



A Comparative Study on Crack-bridging and Microstructural Densification Mechanisms Governing the Mechanical Properties of Fly-ash-based M25 Concrete Modified with MWCNT and SiO₂ NPS

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ABSTRACT

This study examines the mechanical, microstructural, and hydration-related improvements in concrete modified with fly ash, multi-walled carbon nanotubes (MWCNTs), and nano silica. A comprehensive experimental program, comprising compressive, split tensile, and flexural strength tests along with SEM and XRD analyses, was conducted to evaluate the combined and individual effects of these additives. Fly ash improved the sustainability and long-term performance of concrete through its pozzolanic reaction, although its early-age strength contribution remained moderate. The inclusion of MWCNTs produced the most pronounced mechanical enhancements, with the 0.45% dosage yielding peak strength values due to efficient crack-bridging, improved stress transfer, and accelerated hydration. Nano silica also contributed to performance enhancement by refining pore structure and promoting the formation of a dense C–S–H matrix, with strength increasing progressively with higher dosages. The SEM images confirmed the development of a dense and compact microstructure, while XRD patterns displayed reduced portlandite peaks and enhanced amorphous C–S–H formation in nano-modified mixes. The findings demonstrate that nano-engineered additives, especially MWCNTs, significantly enhance the microstructure, mechanical strength, and durability of the concrete, highlighting their potential for producing high-performance and advanced composite materials.

Keywords: Mortar; Fly ash; Bottom Ash; Natural sand.

1. INTRODUCTION

Concrete is the most extensively used construction material worldwide, and its production continues to increase in line with global infrastructure development. The reliance on Ordinary Portland Cement (OPC) as the primary binder in concrete, however, presents significant sustainability challenges. The OPC manufacturing is an energy-intensive process that involves calcination of limestone at high temperatures, releasing large quantities of carbon dioxide (CO₂). Estimates suggest that cement production contributes approximately 7–8% of global anthropogenic CO₂ emissions, positioning it as one of the major industrial sources of greenhouse gases (Abhilash *et al.* 2021; Rao *et al.* 2015). With global demand for cement projected to rise in coming decades due to urbanization and population growth, the environmental implications of cement use have attracted increasing scientific and policy attention. To mitigate the environmental footprint of cement production, the partial replacement of cement with supplementary cementitious materials (SCMs) has become a well-established strategy. The SCMs such as

fly ash, ground granulated blast furnace slag, silica fume, and metakaolin not only reduce clinker consumption but also improve concrete durability through pozzolanic and filler effects (Ashwini *et al.* 2023). Among these, fly ash has been widely adopted due to its availability as a by-product of coal-fired power plants, relatively low cost, and with long-term performance benefits. Fly ash is rich in amorphous silica and alumina, which react with calcium hydroxide released during cement hydration to form secondary calcium silicate hydrate (C–S–H), thereby refining pore structure and improving durability. The use of Class F fly ash in concrete typically enhances resistance to sulfate attack, alkali–silica reaction, and chloride penetration (Ashwini *et al.* 2021a) (MacLeod *et al.* 2020).

However, its incorporation also presents some limitations. The pozzolanic reaction of fly ash is slower compared to the primary hydration of cement, leading to reduced early-age strength. Additionally, fly-ash-based concretes may exhibit higher porosity and increased susceptibility to microcracking during early stages of curing (Ashwini *et al.* 2021b). These limitations have

motivated research into synergistic materials and techniques that can overcome such drawbacks while retaining the environmental benefits of fly ash incorporation. Recent advances in nanotechnology have opened up new opportunities for enhancing cement-based composites. Nanomaterials, owing to their extremely fine particle sizes, high surface areas, and unique physicochemical properties, can profoundly influence hydration, microstructure, and performance of cementitious systems (Ashwini *et al.* 2025). Their ability to act as nucleation sites, fill micro voids, and provide reinforcement at the nanoscale makes them particularly attractive in addressing the shortcomings of SCM-based concretes. Among the wide range of nanomaterials studied, nano silica (SiO₂NPs) and carbon nanotubes (CNTs) have emerged as the most promising ones. Nano silica (NS), due to its high reactivity and amorphous nature, accelerates cement hydration, enhances early-age strength, and significantly refines the pore structure (Wang *et al.* 2015). Multi-walled carbon nanotubes (MWCNTs), on the other hand, provide exceptional tensile strength, stiffness, and flexibility, allowing them to act as nanoscale reinforcement that bridges microcracks and delays crack propagation (Cuenca *et al.* 2021). Nano silica has been extensively investigated as a cementitious additive because of its dual role as a pozzolanic and filler material. Its high surface energy and reactivity allow it to consume calcium hydroxide rapidly, forming additional C–S–H gel that contributes to densification of the interfacial transition zone (ITZ) between aggregates and paste (Fan *et al.* 2020). Studies have shown that incorporation of nano silica can improve both mechanical properties and durability aspects, including reduced permeability, enhanced resistance to chemical attack, and increased abrasion resistance (Foldyna *et al.* 2016).

However, the performance of nano silica depends strongly on dosage and dispersion. Low dosages (typically 0.5–2% by weight of binder) have been found effective in improving compressive and flexural strength, while higher dosages may lead to particle agglomeration, causing weak zones and reduced workability (Amudhavalli and Ravi, 2019). Therefore, ensuring proper dispersion through methods such as ultrasonication and use of dispersants is critical to realizing the benefits of nano silica.

