Chemical Durability of Concrete Containing Pyrolyzed Waste Tyre Wires: A Mechanical and Microstructural Investigation

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Abstract- The depletion of natural resources and need for advancements in industrial waste management have motivated researchers to focus on identifying innovative strategies to lower carbon emissions within the construction industry. Concrete's inherent brittleness limits its ductility. To enhance this property, researchers have explored the incorporation of fibres in concrete. This research evaluates the durability of concrete containing Pyrolyzed Waste Tyre Wires (PWTW) under chemical erosion. Specimens with PWTW dosages of 0%, 0.5%, 1%, and 1.5% by weight were prepared and exposed to H_2SO_4 and $MgSO_4$ solutions for up to 56 days. Compressive strength evolution and microstructural changes (SEM) were assessed. After 56 days, PWTW inclusion significantly mitigated strength loss due to chemical attack. Compared to the control (0% PWTW), the 1% PWTW mix showed the lowest strength loss in H_2SO_4 (11.65% vs 15.93%), while the 1.5% PWTW mix performed best in $MgSO_4$ (20.9% vs 28.88% loss). SEM analysis revealed denser microstructures in PWTW-reinforced samples after chemical exposure. Findings demonstrate that PWTW enhances concrete's resistance to sulfuric acid and magnesium sulphate attack, supporting their use in durable and sustainable construction.

Keywords- End of Life Tyres, Concrete, Durability, Environment, Acid Attack, Sulphate Attack

I. Introduction

Concrete, a fundamental construction material, is widely used in buildings and infrastructure due to its strength, workability, and cost-effectiveness. However, its inherent weaknesses, including high self-weight, low tensile strength, and brittleness, limit its applicability in specialized projects [1], also previous research show concrete's vulnerability to biological and physical damage. Therefore, it must be improved to achieve higher strength, toughness, workability, and durability. To address these weaknesses, researchers have proposed distributing reinforcing fibres throughout the concrete's cross section [2]. This new material called Fibre Reinforced Concrete (FRC) has become widely adopted in industrial infrastructure including industrial floors, sewage and wastewater tunnels, agricultural silos, fermenters, and power plant cooling towers, due to its enhanced strength and ductility. However, these applications are prone to chemical deterioration, underlining the importance of studying the performance of FRC under various chemical attacks [3]. When cementitious composites come into contact with acidic solutions, the main phases of the hardened cement paste begin to break down and dissolve. This chemical reaction diminishes the alkalinity of the pore solution, leads to increased porosity, and consequently reduces the mechanical strength of the concrete. This degradation process is commonly referred to as chemical erosion of concrete [4].

There are different types of chemical erosion of concrete, including acid and sulphate chemical erosion of concrete. Solid sulphates typically have little impact on concrete, but in liquid form, they migrate into the concrete's voids and react with its hydrated cement products. This chemical reaction, known as sulphate attack, involves sulphate ions breaking down the cement paste. The process is driven by water-soluble sulphate salts particularly those of alkali-earth metals (like calcium and magnesium) and alkali metals (such as sodium and potassium) which react with the concrete components. Below is a detailed depiction of the chemical process [5].

$$MgSO_4 + Ca(OH)_2 + 2H_2O = CaSO_4.2H_2O + Mg(OH)_2 \quad (1)$$

Cementitious composites are inherently highly alkaline, with pH levels exceeding 12. When cement paste comes into contact with acids, its components begin to break down, a process termed acid attack. As the pH drops below the stability limits of cement hydrates, these hydrates lose calcium and decompose into an amorphous hydrogel [4]. The final products of this acid attack are the calcium salts corresponding to the attacking acid, along with hydrogels comprising silicon, aluminium, and ferric oxides. As the pH of the acid decreases below approximately 6.5, concrete becomes increasingly vulnerable, leading to the dissolution of both its hydrated and un-hydrated cement compounds as well as calcareous aggregates [6]. The chemical reactions involved in a typical acid attack on cementitious composites can be given as follows:

