The Durability of Concrete Mortars with Different Mineral Additives Exposed to Sulfate Attack

For several years, extensive research investigations have been conducted examining the effects of acids commonly encountered by industrial facilities in manufacturing environments. Numerous studies have been conducted to examine the durability of concrete containing various chemical additives and fine metals when exposed to various acid solutions, as well as the preventive steps taken to avoid the deterioration of concrete associated with these acids. This research includes an examination of enhancing the effectiveness and function of concrete when exposed to sulfuric acid. It explores the use of waterproofing (WP) and complementary cementitious materials (SCMs), including silica fume Nano silica and fly ash, as well as a water-reducing additive. Cube-shaped samples measuring 100 x 100 x 100 mm were prepared and completely immersed in 2.5 % dilute sulfuric acid solution for 90 and 180 days. Compressive strength, tensile strength, and absorption tests were performed after 28 days, as well as after immersion in a 2.5 % dilute acid solution for 90 and 180 days. The results revealed that after 90 days, there was a 31 % reduction in compressive strength for mixtures with 25 % FA and 5 % SF , and a 46 % decrease for mixtures containing WP , when compared to their corresponding results at the 28 day age under standard conditions. Mineral admixtures significantly reduce absorption rates. After 90 days, WP had 3 % absorption during acid exposure, and after 180 days, the 25 % FA and 5 % SF mixture had 2.3 % absorption. This results from reduced permeable voids due to decreased capillary pores, enhancing concrete durability. The findings also indicated that the impact of exposure to acid on the strength characteristics of concrete becomes more pronounced with prolonged exposure. In addition, the inclusion of NS, SF , and FA in cement concrete results in the development of a unique material that can meet the growing need for construction materials. Furthermore, this technique delivers economic and environmental benefits by minimizing pollution caused by waste products such as FA and SF , which are a residual by-products of thermal power plants and ferrosilicon production respectively.

Keywords: Supplementary Cementitious Materials; Water Proof; Silica Fume; Fly Ash; Nano Silica; Absorption; Splitting Tensile Strength; Compressive Strength.
The use of fly ash as a supplementary cementitious material (SCM) in concrete is widely recognized for its ability to improve the strength and durability of concrete. Although the strength and stability of these concrete structures have proven their durability over time, there are current apprehensions regarding their long-term sustainability. This is primarily due to the increasing chemical aggressiveness of the environment. Environmental risks encompass potential damage from abrasion, chemical attacks, alkalinity, carbonation, freezing and thawing, as well as the impact of weathering. The use of fly ash and blast furnace slag as supplemental cementing materials (SCMs) in Portland cement concrete to increase durability is backed by a large body of research. These additives have been shown to improve specific properties, such as sulfate resistance, thereby contributing to the overall longevity of the concrete.

While the inclusion of supplementary cementing materials (SCMs) may assist in preventing or delaying sulfate attack, the same cannot be said for physical sulfate attack. Nehdi et al. conducted a study on concrete samples partially immersed in water. The submerged parts were subjected to traditional chemical sulfate attack, whilst mirabilite crystallization occurred above the water level, causing further damage. Pozzolanc-containing concrete compositions, such as fly ash, metakaolin, and silica fume, were more vulnerable to physical attack than pozzolan-free samples. This was due to an increase in the prevalence of smaller-sized pores, which resulted in higher capillary suction and a larger surface area for drying. These findings are consistent with those of a comparable study conducted by Iraressar et al. There has been a discussion regarding the utilization of a highly concentrated solution, as addressed by Bellman. The use of extremely concentrated liquids typically hastens the attack process. However, Yu et al. discovered that the depth of sulfate penetration in mortar samples is unaffected by sulfate concentration.

