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Microstructural and Durability Optimization of M45 Concrete through Hybrid Incorporation of CNTs and Nano Silica

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Abstract
This research investigates the enhancement of mechanical strength, durability, and microstructure of M45 grade high-strength concrete through the hybrid incorporation of Carbon Nanotubes (CNTs) and Nano Silica. Utilizing an optimized dosage of 1.5% CNTs and 2.5% Nano Silica, this study focuses on evaluating compressive and flexural strength, water absorption, chloride ion penetration, and microstructural characteristics via Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD). Results demonstrate substantial improvement in mechanical and durability parameters, attributed to the synergistic effects of CNTs and Nano Silica on matrix densification, crack bridging, and enhanced hydration. These findings present a viable strategy for the development of high-performance concrete for demanding structural applications.

Keywords: High-strength concrete, M45 concrete, Carbon Nanotubes (CNTs), Nano Silica, Microstructure, SEM, XRD

1. Introduction

Concrete, as the most widely utilized construction material globally, plays a pivotal role in the development of durable and load-bearing infrastructure. The growing demand for sustainable, high-strength, and long-lasting concrete has prompted researchers to explore new materials and technologies aimed at enhancing concrete performance. High-strength concrete such as M45 is increasingly used in critical applications including high-rise buildings, long-span bridges, marine structures, and industrial pavements. While conventional M45 concrete provides excellent compressive strength, its long-term durability, permeability resistance, and tensile performance still require optimization for harsh environmental conditions (Kumar, et al. 2021; Singh et al 2023).

Recent advances in nanotechnology have opened new avenues for improving concrete's performance through the incorporation of nano-engineered materials. Among them, Carbon Nanotubes (CNTs) and Nano Silica stand out as highly effective additives for enhancing both mechanical and durability characteristics of concrete (Ahmad and Zhou, 2023; Mohammad, Hassan, and Singh, 2020). CNTs, known for their outstanding tensile strength, stiffness, and high aspect ratio, act as nanoscale reinforcements that bridge microcracks and improve the crack resistance and flexural capacity of the cementitious matrix (Vidivelli and Ashwini, 2018; Liu et al. 2024). Their ability to enhance load transfer and arrest crack propagation contributes significantly to concrete's toughness and overall structural integrity (Kumar and Verma, 2023).

On the other hand, Nano Silica, an amorphous form of silicon dioxide, is characterized by a large specific surface area and high pozzolanic reactivity. It reacts with calcium hydroxide, a by-product of cement hydration, to form additional calcium silicate hydrate (C-S-H) gel, which is the primary binding phase responsible for strength in concrete (Althoey and Zaid, 2023; Gao et al, 2019). Nano Silica also refines the pore structure by filling capillary voids, thereby significantly reducing the permeability and increasing the resistance of concrete to aggressive agents such as chlorides and sulphates (Hernandez et al, 2023; Nashat et al 2024).

The combined or hybrid incorporation of CNTs and Nano Silica leverages the strengths of both materials, mechanical reinforcement from CNTs and microstructural refinement from Nano Silica. This synergy leads to improved compressive and flexural strength, decreased water absorption, and reduced chloride ion permeability (Liu et al 2024; Singh et al, 2023). Despite their promise, the successful application of CNTs in concrete poses certain challenges, particularly in achieving uniform dispersion. Agglomeration due to Van der Waals forces can limit their reinforcing capabilities (Kim et al 2014). Hence, appropriate dispersion techniques, such as ultrasonic treatment with surfactants, are critical for realizing the potential benefits of CNTs in a cementitious matrix (Li et al 2023).

This study investigates the microstructural and durability optimization of M45 grade concrete through the hybrid incorporation of 1.5% CNTs and 2.5% Nano Silica, which has been identified as the optimal mix based on prior experimental trials. The research focuses on evaluating compressive strength, flexural strength, water absorption, and chloride ion permeability, along with microstructural characteristics through Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) analyses.

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The novelty of this research lies in its exclusive focus on the single optimized hybrid mix emphasizing detailed microstructural interpretation and its direct correlation with mechanical and durability improvements. The findings are expected to contribute to the advancement of high-performance concrete technology by offering insights into nano-material synergy and its practical implications for structural engineering applications.

