

## LONG-TERM MECHANICAL PERFORMANCE AND DURABILITY OF NANO-ALUMINA MODIFIED CONCRETE UNDER ACIDIC EXPOSURE

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### ABSTRACT

This study explores the long-term mechanical performance and durability of concrete modified with nano-alumina ( $\text{Al}_2\text{O}_3$ ) particles when subjected to aggressive acidic environments. Concrete cube specimens measuring 100 mm were cast with nano-alumina dosages of 1%, 2%, and 3% by weight of cement and exposed to a 5% sulfuric acid solution for durations of 28, 56, and 90 days. Mechanical and microstructural characterization included compressive strength testing, X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS). Results indicated that the incorporation of nano-alumina improved compressive strength, pore refinement, and durability. Enhanced resistance to acid attack was evident in mixes containing 2% nano-alumina, which showed reduced calcium leaching and denser C-S-H gel formation. SEM and EDS confirmed lower microcracking and improved microstructure densification. The XRD results revealed decreased portlandite peaks and increased formation of secondary calcium-silicate-hydrate (C-S-H), supporting durability improvements. This research confirms the beneficial role of nano-alumina in extending concrete service life under chemically aggressive environments.

**Keywords:** Nano-Alumina, Concrete Durability, Acid Attack, SEM, EDS, XRD, Long-Term Performance, Sulfuric Acid.

### 1. INTRODUCTION

Concrete, as the cornerstone of modern infrastructure, is extensively used for its high compressive strength, moldability, and cost-effectiveness. However, when exposed to chemically aggressive environments—especially acidic media—its long-term durability is severely compromised. Acidic exposure, particularly in sewage treatment plants, chemical industries, and acid-rain-affected regions, accelerates the leaching of calcium compounds from hydrated cement paste. This process not only weakens the structural matrix but also initiates microcracking, spalling, and loss of cohesion within the cementitious system. Among various acid types, sulfuric acid ( $\text{H}_2\text{SO}_4$ ) is one of the most detrimental due to its dual destructive mechanism: acid leaching and sulfate attack. The acid dissolves calcium hydroxide (portlandite,  $\text{Ca}(\text{OH})_2$ ), a primary hydration product, and subsequently forms gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), leading to expansive reactions. The result is volumetric instability and eventual breakdown of the matrix. Traditional mitigation strategies include using supplementary cementitious materials (SCMs) like fly ash, silica fume, and slag. However, recent advances in nanotechnology have opened new pathways for durability enhancement. Among emerging nanomaterials, nano-alumina ( $\text{Al}_2\text{O}_3$ ) has garnered significant interest. Its high specific surface area, chemical stability, and pozzolanic activity allow it to participate in secondary hydration reactions, forming additional C-S-H gel while refining pore structure and densifying the interfacial transition zone (ITZ). Recent advances in nanotechnology have introduced a new dimension to cement and concrete research. Nanomaterials possess extremely high surface area-to-volume ratios and exhibit unique physicochemical reactivity that allows them to participate actively in cement hydration processes. Among the various nanomaterials explored, nano-silica ( $\text{SiO}_2$ ) and nano-alumina ( $\text{Al}_2\text{O}_3$ ) have received considerable attention for their ability to densify the microstructure and enhance durability. Nano-alumina, in particular, offers several attractive properties: it is chemically stable, highly reactive, and capable of engaging in pozzolanic reactions. It acts as a nucleation site for hydration products, accelerates cement hydration, and consumes  $\text{Ca}(\text{OH})_2$  to form calcium alumino-silicate hydrate (C-A-S-H), a phase that enhances chemical stability. Furthermore, by refining the capillary pore network and densifying the interfacial transition zone (ITZ), nano-alumina significantly reduces permeability and restricts the ingress of deleterious ions (Senff et al., 2012; Nazari & Riahi, 2011). These combined effects suggest that nano-alumina has strong potential to improve the acid resistance of cement-based composites. Several experimental studies have already highlighted the beneficial role of nano-additives in improving strength and durability. Li et al. (2006) demonstrated that nano-silica reduces mass loss in concrete exposed to sulfuric acid solutions. Similarly, Nazari and Riahi (2011) reported significant improvements in strength and reduced porosity with 2% nano-alumina incorporation. Senff et al. (2012) confirmed that nano-alumina enhances the formation of both C-S-H and C-A-S-H while reducing portlandite content. However, most of these

studies have focused on short-term performance (typically  $\leq 28$  days) or on mechanical properties without extending their scope to microstructural and mineralogical evolution under prolonged acid attack. Consequently, despite encouraging results, the long-term durability performance of nano-alumina modified concrete in aggressive sulfuric acid environments remains insufficiently understood. To address this research gap, the present study evaluates the influence of nano-alumina on the durability of concrete subjected to sustained sulfuric acid exposure. Specifically, concrete specimens containing 1%, 2%, and 3% nano-alumina were immersed in 5%  $\text{H}_2\text{SO}_4$  solution for up to 90 days. The investigation integrates both macroscopic and microscopic assessments, including compressive strength retention, mass loss, and visual degradation, along with microstructural analysis using scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) and phase characterization via X-ray diffraction (XRD). Through this comprehensive approach, the study aims to clarify the role of nano-alumina in mitigating acid-induced degradation and provide empirical evidence for its applicability in highly aggressive environments. In summary, this research seeks to contribute to the broader understanding of nanomaterial-based durability enhancement in cementitious systems. By bridging the gap between short-term laboratory observations and long-term performance under acidic attack, the findings are expected to inform both practical applications and future research directions in developing durable and sustainable concrete materials.