The demanding environment necessitated strong resistance against chemical damage. It is widely acknowledged that ordinary Portland cement (OPC) concrete lacks significant resistance to acids due to its chemical composition and hydration byproducts. The addition of pozzolanic elements such as rice husk ash, fly ash, and silica fume improves concrete’s resistance to sulfate attack by increasing both strength and weight loss. This enhancement can be due to concrete specimen porosity reduction and the development of ettringite. (9) The use of fly ash as a supplementary cementitious material (SCM) in concrete is widely recognized for its cost savings and improved performance. It has benefits such as improved workability and durability. The initial strength of concrete with fly ash is considerably lower compared to concrete without fly ash due to the slower pozzolanic reaction of fly ash. Observations revealed that during the initial stages of the corrosion process, the mass of concrete increased under all conditions. However, in the case of higher temperature acid solutions, a more significant long-term mass loss was observed. In general, the presence of sulfuric acid solution during the degradation process leads to an acceleration of mass loss. This acceleration is primarily attributed to the high concentration of solutions, as addressed by Bellman. Exploring the use of impermeabilizing (WP) and materials cementosos complementarios (SCM), including humo de silice Nano silice and cenizas volantes, as well as an aditivo reductor de agua. Se prepararon muestras en forma de cubo de 100 x 100 x 100 mm y se sumergieron completamente en una solución de ácido sulfúrico diluido al 2,5 % durante 90 y 180 días. Se realizaron ensayos de resistencia a la compresión, resistencia a la tracción y absorción después de 28 días, así como después de la inmersión en una solución ácida diluida al 2,5 % durante 90 y 180 días. Los resultados revelaron que, después de 90 días, se produjo una reducción del 31 % en la resistencia a la compresión de las mezclas con un 25 % de AF y un 5 % de SF, y una disminución del 46 % en las mezclas que contenían WP, en comparación con sus resultados correspondientes a los 28 días en condiciones estándar. Los aditivos minerales reducen significativamente los índices de absorción. Después de 90 días, el WP tenía una absorción del 3 % durante la exposición al ácido, y después de 180 días, la mezcla de 25 % FA y 5 % SF tenía una absorción del 2,3 %. Esto es el resultado de la reducción de los huecos permeables debido a la disminución de los poros capilares, lo que mejora la durabilidad del hormigón. Los resultados también indicaron que el impacto de la exposición al ácido en las características de resistencia del hormigón se hace más pronunciado con la exposición prolongada. Además, la inclusión de NS, SF y FA en el hormigón de cemento da lugar al desarrollo de un material único que puede satisfacer la creciente necesidad de materiales de construcción. Además, esta técnica aporta beneficios económicos y medioambientales al minimizar la contaminación causada por productos de desecho como el AF y el SF, que son subproductos residuales de las centrales térmicas y de la producción de ferrosilicio, respectivamente.

Palabras clave: Materiales Cementosos Suplementarios; Impermeabilidad; Humo de Silice; Cenizas Volantes; Nano Silice; Absorción; Resistencia a la Tracción por División; Resistencia a la Compresión.
increase in temperature, which weakens the bonds between aggregates and cement paste. As a consequence, there is a loss of aggregates and an eventual increase in the depth of corrosion.\textsuperscript{(11)} H. Li et al.\textsuperscript{(12)} investigated the mechanical characteristics of cement mortars with nano-\text{Fe}_2\text{O}_3 and nano-SiO\textsubscript{2}. Their experiments revealed that mortars with these nanoparticles exhibited enhanced compressive and flexural strength. Notably, increasing the dosage of nano-SiO\textsubscript{2} led to a stronger effect on mortar strength compared to silica fume. Examination through scanning electron microscopy (SEM) confirmed that both nano-\text{Fe}_2\text{O}_3 and nano-SiO\textsubscript{2} particles filled the pores and reduced the presence of Ca(OH)\textsubscript{2} in the hydration products, contributing to the improved mechanical properties of mortars with nanoparticles. Additionally, G. Li\textsuperscript{(13)} carried out a study in the lab using high-volume, high-strength concrete that contained nano-SiO\textsubscript{2}. The research revealed that the addition of nano-SiO\textsubscript{2} significantly enhanced the pozzolanic activity of fly ash during the hydration process. This improvement increased the early age and final strength of the high-volume fly ash concrete. When compared to plain concrete, the concrete created with nano-SiO\textsubscript{2} had a stunning 3-day strength increase of up to 81 \%, and its 2-year strength reached 115,9 MPa, exceeding the strength of reference Portland cement concrete, which was 103,7 MPa.