$$Ca(OH)_2 + H_2SO_4 = CaSO_4.2H_2O \quad (2)$$

$$3CaO.2SiO_2.3H_2O + H_2SO_4 = CaSO_4.2H_2O + Si(OH)_4 \quad (3)$$

Dsouza et al., 2018 [7] investigated the strength and durability of Steel Fibre Reinforced Concrete (SFRC) produced using M25-grade concrete with steel fibres of aspect ratio 60 at dosages of 0.5%, 1%, and 1.5% by weight. In their study, concrete cubes were immersed in a hydrochloric acid solution of pH 2, with concentration of 5% water weight, for the acid attack test, and in both sodium sulphate and magnesium sulphate solutions each at 5% water weight concentration for the sulphate attack test. Their findings revealed that SFRC exhibits improved resistance to both acid and sulphate attacks compared to conventional concrete with the 1.5% steel fibre mixture demonstrating the highest resistance.

Vegesana and Killamsetty, 2020 [8] investigated the compressive strength of SFRC exposed to chemical attack using M30 grade concrete. In their study, hooked-end steel fibres with an aspect ratio of 50, were incorporated at 0% and 3% of weight of cement and were randomly dispersed throughout the mix. Cube specimens were then immersed in 5% concentrated solutions of H_2SO_4 and $MgSO_4$ for a period of 30, 60, 90, 120, 150 and 180days. The results indicated that steel fibres improved the concrete's resistance to acid attack and sulphate attack.

Zhang et al., 2022 [9] studied the microscopic properties of SFRC under chemical erosion using M30 grade concrete with steel fibre contents of 0%, 1%, and 2%. Specimens were immersed in a 5% sulphuric acid solution and a 10% sodium sulphate solution for 28 days. After immersion, both the microstructural properties and the axial bearing capacity were measured. The results indicated that adding steel fibres significantly improved the axial bearing capacity, and chemical erosion accelerated the concrete's failure, although SFRC had greater resistance. The study identified an optimal steel fibre content of 1% for a sodium sulphate environment and 2% for a dilute sulphuric acid environment.

This research's significance is to explore alternative materials to enhance concrete properties while also sourcing an eco-friendly material from industrial waste. The paper examines the durability of steel fibre reinforced concrete under various adverse environmental conditions. By incorporating pyrolyzed waste tyre wires, the study evaluates both the mechanical and microstructural properties of the concrete.

II. Materials and Methods

A. Materials

The materials used for this study are as follows; cement, fine aggregate, coarse aggregate, water and steel fibre. Details of the materials are as follows:

1) Ordinary Portland cements: All mixes employed locally manufactured Dangote brand 3X 42.5 cement, whose chemical, physical, and mechanical properties adhere to the standards set by EN 197-1:2000 [10].

2) Aggregates: The aggregates used in the experiment consisted of both fine and coarse materials that comply with BS EN 12620:2013 [11]. The fine aggregate was sharp river sand with a maximum particle size of 4.75 mm, while the coarse aggregate was crushed gravel with a maximum size of 20 mm. Both types were sourced from Zaria, as shown in Figure 1.

3) Water: Pipe-borne water sourced from Ahmadu Bello University in Zaria, which adheres to the specifications of BS EN 1008:2015 [12], is used.

4) Steel Fibres: Steel fibres were sourced from Zango Abattoir in Sabon Gari LGA, Zaria, Kaduna State. At this facility, waste tyres are used as fuel during meat processing, which produces steel wires; thus, the fibres are a product of the pyrolysis process. Tyre wires of 1.67mm diameter obtained from heavy-duty truck tyres were used, these steel wires were then cut into 50mm length and used as discrete steel fibres in concrete. Preliminary tests conducted at the Department of Metallurgical and Material Engineering Laboratory, Ahmadu Bello University, Zaria, confirmed that the fibres meet the tensile strength requirements specified in ASTM A820 [13] and BS EN 14889-1 [14]. The results are presented below in Table 1.