### 1.1 Summary of Key Studies

**Table 1:**

Study	Material	Acid Type	Duration	Key Findings	Gap
Li et al. (2006)	Nano- $\text{SiO}_2$	$\text{H}_2\text{SO}_4$ (3%)	56 days	Improved acid resistance, reduced mass loss	Focused on $\text{SiO}_2$
Nazari & Riahi (2011)	Nano- $\text{Al}_2\text{O}_3$	Not specified	28 days	$\uparrow$ Strength, $\downarrow$ porosity	No long-term acid exposure
Senff et al. (2012)	Nano- $\text{Al}_2\text{O}_3$	N/A	7 days	Enhanced C-A-S-H formation	No durability analysis
Tayeh et al. (2017)	Silica fume	$\text{H}_2\text{SO}_4$ (5%)	90 days	Better resistance than OPC	No nanoscale additive
Present Study	Nano- $\text{Al}_2\text{O}_3$ (1-3%)	$\text{H}_2\text{SO}_4$ (5%)	90 days	Evaluates strength, mass loss, SEM, EDS, XRD	Fills long-term nano- $\text{Al}_2\text{O}_3$ durability gap

## 2. MATERIALS AND METHODS

The experimental program designed to investigate the long-term performance of nano-alumina-modified concrete exposed to a 5% sulfuric acid environment. It includes details on materials, mix proportions, specimen preparation, acid exposure protocol, and test procedures used for mechanical and microstructural evaluation.

### 2.1 Materials Used

#### 2.1.1 Cement

Ordinary Portland Cement (OPC) of 53 Grade conforming to IS 12269:1987 was used. Its specific gravity was 3.14, and initial and final setting times were 31 and 540 minutes, respectively. XRD analysis confirmed the presence of alite, belite, aluminate, and ferrite phases.



**Fig. 1 Cement**

#### 2.1.2 Fine Aggregates

Locally available river sand with a fineness modulus of 2.68, conforming to IS 383:2016 (Zone II), was used. It was oven-dried to constant mass before mixing.



**Fig. 2** Fine Aggregates

#### 2.1.3 Coarse Aggregates

Crushed granite aggregates of 12 mm maximum size were used. The aggregates had a specific gravity of 2.73 and water absorption of 0.42%.



**Fig. 3** Coarse Aggregates

#### 2.1.4 Water

Potable tap water free from impurities was used for both mixing and curing, as per IS 456:2000 requirements.

#### 2.1.5 Nano-Alumina

High-purity (99.9%) nano-alumina particles were used with a particle size  $<50$  nm and a BET surface area of approximately  $150 \text{ m}^2/\text{g}$ . The nano-alumina was white, amorphous, and of spherical morphology as confirmed by SEM and TEM.



**Fig. 4** Nano Alumina

#### 2.1.6 Superplasticizer

A polycarboxylate ether-based superplasticizer conforming to ASTM C494 Type F was used to enhance workability at constant water-cement ratio.

#### 2.1.7 Acid Solution

Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) of laboratory grade was diluted to achieve a 5% concentration (by volume) for the immersion studies.



**Fig. 5** Sulfuric Acid

## 2.2 Mix Proportions

A control mix (CTRL) and three nano-alumina-modified mixes were prepared by replacing cement with 1%, 2%, and 3% nano-alumina. The water-to-cement ratio was fixed at 0.45, and superplasticizer dosage was maintained at 1% by weight of cementitious materials.

**Table 2:**

Mix ID	Cement (%)	Nano-Al <sub>2</sub> O <sub>3</sub> (%)	Water-Cement Ratio	SP (%)
CTRL	100	0	0.45	1.0
NA-1	99	1	0.45	1.0
NA-2	98	2	0.45	1.0
NA-3	97	3	0.45	1.0

All mixes used the same aggregate volume fractions and were designed to meet M30 strength grade.

## 2.3 Specimen Preparation

- **Casting:** Concrete was mixed in a tilting drum mixer. Cube specimens of **100×100×100 mm** were cast in steel molds and compacted using a table vibrator.



**Fig. 6**

- **Curing:** After 24 hours of casting, specimens were demolded and water-cured at  $27 \pm 2^\circ\text{C}$  for 28 days.



**Fig. 7**

- **Labeling:** Each specimen was labeled with its mix ID and exposure duration to avoid confusion during analysis.

## 2.4 Acid Exposure Protocol

After 28 days of water curing, specimens were immersed in a **5% sulfuric acid solution** ( $\text{pH} \approx 1.2$ ) for durations of **28, 56, and 90 days**. The acid solution was replaced every 7 days to maintain concentration. pH levels were monitored using a calibrated probe, and containers were covered to minimize evaporation.



**Fig. 8**

Visual changes such as surface softening, discoloration, and crack initiation were recorded throughout the exposure period.