The goal of this experimental investigation is to examine the impact of water-resistant materials and extra cementitious materials on the durability of concrete when subjected to sulfuric acid. Determining the impact of various chemical and fine mineral additives on the strength and durability of concrete when exposed to a 2,5 \% diluted sulfuric acid solution for varying times (90 and 180 days) is the major objective of the research. The study aims to evaluate the compressive strength, splitting tensile strength and absorption of concrete samples treated with different mixtures, including water-resistant materials (WP), silica fume, fly ash, Nano silica and a water-reducing additive. The research seeks to identify the mixture composition that provides the highest resistance against concrete deterioration caused by sulfuric acid exposure. The findings indicate the changes in strength over time and compare the performance of different mixtures, ultimately contributing to the understanding of concrete durability in acidic environments. Furthermore, the ongoing research examines how specimens with various types of additives perform after 28 days and compares these findings with the results obtained at 90 and 180 days.

Experimental Program

Materials

This research utilized sulfate resisting Portland cement (SRPC) as shown in Table 1 \textsuperscript{(14)}, which met the ASTM Type (v) standards.\textsuperscript{(15)} The mass cement company supplied the SRPC, which had a specific gravity of 93,316 cm\textsuperscript{2}/gm. For partial replacement of cement, Class (F) fly ash (FA) (table 2) \textsuperscript{(16)}, silica fume (SF) (table 3) \textsuperscript{(17)}, Nano Silica (NS) (table 4) \textsuperscript{(18)} and water reducing agent (wp) (table 5) were used based on their weight. The coarse aggregate had a maximum size of 12,5 mm, and the fine aggregate was river sand. A third-generation superplasticizer, specifically Sika Visco Crete - 180 GS \textsuperscript{(19)}, containing polycarboxylate polymers, was added as shown in Table 6. In addition, a dilute sulfuric acid solution with a 2,5 \% concentration and a pH of 3,8 was employed.

| Table 1. The chemical and physical compositions of used SRPC \textsuperscript{(14)} |
|-----------------|-----------------|
| Chemical analysis | SRC |
| Silicon dioxide (SiO\textsubscript{2}) | 20,9 |
| Aluminum oxide (Al\textsubscript{2}O\textsubscript{3}) | 4,22 |
| Ferric oxide (Fe\textsubscript{2}O\textsubscript{3}) | 4,94 |
| Calcium oxide (CaO) | 62,77 |
| Magnesium oxide (MgO) | 2,8 |
| Sulfur trioxide (SO\textsubscript{3}) | 2,53 |
| Total | 99,7 |
| C\textsubscript{3}S | 54,00 |
| C\textsubscript{3}A | 19,20 |
| C\textsubscript{2}A | 2,83 |
| C\textsubscript{4}AF | 15,02 |
| Free lime | 1,00 |
| Loss on ignition | 1,5 |
| Physical properties | |
| Specific gravity | 3,08 |
| Compressive strength (MPa) | 28,21 |

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Mix Proportions

A total of thirteen concrete mixes were prepared, each containing different proportions of cement, water-reducing agent (WP), fly ash (FA), and silica fume (SF), and Nano Silica (NS). The preparation of all mixes followed the guidelines provided by ACI 211.1-19. Previous studies on SCMs and their optimal ratios to improve concrete characteristics recommends up to 25% fly ash and 15% silica fume substitution. The specific details of mix proportions can be found in Table 7. The water-to-binder ratio was maintained at a constant value of 0.30 for all mixes. High-range water reducer (HRWR) was employed to regulate the workability of the concrete. The water demand in the mixes increased as the fineness of dry additives increased. The primary target for the control mix was to obtain a compressive strength of 35 MPa after 28 days.

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Table 7. Proportions of tested specimens mixed.