Table 1: Properties of Recycled Steel Fibre

Specimen	Breaking Force (N)	Tensile Strength (MPa)	Elongation at break (%)
Maximum	4300	1938.01	21.5
Minimum	1870	844.5	6.96
Mean	3005	1350.84	10.67

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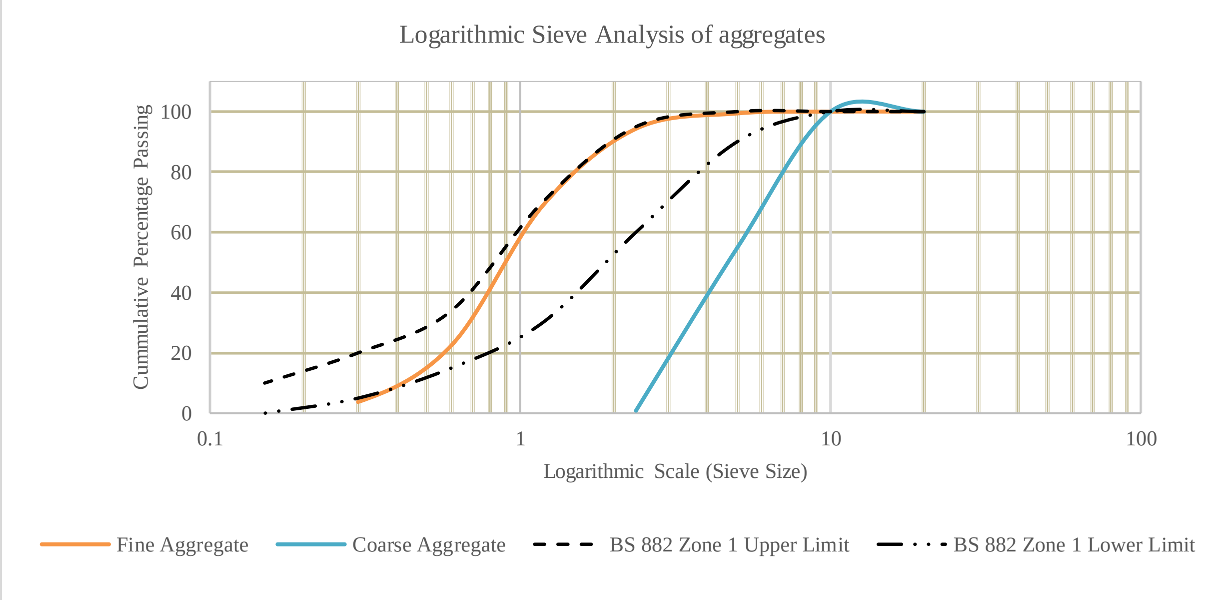

Figure 1: Particle Size Distribution of Aggregates

B. Mix design

Concrete Grade 25 was utilized in this study, with the mix design carried out according to the Building Research Establishment (BRE) design method. The quantities of materials prescribed by the mix design are detailed in Table 2.

Table 2: Quantity of Materials

Materials	Cement	Water	Aggregate		Fibre		
			Fine	Coarse	0.5%	1%	1.5%
Quantity (Kg/m^3)	370	210	675	1150	12	24	36

A total of 180 specimens were produced, using four mix designs with varying steel fibre dosages: a control mix, alongside mixes containing 0.5%, 1%, and 1.5% fibre by volume of concrete.

C. Methods

The study was carried using the following processes which include;

1) Test on fresh properties of concrete: Fresh property test carried out on concrete containing various dosages of the fibre include:

Workability Test: Slump values were measured in accordance with BS EN 12350-2:2019 [15], using a slump cone with a height of 300 mm, a bottom diameter of 200 mm, and a top diameter of 100 mm.

2) Test on harden properties of concrete: To evaluate the compressive strength of concrete, a uniaxial compression test was performed on 100 mm × 100 mm × 100 mm cubes in accordance with BS EN 12390-3:2019 [16].

Resistance to sulphuric acid (H_2SO_4): The resistance to external acid attack was assessed following ASTM C267-01 [17]. Initially, 100 × 100 × 100 mm cubes were cast and cured in water for 28 days. After curing, the samples were removed, allowed to reach a saturated surface-dry condition. Subsequently, the specimens were immersed in a 5% sulfuric acid solution for additional curing periods of 28 and 56 days.