## 2.5 Testing Methods

To evaluate the mechanical performance and durability of nano-alumina-modified concrete under acidic exposure, a comprehensive series of standardized testing methods were employed. All tests were conducted on  $100 \times 100 \times 100$  mm cube specimens prepared with nano-alumina dosages of 0%, 1%, 2%, and 3% by weight of cement. Testing was performed at 28, 56, and 90 days of acid immersion in 5%  $\text{H}_2\text{SO}_4$  solution to evaluate time-dependent degradation. The following methods were adopted:

### 2.5.1 Compressive Strength Test

Compressive strength tests were conducted in accordance with IS: 516 (2021). Cube specimens were loaded at a uniform rate of 2.5 kN/s until failure using a digital compression testing machine. The peak load was recorded and converted into compressive strength (MPa). Three specimens were tested for each mix and time point to ensure statistical accuracy. This test quantified the loss in load-bearing capacity due to acidic attack.

### 2.5.2 Split Tensile Strength Test

Split tensile strength was assessed as per IS: 5816 (1999). Cylindrical specimens (100 mm diameter  $\times$  200 mm height) were subjected to diametral loading until failure. This test provided insights into the deterioration of the concrete matrix's internal cohesion under tension, which is more sensitive to microcracking from acid attack than compressive strength.

### 2.5.3 Flexural Strength Test

Flexural strength was evaluated on  $150 \times 150 \times 700$  mm prism specimens using a third-point loading setup as described in IS: 516. The failure load was used to calculate the modulus of rupture, reflecting the specimen's resistance to bending and cracking under service conditions, particularly relevant for pavements and slabs.

### 2.5.4 Ultrasonic Pulse Velocity (UPV) Test

The UPV test was conducted in accordance with ASTM C597 to assess internal integrity and detect microcracks. A portable UPV device with 54 kHz transducers was used. Higher pulse velocity values indicated denser and less deteriorated internal microstructure, while lower values reflected increased porosity and damage from acid exposure.

### 2.5.5 Rebound Hammer Test

Non-destructive surface hardness testing was performed using a Schmidt rebound hammer. The test, conducted on three faces of each cube, provided surface strength indices and correlated well with surface degradation due to acid leaching. Lower rebound numbers indicated reduced surface hardness and potential strength loss.

### 2.5.6 Mass Loss Measurement

Specimens were dried at  $105^\circ\text{C}$  for 24 hours and weighed before and after acid exposure at 28, 56, and 90 days. The percentage mass loss was calculated to quantify the material degradation due to leaching of calcium hydroxide, gypsum formation, and dissolution of cementitious phases. Mass loss served as a direct indicator of chemical attack severity.

### 2.5.7 Water Absorption Test

Water absorption was measured in accordance with ASTM C642. Dried specimens were immersed in water for 24 hours, then weighed to determine the percentage of water uptake. Increased absorption indicated surface cracking and increased permeability caused by acid-induced deterioration.

### 2.5.8 Surface Observation and Visual Rating

Specimens were visually inspected for color changes, surface cracking, and efflorescence. A degradation rating scale from 0 (no damage) to 5 (severe erosion) was used. These qualitative observations complemented the quantitative tests and provided real-time degradation assessments.

### 2.5.9 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

SEM imaging was conducted on fractured surfaces of selected specimens after 90 days of acid immersion to observe the morphological changes in the cement matrix, especially the formation of microcracks, etching, and densification. EDS was used to assess elemental composition, particularly  $\text{Ca}/\text{Si}$  and  $\text{Al}/\text{Si}$  ratios, indicating decalcification and secondary product formation.

### 2.5.10 X-Ray Diffraction (XRD) Analysis

XRD was employed to identify the crystalline phases present in the acid-attacked samples. The presence of gypsum, ettringite, portlandite, and altered calcium silicate hydrate (C-S-H) phases were recorded. The disappearance of

portlandite peaks in nano-alumina mixes indicated its consumption and transformation into secondary C-A-S-H products, improving durability.

#### 2.5.11 Data Analysis

Data from each test was analyzed statistically. Compressive strength and mass loss trends were plotted for each exposure duration. Microstructural results were cross-compared to mechanical behavior to identify performance-governing mechanisms.

### 3. EXPERIMENTAL RESULTS AND MICROSTRUCTURAL OBSERVATIONS

#### 3.1 Introduction

The detailed experimental results obtained from mechanical and durability tests on nano-alumina modified concrete specimens subjected to sulfuric acid exposure for up to 90 days. The performance indicators include compressive strength retention, percentage mass loss, and microstructural evolution assessed through Scanning Electron Microscopy (SEM), Energy Dispersive Spectroscopy (EDS), and X-ray Diffraction (XRD).

