48 kg/m ³
Water	128.9 kg/m ³
Coarse aggregate	1277.34 kg/m ³
Fine aggregate	553.22 kg/m ³
15% Fly ash	59.47 kg/m ³

The selection of 15% fly ash as a partial cement replacement was based on optimization studies reporting that moderate fly ash contents (10–20%) provide balanced improvements in early-age mechanical performance and long-term durability. Lower replacement levels (less than 10%) may not significantly enhance pore refinement or reduce calcium hydroxide content. Higher replacement levels (more than 20%) may reduce early compressive strength due to slower pozzolanic reaction kinetics. Fly ash contents within this optimal range improve sulfate resistance, decrease permeability, and enhance resistance to acid-induced degradation through microstructural densification and reduced calcium hydroxide (Ca(OH)₂) availability [17-19]. Therefore, 15% replacement was selected to ensure sufficient early-age strength while maximizing durability performance under aggressive acidic exposure. The details of the test specimens are presented in Table II. After casting, the specimens were covered to prevent moisture loss, and then demolded after 24 h. The specimens were subsequently divided into three curing groups: normal water, sulfuric acid, and acid mine water. Figures 2 and 3 display the condition of the concrete after immersion in normal water and acidic environments. The pH of the acidic curing solutions was monitored daily using a digital pH meter. The solutions were adjusted by adding sulfuric acid when necessary and replaced every 14 days to maintain chemical stability throughout the curing period. To ensure statistical reliability, three identical specimens were tested for each curing age (28, 56, and 84 days) and exposure condition. Mechanical testing and microstructural analysis were

conducted at 28-, 56-, and 84-day curing ages to evaluate short- and long-term performance.

C. Mechanical Testing

Compressive strength tests were conducted using a universal testing machine in accordance with standard methods. Each specimen was loaded continuously at a controlled rate until failure, after which the maximum applied load was used to determine the compressive strength value. Splitting tensile strength was determined by subjecting cylindrical specimens to diametral compression. This indirect tensile test provides insights into the cracking resistance and tensile performance of concrete [20]. Flexural strength testing was performed on beam specimens using a third-point loading configuration. This loading scheme produces a constant bending moment region, providing a reliable representation of the flexural behavior of structural elements subjected to bending [21].

TABLE II. TEST OBJECT DETAILS

Test object code	Fly ash (%)	Age (days)			Curing medium (water)
		28	56	84	
Compressive strength					
BN 01	0	3	-	-	Fresh water
BN 02	0	3	-	-	Sulfuric acid
BN 03	0	3	-	-	Acid mine
BF 01	15	3	-	-	Fresh water
BF 02	15	3	3	3	Sulfuric acid
BF 03	15	3	3	3	Acid mine
Splitting tensile strength					
BN 01	0	3	-	-	Fresh Water
BN 02	0	3	-	-	Sulfuric acid
BN 03	0	3	-	-	Acid mine
BF 01	15	3	-	-	Fresh Water
BF 02	15	3	3	3	Sulfuric acid
BF 03	15	3	3	3	Acid mine
Flexural strength					
BN 01	0	3	-	-	Fresh Water
BN 02	0	3	-	-	Sulfuric acid
BN 03	0	3	-	-	Acid mine
BF 01	15	3	-	-	Fresh Water
BF 02	15	3	3	3	Sulfuric acid
BF 03	15	3	3	3	Acid mine



Fig. 2. Concrete after immersion in normal water.

D. Microstructural Analysis

Microstructural characterization was conducted to establish correlations between mechanical performance and internal structural evolution. Prior to analysis, the concrete samples were oven-dried at 60 °C for 24 h to remove free moisture. For

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) observations, the dried samples were coated with a thin layer of gold to enhance electrical conductivity. For X-ray Diffraction (XRD) analysis, the samples were ground into particles smaller than 75 µm. SEM was used to examine morphological features, pore distribution, and crack propagation, whereas EDS was used to determine the elemental composition, particularly the concentrations of calcium, silicon, and aluminum. Additionally, XRD analysis was performed to identify crystalline phases and hydration products, providing deeper insights into the chemical degradation mechanisms associated with acidic exposure [22].



Fig. 3. Concrete after immersion in sulfuric acid water and acid mine water.