Resistance to Magnesium Sulphate ($MgSO_4$) Solution: The resistance to external sulphate attack was assessed according to ASTM C1012 [18]. Cubes measuring 100 × 100 × 100 mm were cast and water-cured for 28 days. Once cured, the specimens were removed, allowed to reach a saturated surface-dry condition. They were then immersed in a 5% magnesium sulphate solution (prepared by dissolving $MgSO_4$ salts in water) for additional curing periods of 28 and 56 days.

Microstructure: The microstructure of the specimens, was examined using a Scanning Electron Microscope (SEM). This analysis was carried out at the National Steel Raw Materials Exploration Agency (NSRMEA) Laboratory in Malali, Kaduna, following the procedures specified in ASTM C1723-16 [19].

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III. Results

A. Properties of Fresh Concrete Specimen

1) Slump: Figure 2 presents the results of the slump test conducted on fresh concrete specimens. It displays the slump values obtained from concrete mixes with varying fibre dosages of 0%, 0.5%, 1%, and 1.5%. As expected, the results reveal a decline in workability as the fibre content increases. The presence of fibres restricts the mobility of the concrete mix, leading to reduced workability, a finding that is consistent with the observations reported by ACI Committee 544 (2002) [20].

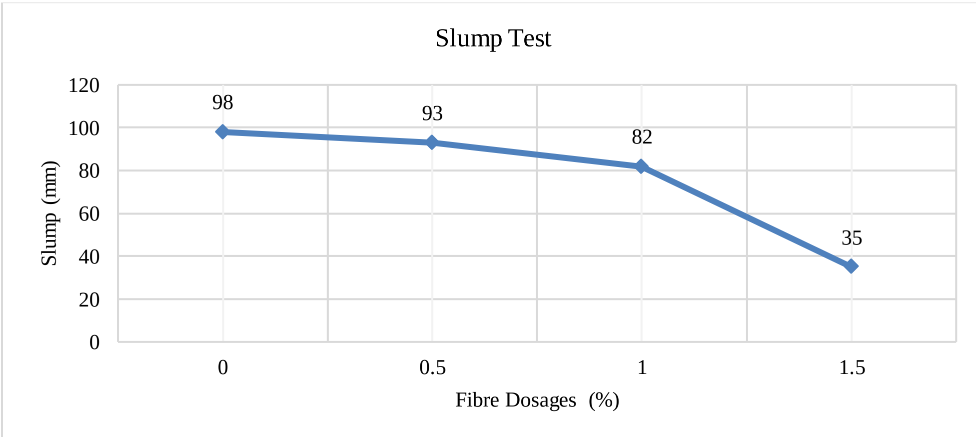

Figure 2: Slump Values at Fresh stage

B. Effect of Chemically Aggressive Environment on Hardened Concrete

1) Resistance to sulphuric acid (H_2SO_4) attack: Figure 3 shows the compressive strength of concrete specimens that were cured in a 5% H_2SO_4 solution for 28 days, while Figure 4 displays the compressive strength after 56 days of acid curing. Overall, all samples experienced a decline in compressive strength following exposure to the acid solution.

After 28 days of acid curing, the percentage reduction in compressive strength relative to normally cured specimens was 7.22% for the control mix, 4.38% for the mix with 0.5% fibre, 1.32% for the 1% fibre mix, and 12.46% for the 1.5% Pyrolyzed Waste Tyre Wires (PWTW) mix. Although all specimens demonstrated some strength loss due to acid exposure, the mixes with 0.5% and 1% fibre content showed considerably better acid resistance compared to the control, while the mix with 1.5% fibre did not perform as well.

After 56 days of acid curing, the decreases in compressive strength compared to normally cured specimens were 15.93% for the control sample, 14.37% for the 0.5% PWTW mix, 11.65% for the 1% fibre mix, and 15.18% for the 1.5% PWTW mix. Notably, the control sample exhibited severe deterioration, resulting in all fibre-reinforced concrete (SFRC) mixes outperforming it. The incorporation of pyrolyzed waste tyre wires led to an optimal reduction in strength deterioration of 5.9% and 4.28% at 28 and 56 days, respectively, which aligns with the results reported by [7] and [3].