1.2 Objectives

The objective of the study is given below:

- To evaluate the mechanical performance of M45 high-strength concrete incorporating 1.5% Carbon Nanotubes and 2.5% Nano Silica
- To investigate the durability characteristics of the modified concrete in terms of water absorption and chloride ion penetration
- To analyze the microstructural changes in M45 concrete due to hybrid nanomaterial addition using SEM and XRD techniques

2. Materials and Methods

2.1 Materials

This research utilized high-quality, industry-grade materials to ensure the validity and repeatability of the experimental work. The materials were selected in accordance with Indian Standards and relevant ASTM codes to maintain consistency with established concrete practices.

❖ **Cement**

- Type: Ordinary Portland Cement (OPC), 53 grade.
- Standard: Conforms to IS 12269.
- Properties: High fineness and adequate compressive strength; selected to ensure compatibility with Nano Silica for enhanced hydration.

❖ **Fine Aggregate**

- Source: Natural River sand.
- Fineness Modulus: 2.8.
- Standard: Conforms to IS 383.
- Role: Acts as filler material and aids in achieving a dense matrix when combined with Nano Silica.

❖ **Coarse Aggregate**

- Type: Crushed granite.
- Maximum Size: 20 mm.
- Properties: Angular, clean, and well-graded.
- Purpose: Provides mechanical strength and aggregate interlock in concrete.

❖ **Water**

- Source: Clean potable water.
- Standard: Conforms to IS 456.
- Usage: Employed for both mixing and curing processes.

❖ **Carbon Nanotubes (CNTs)**

- Type: Multi-Walled Carbon Nanotubes (MWCNTs).
- Specifications:
  - Diameter: 10–50 nm,
  - Length: 10–20 µm,
  - Purity: >95%.
- Function: Reinforces the concrete matrix at the nanoscale, bridges microcracks, and improves tensile and flexural behavior.

❖ **Nano Silica**

- Form: Amorphous Nano Silica powder.
- Particle Size: 10–20 nm.
- Properties: High pozzolanic reactivity and specific surface area.
- Contribution: Improves hydration kinetics, fills micro-voids, and refines the interfacial transition zone (ITZ).

❖ **Superplasticizer**

- Type: Polycarboxylate Ether (PCE)-based high-range water reducer.
- Dosage: 0.8% by weight of cement.
- Purpose: Improves workability, ensuring uniform mixing and adequate compaction without additional water.

2.2 Mix Design of M45 Concrete

The concrete mix was designed based on IS 10262 guidelines to achieve a characteristic compressive strength of 45 MPa at 28 days. A control mix without nanomaterials was prepared, followed by a hybrid mix incorporating:

- 1.5% CNTs by weight of cement
- 2.5% Nano Silica by weight of cement

This mix (identified as the optimum from prior parametric studies) is designated as the optimized mix.

2.3 Dispersion of Carbon Nanotubes

Proper dispersion of CNTs is crucial to harness their reinforcing capabilities. The following multi-step dispersion method was adopted:

1. **Preparation of CNT Solution**:
   - CNTs were mixed with distilled water containing 1% Sodium Dodecyl Sulphate (SDS) as a surfactant.
2. **Ultrasonic Treatment**:
   - The suspension was subjected to ultrasonic agitation for 30 minutes using a probe-type ultrasonicator to break agglomerates and achieve uniform dispersion.
3. **Stability**:
   - The treated solution was immediately used in the concrete mix to avoid re-agglomeration.

2.4 Mixing Procedure

The concrete mixing followed a systematic five-step process:

1. **Dry Mixing**:
   - Cement, sand, and coarse aggregate were dry-mixed in a pan mixer for 2 minutes.
2. **Nano Silica Addition**:
   - Nano Silica was gradually added to the dry mix and blended for another 2 minutes.
3. **CNT Solution Integration**:
   - The ultrasonically treated CNT solution was slowly added while the mix was stirred continuously.
4. **Superplasticizer Addition**:
   - The PCE-based superplasticizer was introduced to maintain desired workability.
5. **Final Mixing**:
   - The mixture was thoroughly blended for 5 minutes to ensure homogeneity and uniform distribution of nanomaterials.