III. RESULTS AND DISCUSSION

A. Atomic Absorption Spectroscopy Testing

An Atomic Absorption Spectrophotometry (AAS) test was conducted to determine the material's chemical composition. For this study, the chemical composition of the fly ash was analyzed based on primary AAS data obtained from the SUCOFINDO laboratory in Samarinda City, as presented in Table III. Based on these results, the pozzolan content of fly ash, consisting of several compounds (SiO₂ (silicon dioxide), Al₂O₃ (aluminum oxide), and Fe₂O₃ (iron trioxide)), is determined. According to ASTM C 618-03, fly ash with a pozzolan content greater than 50% (SiO₂ + Al₂O₃ + Fe₂O₃) is classified as Type F fly ash.

TABLE III. CHEMICAL COMPOSITION OF FLY ASH BASED ON AAS ANALYSIS

No	Parameter	Unit	Value
1	SiO ₂ (Silicon Dioxide)	%	43,36
2	Al ₂ O ₃ (Aluminium Oxide)	%	15,16
3	Fe ₂ O ₃ (Iron Trioxide)	%	19,37
4	CaO (Calcium Oxide)	%	9,27
5	MgO (Magnesium Oxide)	%	6,77
6	Na ₂ O (Sodium Oxide)	%	0,45
7	K ₂ O (Potassium Oxide)	%	1,34
8	MnO ₂ (Manganese Dioxide)	%	0,32
9	TiO ₂ (Titanium Oxide)	%	0,49
10	Cr ₂ O ₃ (Chromium Trioxide)	%	0,01
11	P ₂ O ₅ (Diphosphorus Pentoxide)	%	0,41
12	SO ₃ (Sulphur Trioxide)	%	2,84
13	LOI (Loss On Ignition)	%	2,10
14	Carbon	%	1,01

B. Compressive Strength

The compressive strength results are presented in Table IV and Figure 4. Concrete performance was significantly affected by exposure to acidic environments, with conventional concrete under sulfuric acid curing showing the greatest strength reduction, particularly at later ages. This deterioration was caused by the dissolution of $\text{Ca}(\text{OH})_2$, decalcification of C-S-H, and formation of expansive gypsum, all of which weakened the cementitious matrix. Concrete with fly ash, however, exhibited enhanced resistance to compressive strength degradation. Although fly ash concrete had slightly lower compressive strength after 28 days due to slower early-age pozzolanic reaction kinetics, it demonstrated substantial strength development after 56 and 84 days. Under sulfuric acid and acid mine water curing conditions, fly ash-modified concrete consistently achieved higher compressive strength than conventional concrete. The sustained pozzolanic reaction promoted the formation of secondary C-S-H gel, which led to matrix densification and reduced permeability, constraining acid penetration [7, 23, 24].

TABLE IV. COMPRESSIVE STRENGTH RESULTS

No	Fly ash	Age (days)	Curing medium (water)	f_{cr} (MPa)	Standard deviation	f'_{cr} (Mpa)
1	0%	28	Fresh	29.50	1.33	28.97
2	0%	28	Sulfuric acid	27.46	1.92	26.69
3	0%	28	Acid	27.65	1.80	26.93
4	15%	28	Fresh	29.60	0.43	29.43
5	15%	28	Sulfuric acid	28.69	0.52	28.48
6	15%	28	Acid	30.05	1.04	29.63
7	15%	56	Sulfuric acid	28.11	1.76	27.41
8	15%	56	Acid	29.88	1.86	29.14
9	15%	84	Sulfuric acid	29.84	3.09	28.60
10	15%	84	Acid water	30.24	1.21	29.76

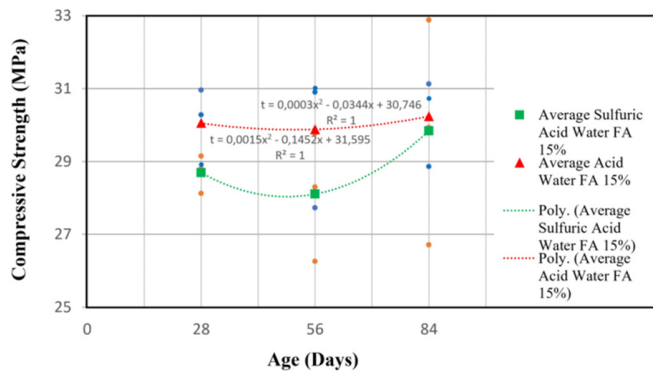


Fig. 4. Compressive strength of normal concrete and fly ash concrete at different curing ages and exposure conditions.