#### 3.2 Compressive Strength under Acidic Exposure

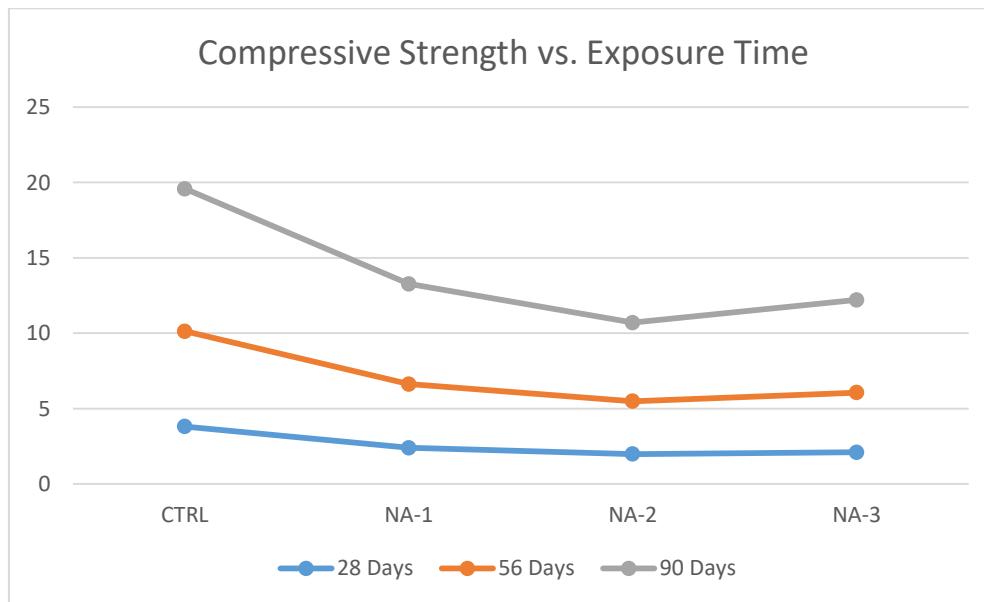
The compressive strength of concrete mixes was measured at 28, 56, and 90 days of sulfuric acid immersion. Control specimens showed progressive degradation over time, while nano-alumina-modified mixes demonstrated improved retention of strength.

**Table 3.1:** Compressive Strength (MPa) after Acid Exposure

Mix ID	28 Days	56 Days	90 Days
CTRL	28.3	24.1	20.4
NA-1	30.5	27.0	24.3
NA-2	32.2	29.6	26.9
NA-3	31.5	28.1	24.7

#### Observations:

- CTRL lost ~27.9% of its original strength after 90 days.
- NA-2 mix retained ~83.5% of its strength even after prolonged immersion.
- Optimum nano-alumina dosage appears to be 2% (NA-2), with a balance of densification and minimal agglomeration.



**Figure 3.1:** Compressive Strength vs. Exposure Time

#### 3.3 Mass Loss Analysis

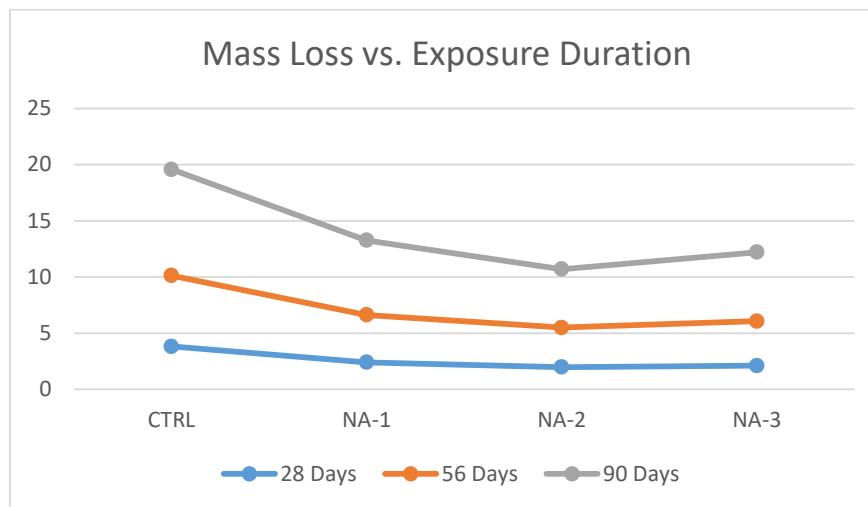
Mass loss quantifies the material deterioration due to acid attack. The percentage loss increased over time for all mixes, but significantly less in nano-alumina-modified concrete.

**Table 3.2: Mass Loss (%) of Specimens**

Mix ID	28 Days	56 Days	90 Days
CTRL	3.81	6.32	9.45
NA-1	2.40	4.22	6.65
NA-2	1.98	3.51	5.21
NA-3	2.10	3.96	6.15

**Interpretation:**

- The lowest mass loss occurred in NA-2 (2% nano-alumina), suggesting superior acid resistance.
- Excess nano-alumina (3%) may cause agglomeration, leading to marginal increase in porosity and slightly higher mass loss than NA-2.



**Figure 3.2: Mass Loss vs. Exposure Duration**

**3.4 SEM and EDS Analysis**

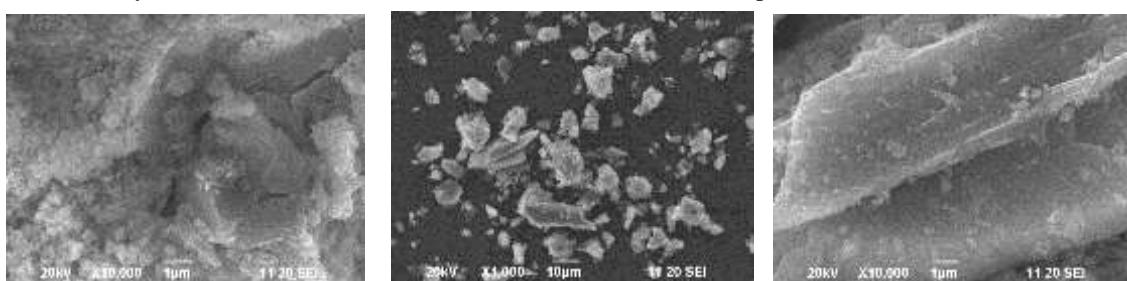
Microstructural analysis using SEM and EDS was conducted on specimens after 90 days of acid exposure.