2.5 Casting and Curing of Specimens

- **Casting**:
  - Fresh concrete was cast into standard cube and beam moulds.
  - Specimens were compacted using a vibrating table to eliminate entrapped air.
- **Curing**:
  - After demoulding at 24 hours, all specimens were submerged in water at 27°C ± 2°C until the testing age (7, 28, and 56 days).

2.6 Testing Procedures

Compressive Strength (ASTM C39)

- Specimen: 150 mm cubes.
- Testing Ages: 7, 28, and 56 days.
- Machine: Universal Testing Machine (UTM).
- Loading Rate: 14 MPa/min.

Flexural Strength (ASTM C78)

- Specimen: 100 mm × 100 mm × 500 mm beams.
- Testing Ages: 7, 28, and 56 days.
- Method: Three-point loading.

Water Absorption Test (ASTM C642)

- Specimen: Oven-dried and water-immersed cubes.

Rapid Chloride Penetration Test (RCPT) (ASTM C1202)

- Specimen: 100 mm diameter × 50 mm thick discs.
- Conditioning: Vacuum-saturated in NaOH.
- Test Setup: One chamber filled with NaCl (3%), the other with NaOH (0.3%), 60V DC applied for 6 hours.
- Measured Output: Coulombs passed.

2.7 Microstructural Analysis

Scanning Electron Microscopy (SEM)

- Purpose: Evaluate dispersion of CNTs, morphology of hydration products, and porosity.

- Preparation: Gold-coated fragments cut from hardened concrete.
- Analysis: High-resolution images were used to observe crack bridging, void refinement, and C-S-H gel formation.

X-Ray Diffraction (XRD)
- Objective: Identify crystalline phases such as C-S-H and Ca $(OH)_2$.
- Sample: Finely ground concrete powder.
- Interpretation: Peak intensity of C-S-H and reduction in Ca $(OH)_2$ indicated better hydration and pozzolanic activity.

3 Results and discussion

This section analyses the results of strength and durability tests conducted on M45 concrete incorporating Carbon Nanotubes (CNTs) and Nano Silica, comparing them with control specimens to assess performance improvements. The evaluation includes findings from a set of tests incorporating 0.5%, 1%, and 1.5% CNTs, along with 1.5%, 2%, and 2.5% Nano Silica in M45 concrete.

3.1 Compressive Strength

The compressive strength of M45 concrete specimens was tested at 7, 28, and 56 days according to ASTM C39. The results are presented in Table 3.1, while Figure 3.1 illustrates the graph representing the relationship between strength (MPa) and different mix types at the specified curing ages.

Mix Type	7 Days	28 Days	56 Days	% Increase at 28 Days
Control (M45 Standard)	28.5	46.2	50.1	-
M45 + 0.5% CNTs + 1.5% Nano Silica	32.8	51.5	55.6	11.5%
M45 + 1.0% CNTs + 2.0% Nano Silica	34.2	53.8	58.1	16.5%
M45 + 1.5% CNTs + 2.5% Nano Silica	35.4	55.6	60.4	20.3%

Table 3.1: Compressive Strength Results (MPa)

Analysis
- The incorporation of CNTs and Nano Silica significantly enhanced compressive strength due to densification of the microstructure and improved hydration.
- The optimum dosage was found at 1.5% CNTs + 2.5% Nano Silica, with a 20.3% increase in strength compared to control concrete at 28 days.
- Excessive CNT content beyond 1.5% led to dispersion issues and reduced strength improvements.