C. Splitting Tensile Strength

The results of the splitting tensile strength test are portrayed in Table V and Figure 5. Compressive strength was less sensitive to acidic attack than this parameter, with normal concrete showing rapid degradation due to microcracking and ITZ deterioration, especially under sulfuric acid curing.

TABLE V. SPLITTING TENSILE STRENGTH RESULTS

No	Fly ash	Age (days)	Curing medium (water)	f_{cr} (Mpa)	Standard deviation	f'_{cr} (Mpa)
1	0%	28	Fresh	2.79	0.20	2.71
2	0%	28	Sulfuric acid	2.39	0.30	2.27
3	0%	28	Acid	2.72	0.10	2.68
4	15%	28	Fresh	3.12	0.03	3.11
5	15%	28	Sulfuric acid	2.60	0.01	2.60
6	15%	28	Acid	3.11	0.25	3.01
7	15%	56	Sulfuric acid	2.68	0.16	2.62
8	15%	56	Acid	2.58	0.45	2.4
9	15%	84	Sulfuric acid	2.90	0.27	2.79
10	15%	84	Acid	2.60	0.45	2.42

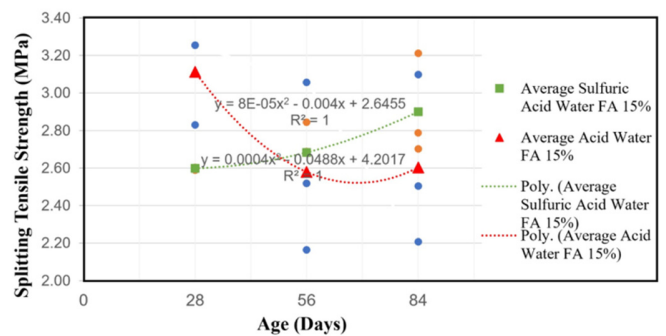


Fig. 5. Splitting tensile strength of concrete specimens under acidic exposure.

After 84 days, the fly ash mixture showed a significant increase in tensile strength compared to normal concrete. This improvement was attributed to the enhanced quality of the ITZ, which resulted from the filler effect of the fine fly ash particles and the formation of a supplementary calcium silicate hydrate (C-S-H) gel. These mechanisms contributed to improved stress distribution and greater resistance to crack propagation [25].

D. Flexural Strength

Flexural strength was the most sensitive parameter to acidic environments, which indicates the vulnerability of structural elements dominated by bending, as shown in Table VI and Figure 6.

TABLE VI. FLEXURAL STRENGTH RESULTS

No	Fly ash	Age (days)	Curing medium (water)	f_{cr} (MPa)	Standard deviation	f'_{cr} (Mpa)
1	0%	28	Fresh	5.19	0.27	5.08
2	0%	28	Sulfuric acid	5.10	0.98	4.71
3	0%	28	Acid	4.68	0.61	4.44
4	15%	28	Fresh	5.14	1.23	4.65
5	15%	28	Sulfuric acid	4.10	0.24	4.00
6	15%	28	Acid	5.01	0.66	4.75
7	15%	56	Sulfuric acid	4.91	0.53	4.70
8	15%	56	Acid	5.10	0.80	4.78
9	15%	84	Sulfuric acid	5.16	0.82	4.83
10	15%	84	Acid	5.25	0.50	5.05

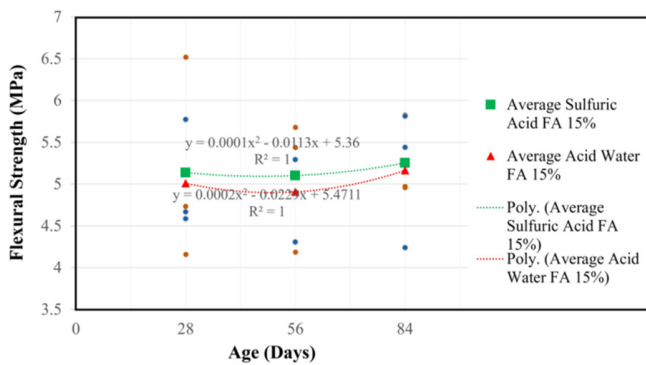


Fig. 6. Flexural strength of normal concrete and fly ash concrete under acidic environments.