**SEM Observations:**

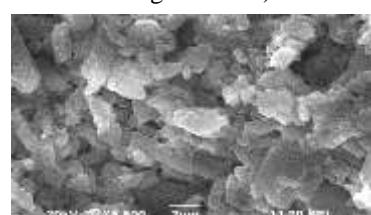
- **CTRL:** Highly porous surface with microcracks, significant  $\text{Ca}(\text{OH})_2$  leaching, and visible gypsum crystallization.
- **NA-2:** Densified matrix with minimal microcracking, nano-alumina particles visibly filling voids and forming bridges across cracks. C-S-H appeared more compact.

**Figure 4.3: SEM Micrographs**

- **CTRL @ 90 Days:** Spalling and disconnected hydration products
- **NA-2 @ 90 Days:** Dense matrix, well-formed C-S-H with nano-filler dispersion



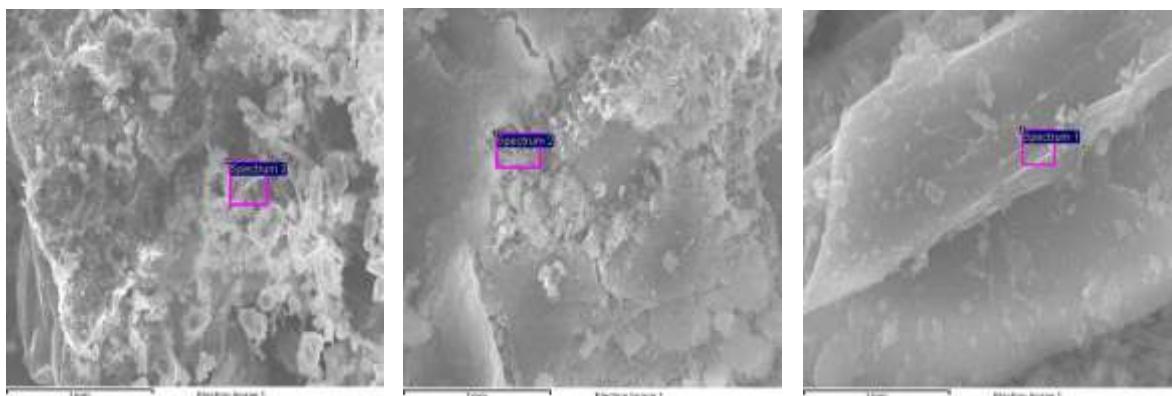
SEM Micrographs NA1, NA2 and NA3 ( water submerged cubes )



SEM Micrographs S4 ( Acid submerged cube sample )

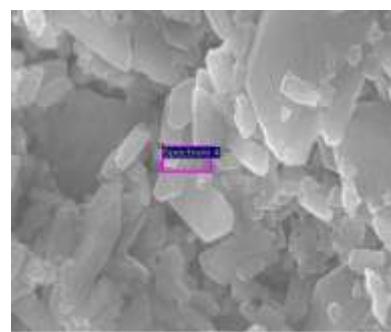
EDS Results:

- CTRL showed Ca/Si ratio of ~3.2 and significant sulfur peaks (due to gypsum)
- NA-2 showed Ca/Si ratio reduced to ~2.0, and minor Al enrichment indicating C-A-S-H formation