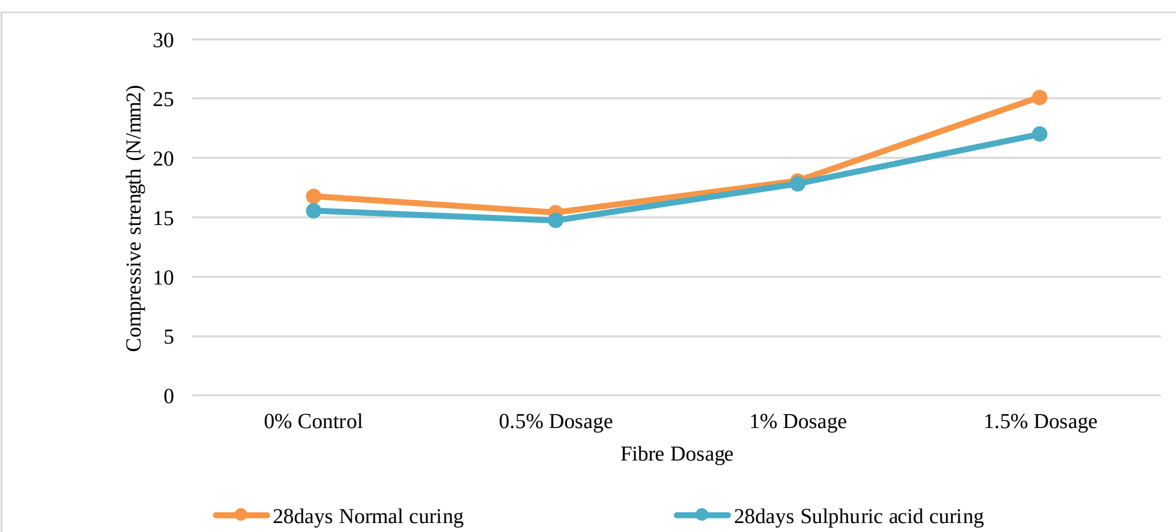

Figure 3: Effect of H_2SO_4 curing on Compressive strength of Concrete with PWTW after 28days

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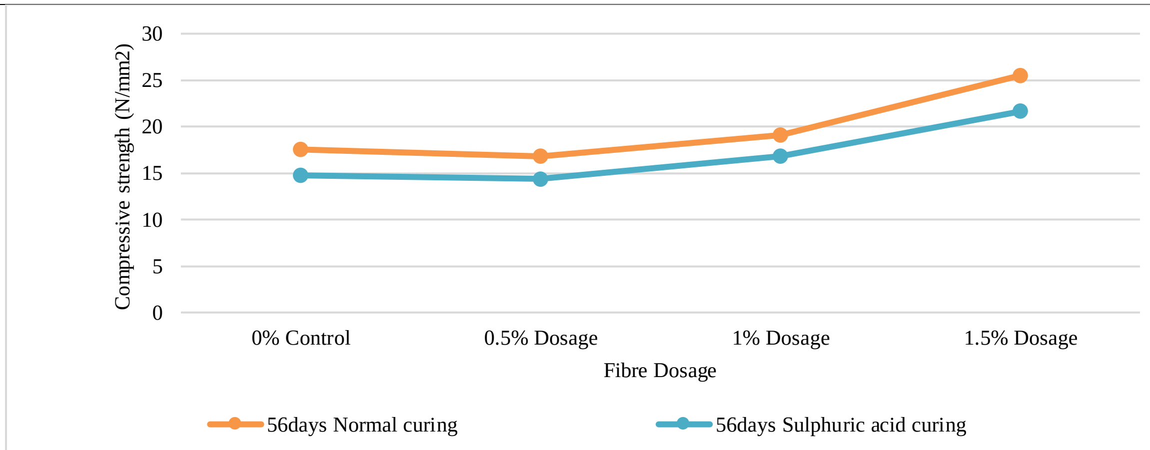