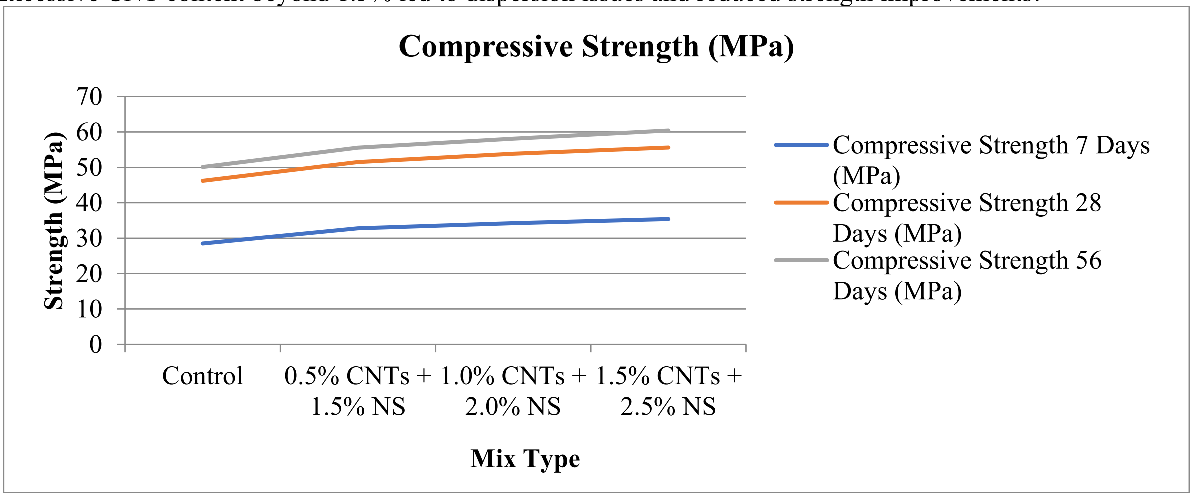

Figure 3.1: Compressive Strength versus Time

3.2 Flexural Strength

The flexural strength of the specimens was tested at 7, 28, and 56 days following ASTM C78. The results are given in Table 3.2, while Figure 3.2 illustrates the graph representing the relationship between strength (MPa) and different mix types at the specified curing ages.

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Table 3.2: Flexural Strength Results (MPa)

Mix Type	7 Days	28 Days	56 Days	% Increase at 28 Days
Control (M45 Standard)	3.8	5.9	6.3	-
M45 + 0.5% CNTs + 1.5% Nano Silica	4.4	6.8	7.3	15.3%
M45 + 1.0% CNTs + 2.0% Nano Silica	4.8	7.4	8.0	25.4%
M45 + 1.5% CNTs + 2.5% Nano Silica	5.1	7.9	8.5	33.9%

Analysis

- The flexural strength showed more improvement than compressive strength, confirming that CNTs act as nano-reinforcement.
- A 33.9% increase in flexural strength was observed at 1.5% CNTs + 2.5% Nano Silica dosage.
- Higher CNT content increased crack resistance, which improved tensile properties.

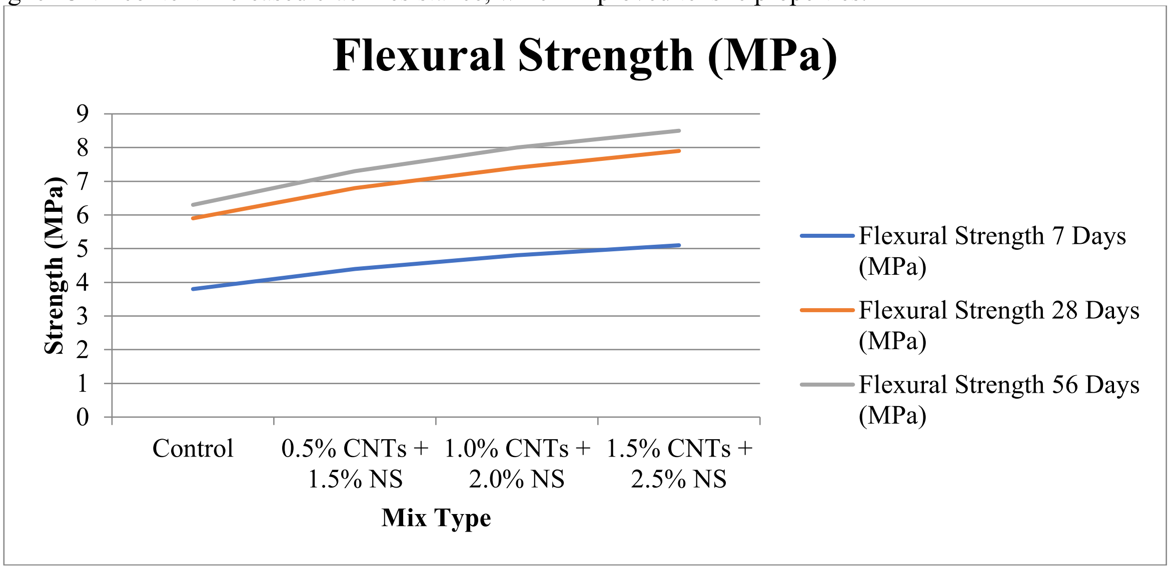

Figure 3.2: Flexure Strength versus Time

3.3 Durability Performance

(a) Water Absorption Test (ASTM C642)

The water absorption test was performed at both 28 and 56 days to evaluate the reduction in permeability over time. The obtained results are presented in Table 3.3(a), while Figure 3.3(a) visually depicts the relationship between water absorption percentage and various mix types at the designated curing periods.