Concrete with fly ash exhibited improved flexural behavior under all curing conditions. At 56 days and 84 days, the fly ash mixture had considerably higher residual flexural strength than normal concrete. This enhanced performance is associated with pore structure refinement and reduced calcium hydroxide content, resulting in slower degradation mechanisms and limited crack propagation under flexural loading [10, 26]. Figure 7 presents a comparison of the compressive strength, splitting tensile strength, and flexural strength at 28, 56, and 84 days.

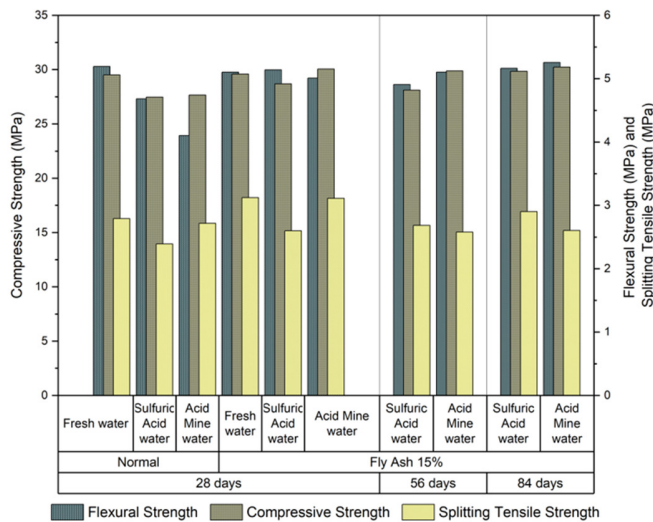


Fig. 7. Comparison of compressive, splitting tensile, and flexural strength results.

E. Microstructural Characteristics

Microstructural characterization was performed using SEM, EDS, and XRD techniques. The analyzed specimens were concrete samples at 28-, 56-, and 84-day curing ages. These results allowed for a comparative evaluation of conventional concrete and fly ash-modified concrete when exposed to freshwater, AMD, and sulfuric acid water [22]. The SEM and EDS analysis results are displayed in Figures 8 and 9.

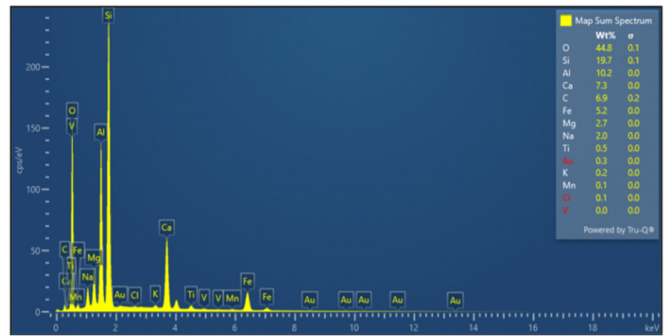


Fig. 8. SEM results of normal concrete.

SEM observations revealed distinct differences in microstructural characteristics between conventional concrete and fly ash-modified concrete following exposure to acids. Normal concrete showed pronounced pore enlargement, dissolution of hydration products, and development of interconnected microcracks, especially when exposed to sulfuric acid. These microstructural changes accelerated the diffusion of acidic solutions into the cement matrix and weakened the bonding between hydration phases, leading to a substantial reduction in mechanical properties. Similar degradation mechanisms have been reported in [27, 28], which identified increased pore connectivity as a key factor in accelerated concrete deterioration in acidic environments.

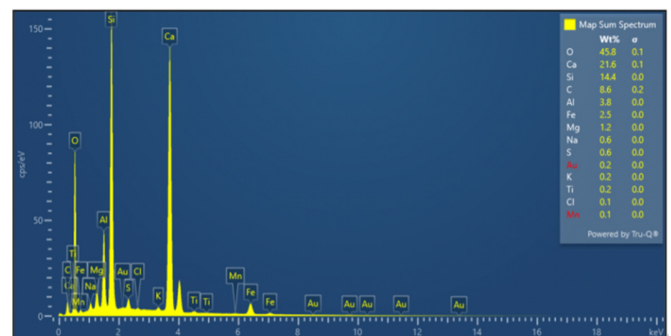


Fig. 9. SEM results of fly ash concrete.