Figure 4: Effect of H_2SO_4 curing on Compressive strength of Concrete with PWTW after 56days

2) Resistance to magnesium sulphate ($MgSO_4$) attack: Figure 5 presents the compressive strength of concrete specimens exposed to a magnesium sulphate ($MgSO_4$) solution after 28 days of curing. All specimens showed a reduction in compressive strength when cured in the $MgSO_4$ solution. Specifically, after 28 days, the control mix experienced a reduction of 24.93%, while the mixes with 0.5%, 1%, and 1.5% fibre dosages exhibited decreases of 12.19%, 16.28%, and 19.09%, respectively.

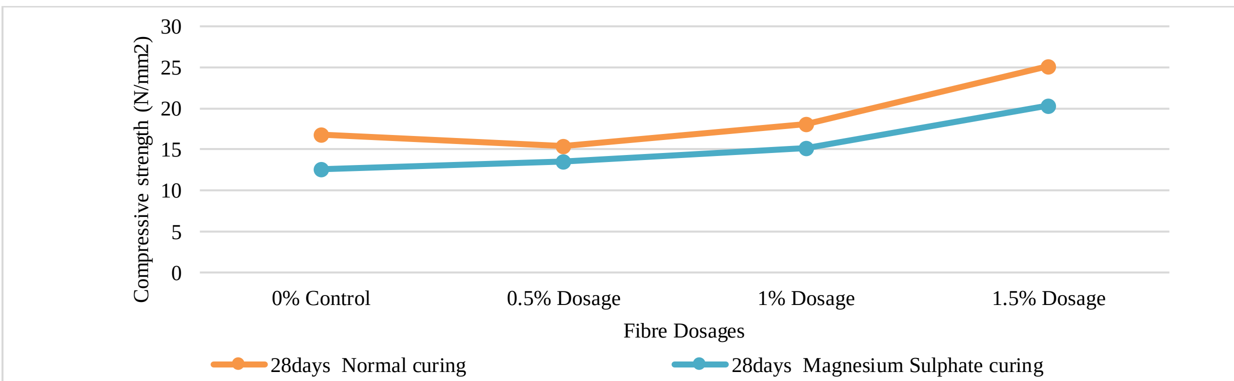

Figure 5: Effect of $MgSO_4$ curing on Compressive strength of Concrete with PWTW after 28days

Figure 6 displays the compressive strength after 56 days of $MgSO_4$ curing. Here, all specimens experienced a further, albeit slight, loss in strength. The control mix showed a 28.88% reduction compared to its normal 56-day strength. Meanwhile, the fibre-reinforced concrete, referred to here as PWTW concrete with 0.5%, 1%, and 1.5% fibre dosages recorded compressive strength losses of 22.2%, 22.9%, and 20.9% respectively. Notably, the overall strength loss between the 28-day and 56-day curing periods was relatively minimal across all specimens.

