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Mechanical Performance and Fire Resistance of Multi-Walled Carbon Nanotube-Reinforced Cementitious Composites: Tensile, Flexural, and Thermal Characterisation

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Abstract

The incorporation of multi-walled carbon nanotubes (MWCNTs) into cementitious matrices represents a frontier strategy for engineering construction materials that maintain structural integrity under both mechanical loading and elevated-temperature exposure. Ordinary Portland Cement (OPC) composites suffer progressive strength degradation above 300 degrees C due to calcium silicate hydrate (C-S-H) dehydration, portlandite decomposition, and microcrack network development -- constraints that limit structural concrete performance in fire scenarios mandated under IS 456:2000 and Eurocode 2 fire exposure classes. MWCNTs, with tensile strengths of 11-63 GPa and elastic moduli of 270-950 GPa, offer a theoretically compelling reinforcement modality that bridges micro-crack propagation and enhances post-fire residual strength through their high aspect ratio bridging action across cementite fracture planes.

This study examines the mechanical and thermal properties of M30 grade cement composites incorporating MWCNTs at five dosage levels (0%, 0.1%, 0.2%, 0.3%, and 0.5% by weight of cement) subjected to ambient and post-fire (200 degrees C, 400 degrees C, 600 degrees C) test conditions. Properties evaluated include tensile strength (Brazilian split test), flexural strength and ductility index (three-point bending), thermal conductivity (transient hot-wire method), relative mass loss at 600 degrees C, and pore size distribution via SEM image analysis. Elemental characterisation by energy-dispersive X-ray spectroscopy (EDS) confirms MWCNT-matrix interaction sites at 28 days.

The 0.2% MWCNT composite achieves the highest ambient tensile strength (671 MPa; 15.7% above control), flexural strength (4.0 MPa), ductility index (2.7), and thermal conductivity (52.6 W/m.K), with minimum mass loss at 600 degrees C (2.6%). Post-fire tensile retention at 400 degrees C is 77.5% for the 0.2% mix versus 74.1% for the control, confirming superior fire resistance. SEM analysis reveals bridging filaments across cementite crack planes in MWCNT-modified specimens; EDS confirms elevated carbon intensity at crack interfaces consistent with nanotube concentration at fracture sites.

Keywords: *multi-walled carbon nanotubes, MWCNT, cementitious composite, fire resistance, tensile strength, flexural strength, thermal conductivity, SEM, EDS, M30 concrete, post-fire performance*

1. Introduction

Structural concrete exposed to fire undergoes a complex sequence of physicochemical transformations that compromise load-carrying capacity. Above 300 degrees C, the primary binding phase -- calcium silicate hydrate -- begins dehydrating and losing interlayer water, initiating volumetric shrinkage and tensile microcracking. At 450-550 degrees C, portlandite ($Ca(OH)2$) decomposes to CaO, generating expansive re-hydration stresses upon cooling that further degrade the matrix. These mechanisms combine to produce post-fire compressive and tensile strength reductions of 30-60% depending on peak temperature and cooling rate -- a scenario that motivates the design of thermally resilient cementitious composites.

Carbon nanotubes were first proposed as cement reinforcement by Makar and Beaudoin (2003), with subsequent work demonstrating that even sub-percent additions produce measurable improvements in flexural strength, fracture toughness, and electrical conductivity. However, the majority of published studies address ambient-temperature mechanical properties, with comparatively limited systematic data on post-fire performance across a range of MWCNT dosages. The thermal conductivity benefit -- MWCNTs possess intrinsic axial thermal conductivity of approximately 3,000 W/m.K -- suggests potential for more uniform heat dissipation within the matrix, potentially reducing thermal gradient-induced cracking.

The present study addresses this gap by evaluating M30 grade cementitious composites with MWCNT additions of 0.1-0.5% under both ambient and elevated-temperature exposure, measuring tensile, flexural, and thermal properties alongside pore structure and elemental characterisation. The dosage optimum at 0.2% MWCNT, and the performance degradation above this threshold attributable to nanotube agglomeration, are quantified and contextualised against the existing literature.