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Description</th>
<th>Cement (kg)</th>
<th>Sand (kg)</th>
<th>Gravel (kg)</th>
<th>Water (kg)</th>
<th>w/b</th>
<th>WP</th>
<th>SF</th>
<th>FA</th>
<th>NS</th>
<th>HRW (%)</th>
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<tr>
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<td>460</td>
<td>750</td>
<td>875</td>
<td>216</td>
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<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
<td>/</td>
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<tr>
<td>M5</td>
<td>WP2</td>
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<td>750</td>
<td>875</td>
<td>216</td>
<td>0,47</td>
<td>9,2</td>
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<td>/</td>
<td>/</td>
<td>/</td>
</tr>
<tr>
<td>M1-1</td>
<td>FA15SF5</td>
<td>368</td>
<td>750</td>
<td>875</td>
<td>138</td>
<td>0,3</td>
<td>/</td>
<td>23</td>
<td>69</td>
<td>/</td>
<td>1</td>
</tr>
<tr>
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<td>345</td>
<td>750</td>
<td>875</td>
<td>138</td>
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<td>/</td>
<td>46</td>
<td>69</td>
<td>/</td>
<td>1</td>
</tr>
<tr>
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<td>322</td>
<td>750</td>
<td>875</td>
<td>161</td>
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<td>/</td>
<td>69</td>
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<td>/</td>
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<tr>
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<tr>
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<td>750</td>
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<td>46</td>
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<tr>
<td>M2-3</td>
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<td>875</td>
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<td>0,35</td>
<td>/</td>
<td>46</td>
<td>69</td>
<td>0,92</td>
<td>1</td>
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</tbody>
</table>

*CM-control mix, WP2- Mix containing 2 % waterproof, FA15SF5- Mix containing 15 %FA and 5 %SF, FA15SF10- Mix containing 15 %FA and 10 %SF, FA20SF5- Mix containing 20 %FA and 5 %SF, FA20SF10- Mix containing 20 %FA and 10 %SF, FA15SF5NS0,2- Mix containing 15 %FA, 5 %SF, and 0,2 NS and FA15SF5NS0,5- Mix containing 15 %FA, 5 %SF, and 0,5 NS.

Specimen Preparation
The components were first weighed and prepared, then a high-range water reducer (HRWR) was added to the water, which was then set aside. The coarse aggregate and silica fume were then combined for 5 minutes. After pausing the mixing process, cement, sand, fly ash, and HRWR-infused water were added. To guarantee a homogenous mixture, the mixer was turned on for 10 minutes. Additionally, in the case of the second group where nano-silica was introduced into the mixture, a distinct mixing procedure was employed. The items were painstakingly measured and prepared at first. Following that, the superplasticizer was added to the mix. The coarse and fine aggregates, as well as the nano-silica, were then mixed during a five-minute mixing period. The resulting mixture was then poured into the moulds of 10 cm cubes and vibrated on a vibrating table in two layers. The moulds were opened after 24 hours at room temperature, and the samples were placed in a curing tank for 28 days. The samples were then subjected to a dilute sulfuric acid solution with a concentration of 2,5 % for 90 and 180 days. The same mixing process was used for all mixes to maintain consistency and efficiency, with the goal of achieving uniformity and homogeneity.

Testing Methods
Once the initial 28-day curing period elapsed, compressive strength test, splitting tensile strength, and absorption test were conducted on a group of specimens under normal conditions and age of 28 days. Other specimens were placed in dilute acid with a concentration of 2,5 % and a PH of 3,8 for a period of 90-day and 180-day. After the expiration of a period, the specimens were extracted from the acid solution. They underwent a thorough water washing procedure to prepare them for a battery of tests, encompassing assessments of compressive strength, splitting tensile strength, and absorption at 90 and 180-days. To bolster the credibility of the results, three specimens were employed for each test, contributing to the overall reliability of the data. The average values of the recorded measurements were meticulously recorded. The subsequent sections offer an in-depth exploration of these aforementioned tests.

Compressive Strength
Following a curing period of 28 days, the cube specimens were submerged in a 2,5 % concentration sulphate acid (H₂SO₄) solution with a pH value of 3,8 for durations of 90 and 180 days. The acid solution was prepared by combining concentrated acids with a predetermined quantity of distilled water, ensuring the desired concentration was achieved by accurately measuring the amount of water needed. After the immersion period in the acid solution, the samples were removed, washed, and dried using a towel to eliminate any loose particles. Subsequently, the samples underwent the compressive strength test after 28, 90, and 180 days of curing period. The compressive strength test was carried out following the guidelines outlined in British standards BS. Part 3, 2002[21]
Splitting Tensile Strength

After a curing period of 28 days, the initial batch of cube specimens underwent a splitting tensile test. At the same time, another set of cube specimens were placed in a solution of 2.5 % sulfuric acid ($H_2SO_4$) with a pH of 3.8 for durations of 90 and 180 days. Once the immersion periods were completed, the samples were removed from the acid solution, rinsed, and dried using a towel to remove any loose particles. Subsequently, the splitting tensile strength test was conducted on these samples as well. The guidelines specified in British standards BS. Part 6, 2009 (22) were followed for the performance of the splitting tensile strength test.