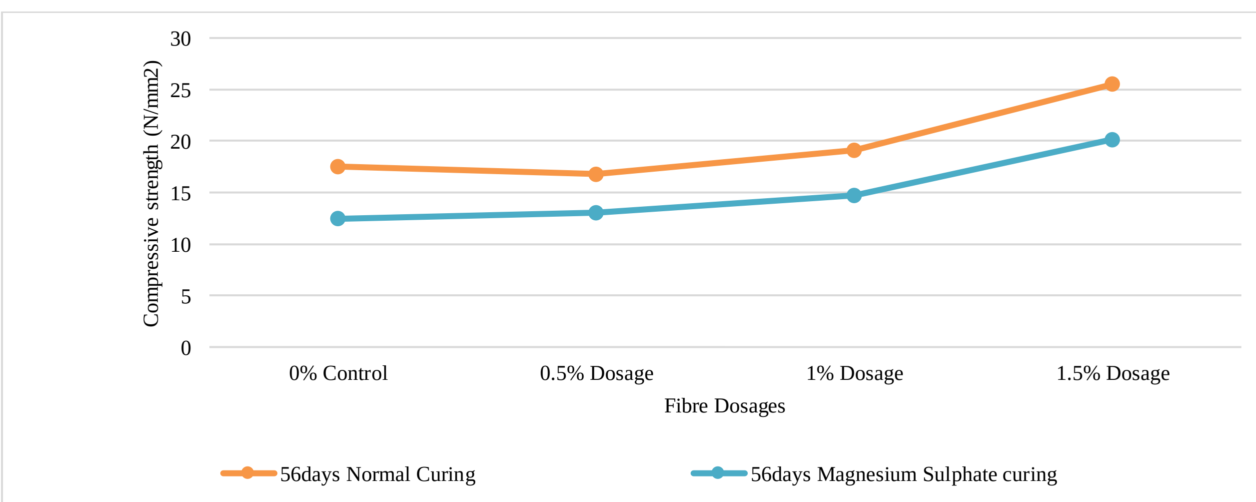

Figure 6: Effect of $MgSO_4$ curing on Compressive strength of Concrete with PWTW after 56days

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C) Microstructural analysis of concrete exposed to Chemically aggressive environment

Sulphuric acid (H_2SO_4): Figure 7 illustrates the SEM micrographs of 0% PWTW concrete and 1.5% PWTW concrete specimens, captured both before and after exposure to an acid solution. In both cases, the cement paste, which appeared dense prior to acid immersion, becomes noticeably loose afterward, with dislodged fragments, enlarged pore sizes, and numerous discernible cracks evident in the post-exposure images.

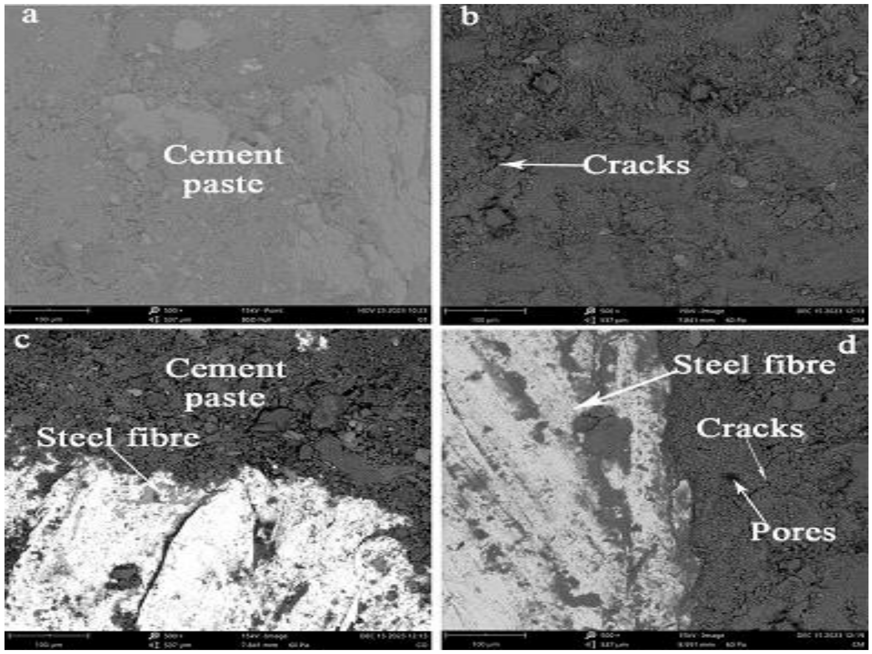

Figure 7: SEM Images of Concrete Samples before and after exposure to H_2SO_4, a) Control Sample Before; (c) 1.5% PWTW Concrete before exposure; (d) 1.5% PWTW Concrete after exposure

Magnesium sulphate ($MgSO_4$): Figure 8 displays the microscopic morphology of specimens with 0% and 1.5% fibre dosage after immersion in a 5% $MgSO_4$ solution for 56 days. The dense cement paste, initially observed in both the 0% and 1.5% PWTW concrete samples has deteriorated, becoming loose and highly porous. Additionally, more microcracks are apparent after exposure. In the 1.5% PWTW concrete, the cracks predominantly occur along the aggregate-cement interfacial transition zone (ITZ) rather than along the steel fibre, which indicates a stronger bond in the steel-cement ITZ compared to the aggregate-cement ITZ. Prior to $MgSO_4$ immersion, the 1.5% PWTW concrete exhibited a strong bond between the steel fibre and the cement paste; however, after immersion and during sample preparation for SEM the cement paste tends to separate from the steel fibre.