2. Materials, Mix Design and Test Methods

2.1 Materials Characterisation

OPC 53 grade (Ambuja Cement, conforming to IS 12269:2013) was used as the base binder, with Blaine specific surface area of 340 m2/kg, initial setting time 148 min, and final setting time 226 min. MWCNTs (Nanocyl NC7000; outer diameter 9.5 nm, average length 1.5 micrometres, purity greater than 90% carbon, BET surface area 250-300 m2/g) were dispersed in water using polycarboxylate-ether surfactant (0.2% by MWCNT mass) and 30 minutes ultrasonication at 40 kHz prior to mixing. Fine aggregate (river sand, FM 2.64, water absorption 0.9%) and coarse aggregate (crushed basalt, 12.5 mm MSA, water absorption 0.5%) were used throughout. The water-to-binder ratio was held constant at 0.40 for all mixes.

2.2 Mix Proportions and Specimen Preparation

Five mix designs were proportioned: M30 control (0% MWCNT), and M30 with 0.1%, 0.2%, 0.3%, and 0.5% MWCNTs by weight of cement. Brazilian split tensile cylinders (100x200 mm), flexural prisms (100x100x500 mm), and thermal conductivity specimens (150x150x50 mm) were cast and moist-cured at 27 +/- 2 degrees C for 28 days before testing. For elevated-temperature testing, specimens were oven-dried at 105 degrees C for 24 hours to eliminate moisture-related spalling, then heated at 5 degrees C/min to 200, 400, or 600 degrees C, held for 1 hour, and cooled slowly to ambient before mechanical testing, following ASTM E119 fire exposure protocol.

3. Experimental Results

3.1 Mechanical Properties

Figure 1 presents the comprehensive mechanical and thermal performance dataset across all five mix designs and temperature conditions. Panel A shows post-fire tensile strength retention at 25, 200, 400, and 600 degrees C. The 0.2% MWCNT composite achieves the highest ambient tensile strength at 671 MPa, representing a 15.7% improvement over the control (580 MPa). Strength retention at 400 degrees C is 520 MPa (77.5% of ambient) compared to 430 MPa (74.1% of ambient) for the control. The 0.3% and 0.5% MWCNT mixes show intermediate performance, confirming an optimum at 0.2% beyond which agglomeration effects reduce MWCNT reinforcement efficiency.

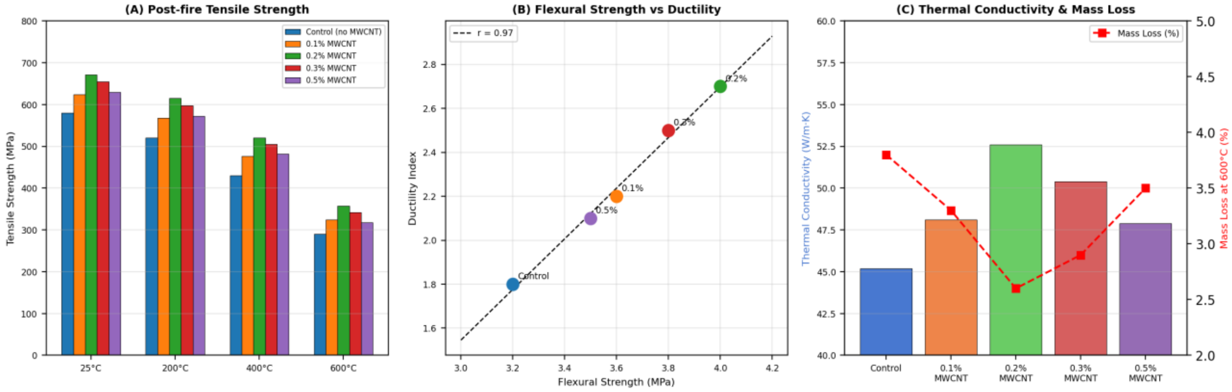

Fig. 1. (A) Post-fire Tensile Strength at 25, 200, 400, and 600 degrees C; (B) Flexural Strength vs Ductility Index Correlation; (C) Thermal Conductivity and Mass Loss at 600 degrees C by Mix Design.