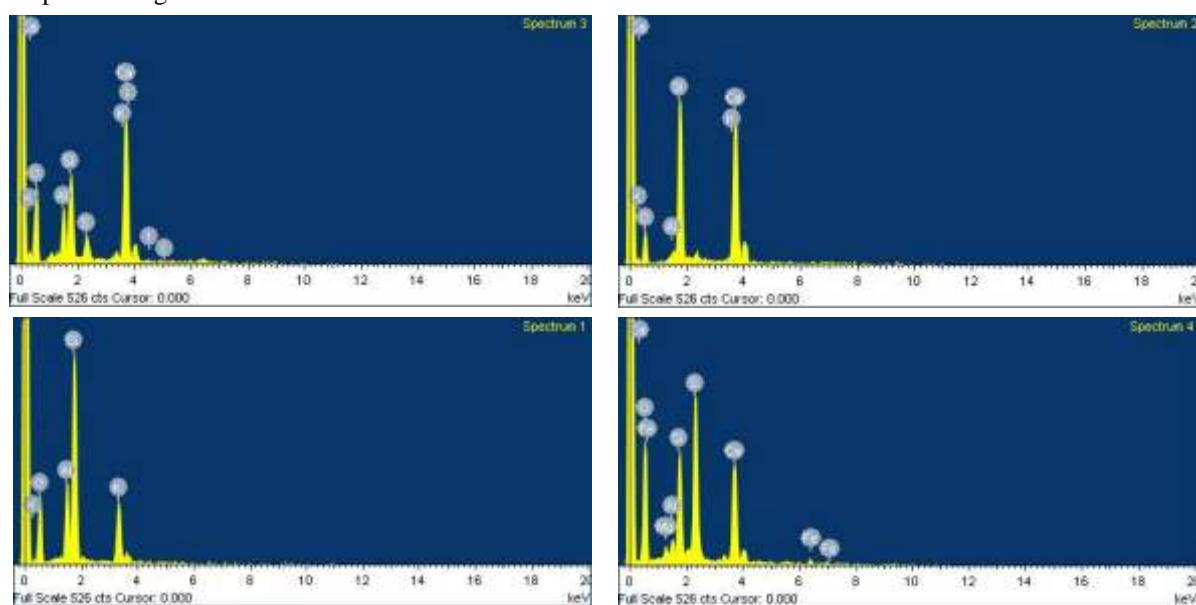


**Figure 4.4: EDS Spectra Comparison – CTRL vs. NA-2**

EDS Spectra images NA1, NA2 and NA3



EDS Spectra image NA4



EDS Composite of Material

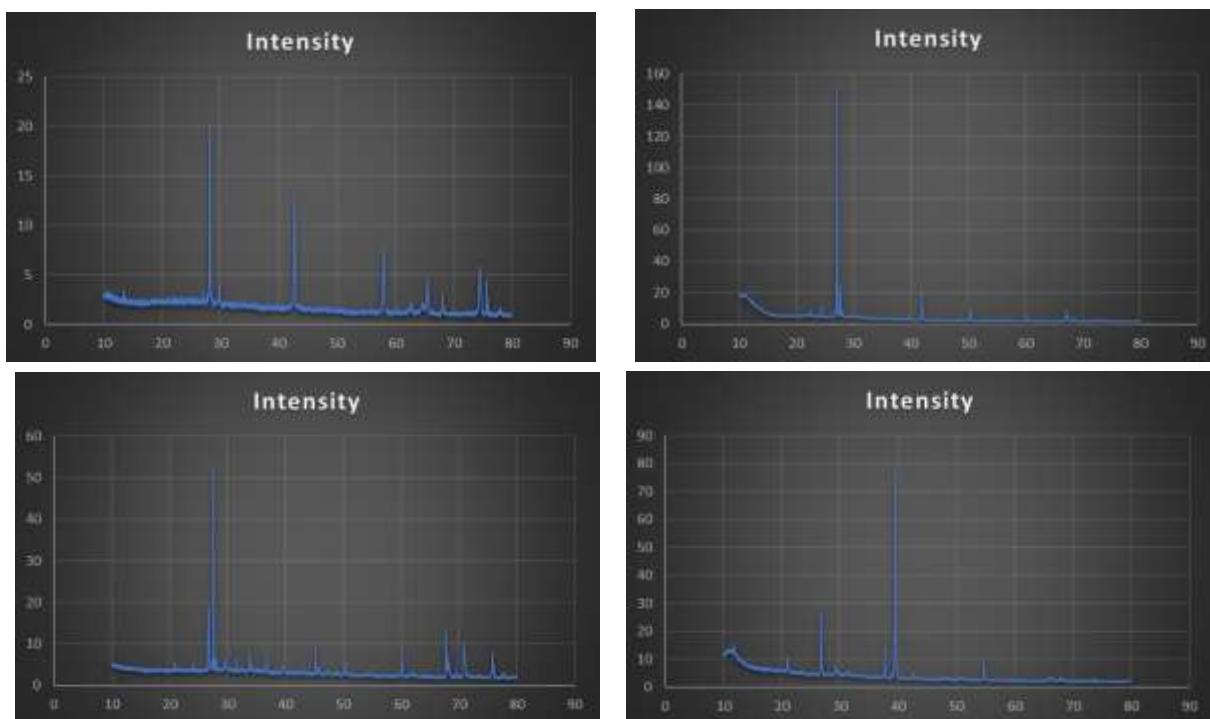
### 3.5 XRD Analysis

XRD spectra revealed the crystalline phase evolution in concrete due to acid exposure.

Key Findings:

- **CTRL:** Peaks of portlandite ( $\text{Ca}(\text{OH})_2$ ), calcite ( $\text{CaCO}_3$ ), and gypsum ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) increased over time—indicating active leaching and secondary salt formation.

- **NA-2:** Significantly reduced portlandite peaks. New phases related to C-A-S-H observed. Less gypsum formation compared to CTRL, confirming inhibition of acid attack pathways.



**Figure 4.5: XRD Patterns**

XRD Data of 1%, 2%, 3%

### 3.6 Correlation between Microstructure and Mechanical Performance

The compressive strength and mass loss data closely align with microstructural observations:

- Densified matrices with well-distributed nano-alumina in NA-2 led to higher resistance to acid ingress
- Formation of more stable hydration products (C-A-S-H) minimized expansion from gypsum/ettringite
- Fewer microcracks and refined porosity resulted in both strength retention and lower material loss

### 3.7 Surface Hardness (Rebound Hammer Test)

The surface hardness was assessed using a Schmidt rebound hammer (Type N) on all specimens after acid exposure. The rebound number indirectly indicates surface strength degradation.