Absorption Test

In accordance with the standard ASTM C642-13, (23) the absorption test was performed on (100*100*100) mm cubic specimens. This test was performed after a 28-day curing period and a 90-day exposure period to sulfuric acid at both doses.

RESULTS AND DISCUSSION

Compressive Strength Results

The compressive strength test results for all samples are depicted in figure 1, where each value represents the average of three samples. Under standard conditions (at 28 days), the compressive strength values are as follows: 39 % for the mixture containing (15 % FA and 15 % SF), (50 %) for mixtures containing (20 % FA and 15 % SF), 92 % for mixtures containing (25 % FA and 15 % SF), and 68 % for mixtures containing (FA, SF, and NS). In contrast, the mixture containing WP exhibited a (25,3 %) decrease in strength at the 28-day curing period. At the 90-day exposure period, and under the influence of severe conditions, the compressive strength values exhibited a decline when compared to their corresponding values at 28 days. Specifically, there was a reduction of 16 % for the mixture containing WP, 12 % for the mixture comprising 20 % FA and 10 % SF, 9,6 % for the mixture with 25 % FA and 5 % SF, and 9,6 % for the mixture containing FA, SF, and NS, respectively. Moving to the 180-day exposure period, the decrease in compressive strength percentages were more pronounced. The mixture containing WP experienced a substantial decrease of 38 %, while the mixture consisting of 15 % FA and 10 % SF exhibited a reduction of 28,5 %. Additionally, the mixture incorporating 25 % FA and 15 % SF showed a decrease of 13,6 % in compressive strength. This boost in compressive strength for these mixtures compared to the reference mixture can be attributed to several factors, including the dispersion of cement agglomerates into individual particles, the high pozzolanic activity of silica fume and fly ash, the presence of Nano-SiO$_2$ particles that both fill the pores and reduce the Ca(OH)$_2$ content within the hydration products, along with substantial water reduction due to the use of HRWR.

It's noteworthy that the influence of a 2.5 % sulfuric acid solution on compressive strength at 90 days is relatively minimal. The reduction percentages are as follows: (9 %) for mixtures containing (20 % FA and 15 % SF), (5 %) for mixtures containing (20 % FA and 15 % SF), and (8 %) for mixtures containing (FA, SF, and NS).
SF), (4.5 %) for mixtures containing (25 % FA and 15 % SF), and (6.9 %) for mixtures containing (15 % FA, 10 % SF, and 0.2 % NS), when compared to their respective compressive strengths at the 28-day mark under normal conditions. At the 180-day age, using the same concentration of sulfuric acid, the reduction percentages are 13.6 % for mixtures containing (25 % FA and 15 % SF), and 15 % for mixtures containing (FA, SF, and NS), compared to their compressive strengths at the 28-day mark under normal conditions.

This observation is consistent with G. Li’s findings in his study (13), which revealed that the pozzolanic activity of fly ash may be greatly increased by including nano-SiO\(_2\). The researchers concluded that using nano-SiO\(_2\) increases the early-age and final strength of fly ash concrete. Furthermore, the effective dispersion of cement particles within the mixtures and a significant reduction in permeability, which inhibits the entrance of a strong acid solution into the concrete, might be linked to this improvement.

The inclusion of pozzolanic materials is known to enhance concrete’s resistance to acid attacks, a fact supported by prior studies. The use of pozzolanic minerals in cement mortar, for instance, has been shown to significantly improve its durability when subjected to chemical sulphate attacks. (24, 25) This improvement is due to the reduction in porosity and the consumption of calcium hydroxide, making the concrete less susceptible to chemical sulphate attacks.

**Splitting Tensile Strength Results**

Figure 2 reveal that, under normal conditions and at the 28-day age curing period, the testing results consistently fall within a range of 6 to 8 MPa. This uniformity can be primarily attributed to a substantial decrease in the capillary porosity within the cement matrix. Notably, the results clustered between 6 and 8 MPa, with the exception of the mixture containing WP, which recorded a value of 4.5 MPa. The results generally show that the tensile strength decreases as the exposure period increases. When considering a specific acid concentration and exposure duration, the extent of acid damage is influenced by the concrete type, the amount, and the substitution level of additives.