Degradation was slower in fly ash concrete due to the development of a denser microstructure. After 28 days of exposure, the microstructure was characterized by a compact C-S-H matrix, minimal microcracking, and low pore connectivity. These conditions resulted from secondary pozzolanic reactions between the fly ash and the calcium hydroxide (Ca(OH)₂), which generated additional calcium silicate hydrate (C-S-H) and refined the pore structure. This densification mechanism aligns with the results in [29], reporting the critical role of pozzolanic reactions in restricting the ingress of aggressive ions during the initial exposure stages. After 84 days of acidic exposure, more pronounced microstructural degradation occurred, including increased porosity, wider microcracks, and partial dissolution of calcium-rich phases. These changes suggest that long-term acid exposure gradually induces the decalcification of C-S-H, even in refined pore systems. However, compared with normal concrete, fly ash concrete retained a relatively intact matrix,

suggesting that the combined effects of pore refinement and reduced $\text{Ca}(\text{OH})_2$ content effectively slow the diffusion of H^+ and SO_4^{2-} ions. Authors in [25] reported similar mechanisms in long-term durability studies of SCM-based concretes. Figures 10 and 11 show EDS analyses indicating that fly ash–modified concrete has lower calcium levels and higher concentrations of silica and alumina than normal concrete. This suggests the effective consumption of $\text{Ca}(\text{OH})_2$ through ongoing pozzolanic reactions. The reduced availability of free calcium decreased the number of phases vulnerable to acid dissolution, thereby slowing leaching processes. This finding aligns with [30], demonstrating that a lower Ca/Si ratio enhances chemical stability in acidic environments. The XRD results, illustrated in Figures 12 and 13, revealed that the crystalline phases of the fly ash concrete were primarily quartz (SiO_2), a highly stable compound in acidic environments. The presence of mullite as a secondary phase also contributed to microstructural stability. The low intensity of the portlandite peaks confirmed the effective consumption of $\text{Ca}(\text{OH})_2$ through pozzolanic reactions. Additionally, the absence of significant amounts of degradation products, such as gypsum and ettringite, suggests that sulfate reactions were effectively suppressed [28, 29].

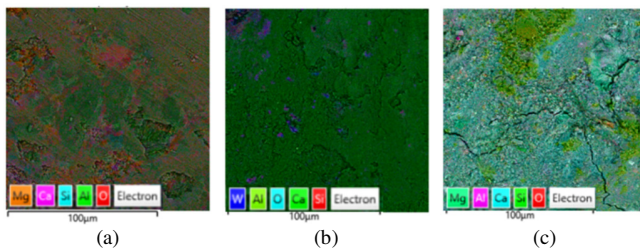


Fig. 10. EDS results of normal concrete in: (a) fresh water, (b) sulfuric acid and (c) acid mine water.

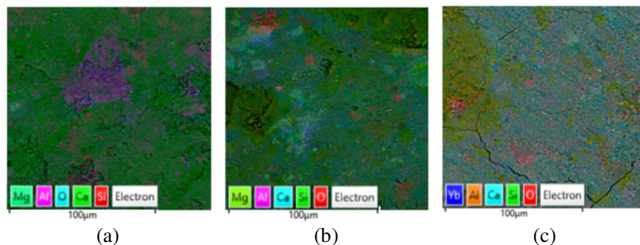


Fig. 11. EDS results of fly ash concrete in (a) fresh water, (b) sulfuric acid, and (c) acid mine water.

Phase percentage analysis revealed that quartz was the dominant phase, followed by mullite and magnetite. Calcium-based hydrate phases were present only in small amounts. These results suggest that, despite the initial dissolution of calcium compounds, the formation of expansive degradation products was effectively suppressed, playing a crucial role in maintaining microstructural integrity and restricting microcrack propagation. The improved durability of fly ash–modified concrete in acidic environments is attributed to three primary mechanisms: densification of the pore structure resulting from secondary pozzolanic reactions, reduction of calcium-rich phases highly susceptible to acid-induced leaching, and predominance of chemically stable mineral phases within the

cementitious matrix. These mechanisms act synergistically to delay acid ingress, mitigate C–S–H decalcification, and suppress microcrack development. Integrating SEM, EDS, and XRD results with mechanical performance data shows that these microstructural mechanisms directly contribute to retaining compressive, splitting tensile, and flexural strengths over the long term.