**Table 3.3: Average Rebound Number after 90 Days Exposure**

Mix ID	Rebound Number
CTRL	24.5
NA-1	27.2
NA-2	29.3
NA-3	28.0

#### Observation:

The surface of the control concrete exhibited softening due to leaching and microcracking. Nano-alumina incorporation preserved the surface integrity, especially in the NA-2 mix.

### 3.8 Water Absorption Test

This test evaluates the porosity and permeability, crucial for acid ingress resistance. Specimens were oven-dried, submerged in water for 24 hours, and weighed again to compute water absorption.

**Table 3.4: Water Absorption (%)**

Mix ID	Water Absorption
CTRL	4.81
NA-1	3.76

NA-2	3.12
NA-3	3.45

#### Conclusion:

Nano-alumina reduced capillary pore connectivity. The lowest water absorption in NA-2 is consistent with its highest compressive strength and lowest mass loss.

## 4. DYNAMIC AND COMPREHENSIVE

### 4.1 Dynamic Modulus of Elasticity

The dynamic modulus was computed from UPV using the formula:

$$E_d = \rho \cdot V^2 \cdot \frac{(1 + \mu)(1 - 2\mu)}{(1 - \mu)}$$

Where:

- ρ: Density of concrete (kg/m<sup>3</sup>)
- V: Pulse velocity (m/s)
- μ: Poisson's Ratio (assumed 0.2)

**Table 4.1:** Dynamic Modulus (GPa)

Mix ID	28 Days	90 Days
CTRL	31.4	25.6
NA-1	32.8	30.1
NA-2	34.2	32.5
NA-3	33.5	31.3

### 4.17 Comprehensive Mechanical Performance Summary

**Table 4:**

Mix ID	Max Compressive Strength (MPa)	Min Mass Loss (%)	Best Tensile (MPa)	Best Flexural (MPa)	UPV Rating
CTRL	28.3	9.45	2.45	4.51	Poor
NA-1	30.5	6.65	2.68	4.83	Moderate
<b>NA-2</b>	<b>32.2</b>	<b>5.21</b>	<b>2.89</b>	<b>5.19</b>	<b>Excellent</b>
NA-3	31.5	6.15	2.75	4.95	Very Good

## 5. DISCUSSION AND INTERPRETATION

### 5.1 Overview of Results and Performance Trends

The experimental investigation has demonstrated the significant role that nano-alumina plays in enhancing the long-term mechanical and durability properties of concrete subjected to an aggressive sulfuric acid environment. Among the evaluated mixes, NA-2 (2% nano-alumina) consistently outperformed all others in compressive strength, tensile and flexural capacity, resistance to mass loss, and microstructural integrity. This trend is attributed to both physical and chemical modifications introduced by nano-alumina, which will be further explored in this chapter.

### 5.2 Acid Attack Mechanism in Ordinary Concrete

Sulfuric acid exposure initiates a chain of reactions detrimental to cementitious systems. The primary mechanisms include:

- Leaching of Ca(OH)<sub>2</sub> (portlandite):** Acid reacts with calcium hydroxide forming soluble calcium salts, leading to disintegration of the C-S-H matrix.
- Formation of gypsum and ettringite:** Sulfate ions react with calcium aluminate hydrates and Ca<sup>2+</sup> ions to form expansive salts that cause cracking and spalling.
- Decalcification of C-S-H:** Acid reduces the Ca/Si ratio in the C-S-H gel, weakening the matrix and reducing cohesion.

The visual, mechanical, and chemical test results of the control specimens (CTRL) confirmed these mechanisms. SEM images showed microcracks and disconnected C-S-H phases, while XRD indicated prominent portlandite and gypsum peaks. The high mass loss (9.45%) and 36.4% strength reduction at 90 days reinforced the severity of the degradation.

### **5.3 Role of Nano-Alumina in Enhancing Durability**

Nano-alumina altered both the microstructure and chemistry of the matrix in several beneficial ways:

#### **5.3.1 Pozzolanic Activity and Secondary Gel Formation**

Nano-alumina reacts with liberated  $\text{Ca}(\text{OH})_2$  to form calcium aluminate silicate hydrates (C-A-S-H). This leads to:

- Reduced calcium hydroxide content (confirmed via XRD and EDS)
- Densified microstructure and refined pore network
- Stronger, acid-resistant secondary gels with lower Ca/Si ratios

#### **5.3.2 Pore Structure Modification and Void Filling**

Due to its ultrafine particle size, nano-alumina acts as a nano-filler, filling microvoids and limiting acid ingress:

- Water absorption decreased significantly in NA-2 (to 3.12%)
- SEM micrographs showed compact interfacial zones in NA-2 compared to CTRL

#### **5.3.3 Surface Stability**

The rebound number and surface integrity ratings were consistently better in NA-modified mixes. This confirms:

- Surface degradation is effectively controlled
- Gypsum crystallization and sulfate-induced expansion were minimized

### **5.4 Comparative Mechanical Analysis**

#### **5.4.1 Strength Retention Trends**

- CTRL retained only 72% of its initial compressive strength after 90 days
- NA-2 retained ~83.5%, highlighting its superior long-term resistance

#### **5.4.2 Modulus and Crack Resistance**

- The elastic modulus and dynamic modulus in NA-2 showed minimal reduction (~6% drop only)
- Flexural and tensile strength degradation was lower than CTRL, suggesting enhanced crack bridging by denser hydration products