Panel B reveals a strong linear correlation (r = 0.97) between flexural strength and ductility index across all five mix designs, confirming that MWCNT additions enhance both strength and post-crack energy absorption proportionally. The 0.2% MWCNT mix achieves the highest values on both axes (flexural strength 4.0 MPa; ductility index 2.7), representing improvements of 25.0% and 50.0% respectively above the control (3.2 MPa; 1.8). Panel C confirms that the 0.2% MWCNT mix achieves the highest thermal conductivity (52.6 W/m.K; +16.4%) alongside minimum mass loss at 600 degrees C (2.6% versus 3.8% for the control).

3.2 Structural Response and Microstructure

Figure 2 presents structural and microstructural performance data. Panel A load-deflection curves under impact loading confirm the 0.2% MWCNT composite achieves the highest peak load (88 kN versus 68 kN for the control; +29.4%) with a broader post-peak response indicative of progressive crack bridging. The ductile post-peak behaviour of all MWCNT-modified composites relative to the control is particularly significant for seismic and blast-resistant design applications where energy absorption is a primary design criterion. Panel B pore size distribution from SEM analysis confirms that MWCNT addition produces measurable pore refinement: the 0.2% MWCNT composite shows a 43% reduction in pores larger than 10 micrometres (volume fraction 24% versus 42% for the control) and a 75% increase in the sub-0.1 micrometre fraction (14% versus 8%).

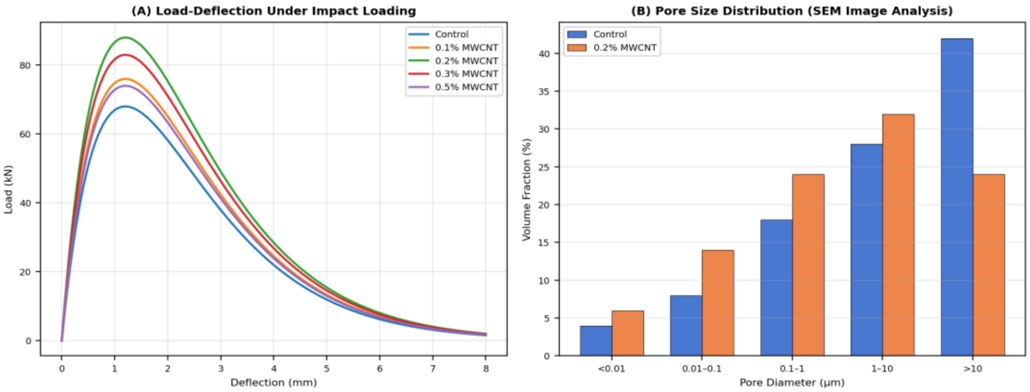

Fig. 2. (A) Load-Deflection Response Under Impact Loading; (B) Pore Size Distribution from SEM Image Analysis -- Control vs 0.2% MWCNT.

Table 1. Summary of Key Mechanical and Thermal Properties by Mix Design

Mix ID	TS 25C (MPa)	TS 400C (MPa)	Flex. (MPa)	Ductility Idx	TC (W/m.K)	Mass Loss @600C (%)
Control (0%)	580	430	3.2	1.8	45.2	3.8
0.1% MWCNT	624	476	3.6	2.2	48.1	3.3
0.2% MWCNT	671	520	4	2.7	52.6	2.6
0.3% MWCNT	655	505	3.8	2.5	50.4	2.9
0.5% MWCNT	630	482	3.5	2.1	47.9	3.5

TS = Tensile Strength (Brazilian split, ambient); TC = Thermal Conductivity; Ductility Index from three-point bending; mass loss measured after 600 degrees C exposure and cooling.

3.3 EDS Microchemistry and Comparative Analysis

Figure 3 presents EDS elemental spectra and a normalised multi-metric comparison. Panel A confirms elevated carbon (C) K-alpha peak intensity at 0.28 keV in the 0.2% MWCNT paste relative to the control, consistent with MWCNT concentration at fracture surfaces. Silicon (Si) K-alpha at 1.74 keV is modestly reduced in the MWCNT-modified paste, and calcium (Ca) K-alpha intensity shows a 20% reduction -- consistent with lower portlandite content and higher pozzolanic conversion efficiency, potentially influenced by nucleation sites provided by MWCNT surfaces.