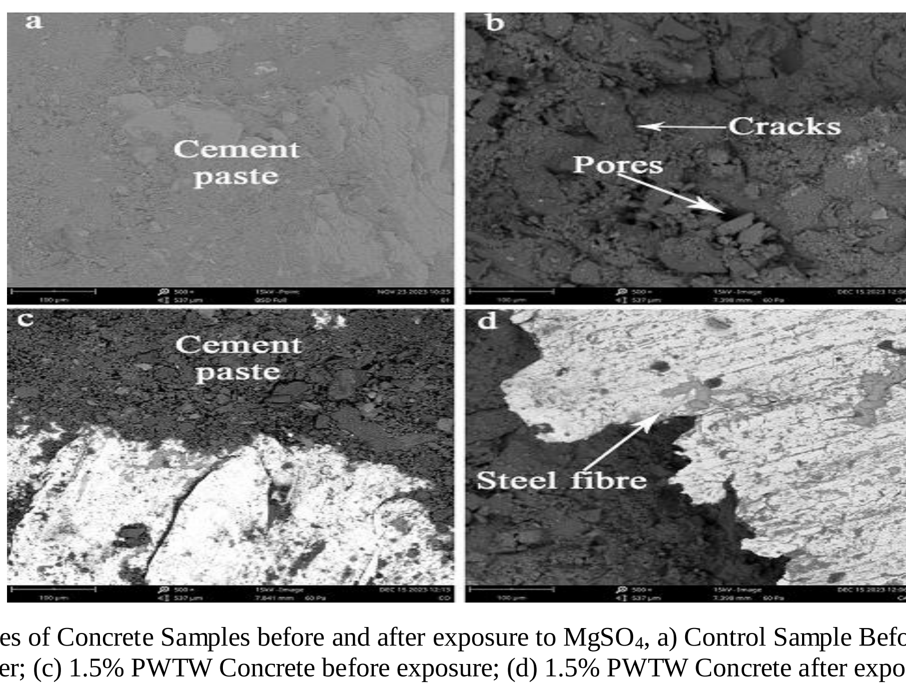

Figure 8: SEM Images of Concrete Samples before and after exposure to $MgSO_4$, a) Control Sample Before; b) Control Sample after; (c) 1.5% PWTW Concrete before exposure; (d) 1.5% PWTW Concrete after exposure

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IV. Conclusions

Based on the experimental findings, the following conclusions can be deduced:

1. An increase in the steel fibre content significantly reduces the workability of the concrete. Specifically, the slump decreased by 64.3% from 98 mm in the 0% fibre dosage to 35 mm at a 1.5% fibre dosage.
2. The incorporation of steel fibres was found to significantly enhance the compressive strength of SFRC. Notably, at 56 days, a 1.5% fibre dosage resulted in a 45.7% increase in compressive strength compared to the control mix.
3. Specimens exposed to the H_2SO_4 solution demonstrated a loss in compressive strength. However, SFRC proved to be more resistant to acid attack. Specifically, the concrete mixes with fibre dosages of 0%, 0.5%, 1%, and 1.5% experienced compressive strength losses of 15.93%, 14.37%, 11.65%, and 15.18%, respectively, highlighting enhanced durability with fibre incorporation.
4. The results indicate that SFRC exhibits enhanced resistance to sulphate attack compared to conventional concrete. When immersed in a $MgSO_4$ solution, the compressive strength reductions for concrete with fibre dosages of 0%, 0.5%, 1%, and 1.5% were recorded as 28.88%, 22.2%, 22.9%, and 20.9%, respectively. This clearly demonstrates that incorporating steel fibres improves sulphate resistance, with the SFRC mixes suffering significantly lower strength losses.

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