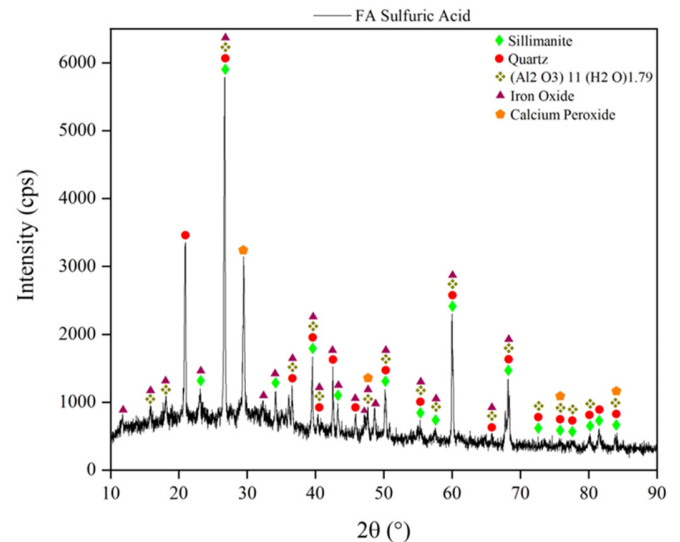


Fig. 12. XRD of sulfuric acid water fly ash concrete.

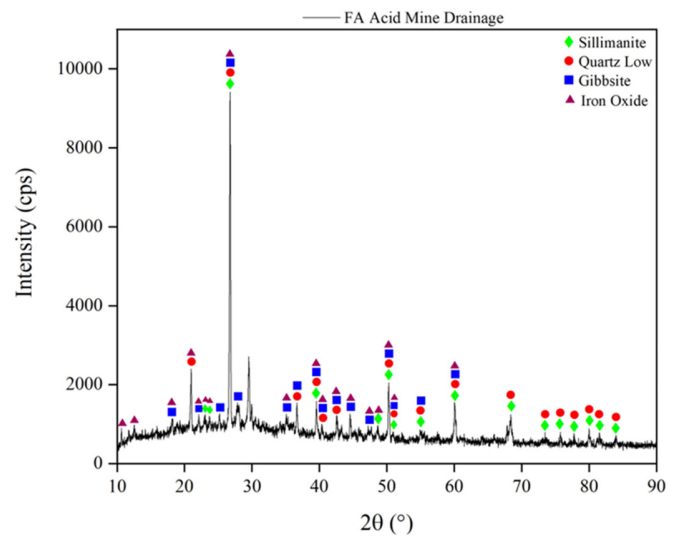


Fig. 13. XRD of acid mine water fly ash concrete.

IV. CONCLUSIONS

Based on results obtained at 28, 56, and 84 days, adding fly ash significantly improved the resistance of concrete to Acid Mine Drainage (AMD) and sulfuric acid exposure. Mechanical testing showed that fly ash concrete experienced slower reductions in compressive, splitting tensile, and flexural strengths than normal concrete. Microstructural analysis confirmed reduced porosity, a denser matrix structure, and lower levels of microdamage, including limited crack

development and minimal gypsum and ettringite formation. When exposed to AMD, the fly ash mixture maintained a stable, compact microstructure. In a more aggressive sulfuric acid environment, however, degradation was observed; it was less severe than in conventional concrete. Overall, replacing some of the cement with fly ash enhances the concrete's durability and microstructural stability in acidic environments, making fly ash concrete a promising material for infrastructure in mining and other acid-prone regions. However, this study was limited to one replacement level (15%) and a maximum exposure duration of 84 days under controlled laboratory conditions. Only two acidic environments were investigated without variations in pH or cyclic exposure effects. Future research should evaluate different fly ash replacement levels and extended exposure durations, as well as additional durability indicators, such as permeability, sorptivity, and mass loss. Field-scale validation is also necessary to confirm long-term performance under actual mining conditions.

DECLARATION OF COMPETING INTERESTS

The authors declare that there are no competing interests regarding the publication of this paper.

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DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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