It’s worth noting that the effect of a 2.5 % sulfuric acid solution on splitting tensile strength at 90 days is relatively low. The reduction percentages are 2.8 % for mixtures containing (15 % FA and 15 % SF) and 31 % for mixtures containing (25 % FA and 5 % SF), compared to their respective results at the 28-day age under standard conditions. After 180 days, the reduction percentages are 46 % for mixtures containing (WP) and 15 % for mixtures containing (FA, SF, and NS), compared to their results at the 28-day age under normal conditions.

Generally, it can be observed that there is minimal variation among the specimens containing (FA, SF, and NS). This can be primarily attributed to a substantial decrease in the capillary porosity within the cement matrix. Furthermore, the deflocculated system maintains particles at a more consistent spacing from each other. Consequently, as hydration continues, there is a higher likelihood of the interlocking of hydration products with the surfaces of both fine and coarse aggregates. This results in the development of a micro-scale system with greater integrity, leading to higher splitting tensile strength.

![Figure 2](https://doi.org/10.56294/sctconf2024851)
Absorption Test Results

The absorption test results are depicted in figure 3, 4, and 5. Based on the outcomes, it becomes evident that under standard conditions and at the 28-day age, the absorption rates were as follows: 2.2% for mixtures containing (WP), 1.3% for mixtures containing (25% FA and 15% SF), and 0.5% for mixtures containing (15% FA and 5% SF).

In more challenging conditions and at the 90-day exposure period, the absorption rates were as follows: 3% for mixtures containing (WP), 1.3% for mixtures containing (20% FA and 15% SF), and 1.8% for mixtures containing (FA, SF, and NS). After an exposure period of 180 days, the results were 3.9% for mixtures containing (WP), 1.75% for mixtures containing (20% FA and 15% SF), and 2% for mixtures containing (FA, SF, and NS). It’s worth noting that, irrespective of the type of concrete, all concrete specimens exhibited a decrease in the absorption percentage, particularly those containing Nano Silica.
CONCLUSIONS

1. Under normal conditions, the introduction of admixtures results in relatively modest increases in strength properties compared to their impact in acidic conditions.

2. Under normal conditions at 28 days, the combination of 25 % FA and 15 % SF can greatly boost compressive strength by 92 %, and in a lower range by 69 % when 15 % FA, 10 % SF, and 0.2 % NS were included.

3. At 90 days, the decline in strength was 16 % for the mixture with WP, and very small not more than 4.5 % for the mixture comprising 25 % FA and 15 % SF, or 6.9 % for the mixture having 15 % FA, 5 % SF, and 0.5 % NS. At 180 days, the strength reduction rose slightly to 38 % for the mixture with WP, 13.6 % for the mixture with 25 % FA and 15 % SF, and 15 % for the mixture with 15 % FA, 10 % SF, and 0.2 % NS.

4. The additives used do not significantly affect the splitting tensile strength under standard conditions, but a noticeable decrease is observed after subjecting the samples to acidic conditions.

5. Mineral admixtures exert a notable influence on reducing absorption rates. Specifically, at 60 days (during acid exposure), the absorption rate was 3 % for WP, and after 180 days, it was 2.3 % for the mixture with 25 % FA and 5 % SF. This effect primarily stems from the reduction in permeable voids, resulting from a decrease in both the quantity and size of capillary pores. Consequently, these improvements enhance the concrete’s overall durability.

6. The incorporation of NS, SF, and FA into cement concrete creates a novel material to address the rising demand for construction materials. This method also has economic and environmental benefits because it reduces pollution from FA and SF wastes.

REFERENCES


FINANCING
None.

CONFLICT OF INTEREST
None.

AUTHORSHIP CONTRIBUTION
Conceptualization: Shereen Jalil Saif Allah, Muayad Mohammed Kassim, Ghazwan Abdulsamad Salman.
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Writing - revision and editing: Shereen Jalil Saif Allah, Muayad Mohammed Kassim, Ghazwan Abdulsamad Salman.