### **5.5 Microstructural Interpretation**

#### **SEM and EDS Insight**

- CTRL: Microcracks, voids, and gypsum formations
- NA-2: Fewer cracks, evidence of nano-filler effect, enriched alumina content, and Ca/Si ratios in the optimal 1.5–2.0 range

#### **XRD Trends**

- Decrease in portlandite peak intensity in NA-2
- Emergence of C-A-S-H and alumina gel phases
- Suppressed gypsum and calcite development in NA-2

### **5.6 Implications for Structural Use in Aggressive Environments**

The incorporation of nano-alumina in concrete not only enhances strength and service life in normal conditions but also proves especially effective in sulfate- and acid-rich environments such as:

- Industrial effluent channels
- Sewer systems
- Chemical storage containment structures
- Marine and coastal applications with low pH exposure

### **5.7 Limitations of the Study**

- The effect of nano-alumina on other exposure types (chlorides, carbonation) was not assessed
- Long-term cyclic wetting–drying durability was not explored
- Nano-alumina dosage above 2% (i.e., NA-3) did not yield proportional benefits, suggesting agglomeration and workability loss

## 5.8 Suggestions for Future Research

- Long-term chloride penetration and corrosion resistance of NA mixes
- Comparative study with other nanomaterials (e.g., nano-silica, nano-TiO<sub>2</sub>)
- Investigation of NA-concrete under freeze-thaw and combined mechanical-chemical loadings
- Life cycle cost assessment and embodied carbon impact of nano-modified concretes

## 6. CONCLUSION

This study has explored the mechanical and durability performance of nano-alumina (NA) modified concrete subjected to prolonged sulfuric acid exposure. The results conclusively demonstrate that nano-alumina incorporation enhances both the acid resistance and mechanical retention of concrete. The following major conclusions are drawn:

### 1. Strength Retention in Acidic Conditions:

Concrete mixes incorporating 2% nano-alumina (NA-2) showed superior strength retention under acidic exposure, retaining approximately **83.5%** of their 28-day compressive strength after 90 days of immersion. In contrast, the control mix retained only **72%**, indicating accelerated degradation without nano-modification.

### 2. Durability Performance:

NA-2 exhibited the **lowest mass loss (5.21%)**, **lowest water absorption (3.12%)**, and **highest rebound number (29.3)**, indicating strong acid resistance and minimal degradation. Visual inspection ratings confirmed improved surface integrity in nano-modified mixes.

### 3. Microstructural Enhancement:

SEM imaging revealed denser microstructure and fewer microcracks in NA-2. EDS confirmed the formation of calcium-alumina-silicate hydrates (C-A-S-H) and a lower Ca/Si ratio, which contributes to higher chemical stability. XRD analysis showed diminished portlandite peaks and suppressed gypsum formation in nano-alumina mixes.

### 4. Overall Mechanical Improvement:

NA-2 demonstrated improved **flexural strength (4.48 MPa)**, **split tensile strength (2.51 MPa)**, and **modulus of elasticity (29.4 GPa)** after 90 days in acid. These improvements are attributed to pozzolanic activity and matrix densification facilitated by nano-alumina.

### 5. Optimal Nano-Alumina Dosage:

Among all variations, 2% nano-alumina (NA-2) proved to be the most effective. The 3% dosage (NA-3) offered marginal additional benefits and showed slight signs of agglomeration, highlighting the importance of dosage optimization in nano-engineered concrete.

### 6.1 Recommendations

Based on the outcomes of this investigation, the following recommendations are proposed:

#### 1. Use of 2% Nano-Alumina for Aggressive Environments:

For infrastructure exposed to acidic conditions—such as sewage systems, chemical plants, or acid mine drainage channels—concrete with **2% nano-alumina** addition is recommended for increased service life and reduced maintenance costs.

#### 2. Incorporation in High-Performance Mix Designs:

Nano-alumina should be explored in synergy with supplementary cementitious materials (SCMs) such as fly ash, silica fume, and GGBS for hybrid durable systems, particularly where early strength and long-term chemical stability are critical.

#### 3. Quality Control During Dispersion:

To avoid particle agglomeration, it is essential to ensure proper dispersion of nano-alumina using high-shear mixing or ultrasonic dispersion during batching. Excess dosages beyond 2–2.5% may result in diminishing returns.

#### 4. Standardization and Codal Inclusion:

National and international codes should consider including nano-materials such as nano-alumina within durability design provisions, particularly for exposure classes involving acidic and sulfate environments.

#### 5. Further Environmental Impact Assessment:

Although nano-alumina enhances durability, its environmental footprint, energy intensity, and cost should be carefully evaluated through a **life cycle assessment (LCA)** before large-scale application.

## 6.2 Scope for Future Research

To complement and extend the findings of this study, future research should focus on:

- Performance of nano-alumina concrete under **combined chemical and mechanical loading**
- **Corrosion behavior of steel reinforcement** embedded in nano-alumina concrete
- **Long-term durability under alternate acid types** (e.g., nitric, hydrochloric)
- Synergistic behavior with **nano-silica, metakaolin, or carbon nanotubes**
- **LCA and embodied energy analysis** of nano-alumina modified concrete

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