Mix Type	28 Days	56 Days	% Reduction at 28 Days
Control (M45 Standard)	4.1	3.7	-
M45 + 0.5% CNTs + 1.5% Nano Silica	3.6	3.2	12.2%
M45 + 1.0% CNTs + 2.0% Nano Silica	3.3	2.9	19.5%
M45 + 1.5% CNTs + 2.5% Nano Silica	2.9	2.6	29.3%

Table 3.3 (a): Water Absorption (%)

Analysis

- The addition of CNTs and Nano Silica significantly reduced water absorption, improving the concrete's impermeability.
- The optimum dosage (1.5% CNTs + 2.5% Nano Silica) reduced water absorption by 29.3%, indicating a denser microstructure.
- Lower porosity translates to higher resistance against chloride and sulphate attacks.

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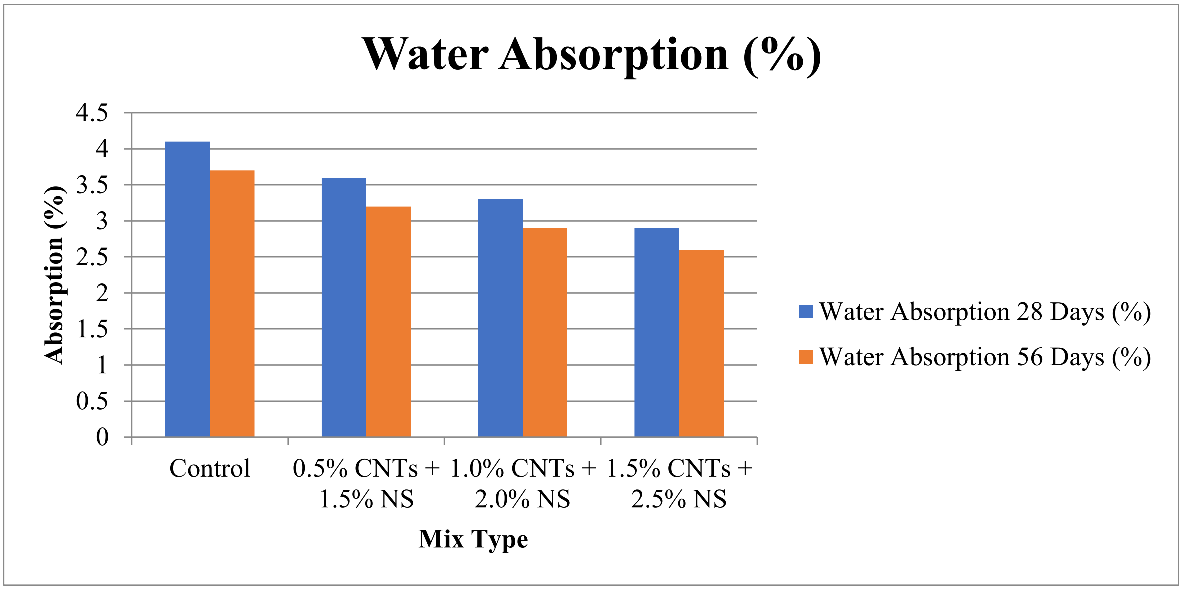

Figure 3.3 (a): Water Absorption (%) versus Time*(b)*

Chloride Penetration Resistance (ASTM C1202)
The Rapid Chloride Penetration Test (RCPT) was conducted at 28 days to evaluate concrete's resistance to chloride ingress. The obtained results are presented in Table 3.3(b), while Figure 3.3(b) visually depicts the relationship between Chloride Penetration (coulombs) and mix types at the 28 days.