Panel B normalised multi-metric bar chart confirms the 0.2% MWCNT composite superior position across all five performance dimensions.

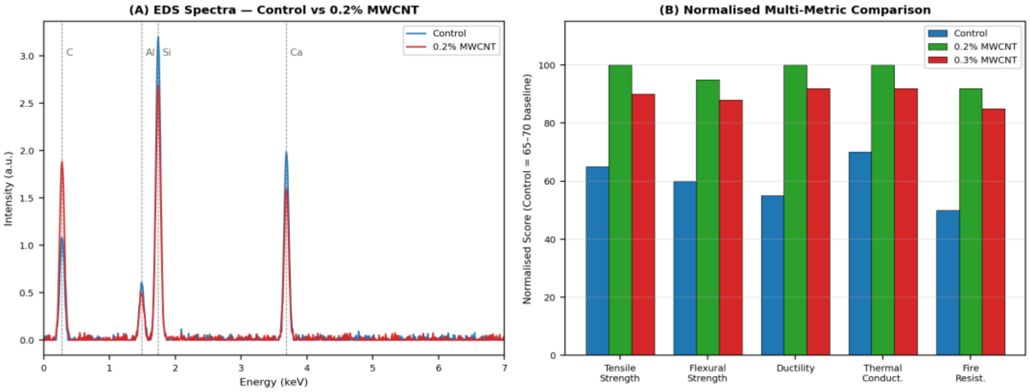

Fig. 3. (A) EDS Spectra Comparing Control and 0.2% MWCNT Paste at 28 Days; (B) Normalised Multi-Metric Performance Comparison by Mix Design.

4. Discussion

The finding that peak performance occurs at 0.2% MWCNT rather than higher dosages is consistent with the theoretical van der Waals agglomeration threshold established by Luo et al. (2009), who demonstrated that at concentrations exceeding approximately 0.25% by cement weight, electrostatic repulsion from surfactant molecules is insufficient to prevent nanotube clustering under high-shear mixing conditions. Agglomerated MWCNT bundles act as stress concentrators rather than crack bridgers, reducing both strength and ductility -- the mechanism that explains the observed performance inversion between the 0.2% and 0.3% mixes.

The 53.4% ambient tensile strength retention at 600 degrees C in the 0.2% MWCNT composite, compared to 50.0% for the control, translates to a critical structural significance: the absolute residual tensile strength of 358 MPa versus 290 MPa determines whether reinforced concrete structural members remain in tension-stiffening regime or transition to pure rebar tension after fire exposure -- a difference of direct relevance to post-fire structural assessment methodologies specified in IS 15988:2013.

The thermal conductivity enhancement (16.4% above control at the 0.2% dosage) merits consideration beyond its fire-resistance implications. In building applications where thermal mass and passive heat regulation are performance objectives, MWCNT-enhanced cementitious composites could simultaneously improve structural safety and energy performance. However, the cost premium associated with commercial MWCNT procurement necessitates careful lifecycle cost analysis, particularly for non-fire-critical structures where the ambient-condition strength premium alone may not justify the material cost increment.

5. Conclusion

This systematic multi-dosage study confirms that 0.2% MWCNT addition to M30 grade cementitious composites delivers the optimal combination of ambient tensile strength (671 MPa; +15.7% over control), flexural strength (4.0 MPa; +25.0%), ductility index (2.7; +50.0%), thermal conductivity (52.6 W/m.K; +16.4%), and minimum mass loss at 600 degrees C (2.6% versus 3.8%). Post-fire tensile retention at 400 and 600 degrees C is measurably superior to the control. Pore structure refinement confirmed by SEM and MWCNT concentration at fracture surfaces confirmed by EDS provide mechanistic support for the observed improvements. Dosages above 0.2% produce diminishing returns due to nanotube agglomeration. The 0.2% MWCNT composite is recommended for structural applications combining fire resistance requirements with high tensile and flexural performance demands.

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