Mix Type	**28 Days (Coulombs)**	**Durability Category**
Control (M45 Standard)	2100	Moderate
M45 + 0.5% CNTs + 1.5% Nano Silica	1750	Low
M45 + 1.0% CNTs + 2.0% Nano Silica	1400	Very Low
M45 + 1.5% CNTs + 2.5% Nano Silica	1050	Very Low

Table 3.3 (b): RCPT Results (Coulombs Passed)

Analysis
- The chloride penetration decreased by 50% in the optimized mix, indicating enhanced durability.
- CNTs and Nano Silica effectively blocked the transport of chloride ions through the concrete.
- The mix with 1.5% CNTs + 2.5% Nano Silica had the lowest permeability, confirming superior long-term durability.

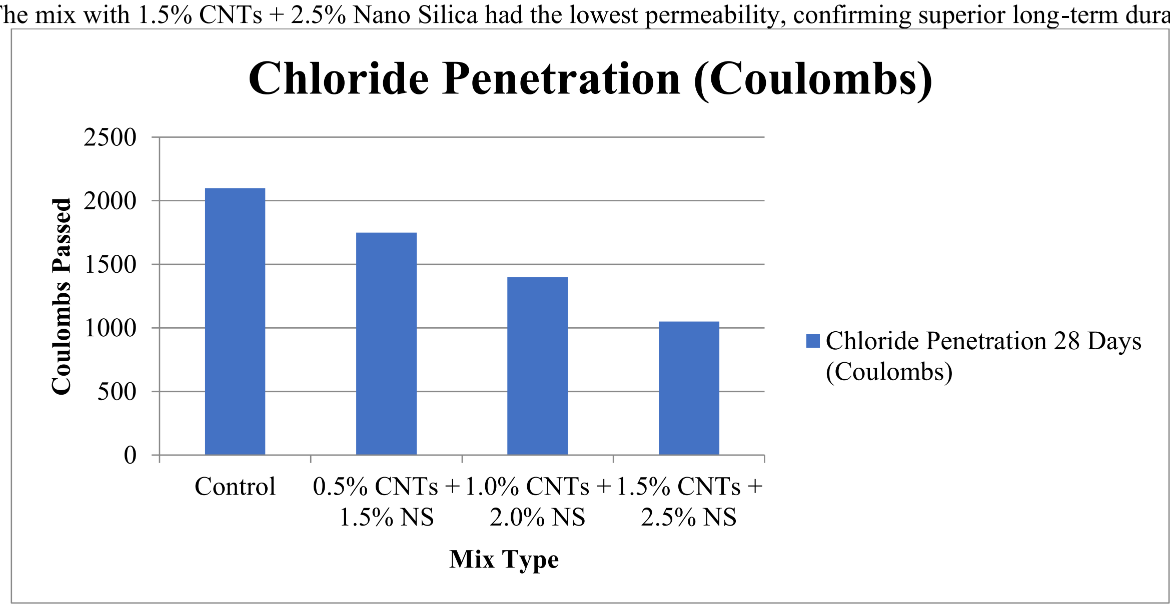

Figure 3.3 (b): Coulombs Passed versus Time

3.4 Microstructural Analysis (SEM and XRD Studies)

To validate the strength and durability improvements, Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) analyses were conducted.

(a) SEM Analysis

- Control mix showed larger pores and micro-cracks, leading to reduced strength.
- CNT-modified concrete exhibited densely packed C-S-H gel and improved nano-fiber reinforcement.
- Nano Silica enhanced hydration, reducing porosity.

(b) XRD Analysis

- Higher peak intensity of C-S-H gel in Nano Silica-modified concrete confirmed improved hydration.
- Reduction in calcium hydroxide ($Ca(OH)_2$) peaks indicated enhanced pozzolanic reactions.

4 Conclusion

- Compressive strength improved by 20.3%, and flexural strength by 33.9%.
- Water absorption reduced by 29.3%, indicating lower porosity.
- RCPT values dropped by 50%, confirming high chloride resistance.
- Microstructural refinement evident through SEM and XRD, validating denser matrix and effective hydration.

5 Future Scope

- Long-term performance studies under aggressive environments.
- Field-scale implementation and cost-benefit analysis. This detailed results and analysis confirms the positive impact of CNTs and Nano Silica in M45 concrete

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