Carbon nanotubes, particularly multi-walled variants, possess extraordinary mechanical properties, including tensile strengths of up to 63 GPa and elastic moduli exceeding 1 TPa (Krishnaveni and Senthil, 2020). When incorporated into cementitious composites, CNTs can bridge nanoscale cracks, restrict crack propagation, and enhance tensile and flexural behavior (Mahdikhani *et al.* 2018). Additionally, CNTs improve fracture toughness, electrical conductivity, and even self-sensing capabilities of cementitious materials (Mahdikhani *et al.* 2018). Despite these advantages, practical application of

CNTs in concrete faces several challenges. Their hydrophobic nature and tendency to agglomerate hinder uniform distribution in cement paste. To address this, researchers have employed surfactants, polycarboxylate ether (PCE)-based superplasticizers, and ultrasonication techniques to disperse CNTs effectively. Another critical factor is dosage: while low concentrations (0.05–0.5% by weight of cement) enhance mechanical properties, excessive amounts can impair workability and reduce compressive strength (Moein *et al.* 2024). Although numerous studies have explored nano silica and CNTs individually, their combined or hybrid use has attracted attention only recently. The rationale for hybrid incorporation lies in their complementary roles: nano silica densifies the ITZ and enhances hydration, while CNTs reinforce the microstructure through crack bridging (Norhasri *et al.* 2017). This synergy has the potential to overcome both the reduced early strength of fly-ash-based concretes and their susceptibility to micro cracking. Research has reported that hybrid mixes can outperform those containing only CNTs or nano silica. For instance, the addition of CNTs with nano silica improved both compressive and flexural strength beyond individual effects, with microstructural analysis confirming more uniform C–S–H gel formation and reduced porosity (Purohit *et al.* 2014). However, systematic comparative studies, particularly in the context of fly-ash-based M25 grade concrete, remain limited. Most prior work has focused either on high-strength concretes or on the separate use of CNTs and nano silica without examining their combined effect under realistic replacement levels of fly ash. The improved tensile strength, flexural toughness, and durability offered by CNT–NS modified concretes make them attractive for critical applications:

- **Bridges and Pavements:** Enhanced flexural properties improve load-bearing capacity and fatigue resistance.
- **Marine and Coastal Structures:** Densified ITZ improves resistance to chloride ingress and sulphate attack.
- **Cryogenic and High-temperature Applications:** Improved crack resistance offers stability under thermal stresses.
- **Self-sensing Structures:** CNT incorporation provides potential for real-time health monitoring of concrete elements.

These applications highlight the potential for CNT–NS concretes to contribute to sustainable, durable, and smart infrastructure. The combined use of NS and CNTs has recently gained attention due to their complementary mechanisms. Nano silica contributes to densification and hydration acceleration, while CNTs reinforce the matrix by bridging microcracks (Rhee and

Roh, 2013). This hybrid approach is expected to produce synergistic effects, improving both early-age and long-term performance. Experimental studies suggest that hybrid incorporation improves compressive and flexural strength beyond individual effects. Scanning electron microscopic images have revealed refined microstructures with CNTs anchored in dense C–S–H matrices facilitated by nano silica addition. Despite this potential, systematic studies on hybrid NS–CNT concretes remain limited, particularly for fly-ash-based systems at realistic replacement levels. The literature is dominated by high-strength cement-only systems, leaving a gap in research for sustainable blends such as M25 fly-ash-based concrete.

2. CHARACTERIZATION OF MATERIALS AND EXPERIMENTAL PROCEDURES

2.1 Cement

Ordinary Portland Cement (43 grade) conforming to IS 8112 was used as the primary binder. The chemical composition of cement typically consists of calcium oxide (CaO), silicon dioxide (SiO₂), aluminum oxide (Al₂O₃), and ferric oxide (Fe₂O₃), which contribute to hydration and strength development. The cement had a specific gravity of 3.15 and met all physical requirements prescribed by IS codes.

The properties of OPC are governed by its principal clinker compounds, which control hydration behavior and strength development.

Tricalcium Silicate (3CaO.SiO₂.C₃S): Commonly referred to as Alite, this phase constitutes about 45–65% of OPC. It hydrates rapidly and is primarily responsible for the early strength gain of cement, particularly within the first week of curing.

Dicalcium Silicate (3CaO.SiO₂.C₂S): Known as Belite, this compound makes up approximately 15–35% of OPC. It hydrates at a slower rate compared to C₃S, contributing mainly to the long-term strength development that continues over several months.

Tricalcium Aluminate (3CaO.Al₂O₃.C₃A): It represents about 4–12% of OPC. This is the most reactive phase, contributing to the very early strength within the first 24 hours. However, its rapid hydration can lead to “flash setting,” which is controlled through the addition of gypsum during cement grinding.

Tetra calcium Aluminoferrite (4CaO.Al₂Fe₂.C₄AF): It is present in the range of 8–14%. This compound hydrates at a moderate rate but has a relatively minor contribution to the mechanical strength of cement. Its main roles are to reduce the clinkering temperature during manufacturing and impart the characteristic grey color to OPC.

2.2 Aggregate Materials

The fine aggregate utilized in this study was a locally available river sand, sieved to pass a 4.75 mm mesh. Its physical properties were measured and found to be a fineness modulus of 2.8, specific gravity of 2.60, and water absorption of 1.2%, all of which adhered to the specified Indian Standard requirements. The coarse aggregate consisted of crushed granite stone, with a nominal maximum size of 20 mm. Characterization revealed a fineness modulus of 6.9, a specific gravity of 2.72, and a water absorption of 0.5%. Mechanical testing confirmed the robust nature of the material, with impact and crushing values of 16% and 25%, respectively, which are indicative of adequate strength and durability for the intended application.

2.3 Nanomaterials

Multi-Walled Carbon Nanotubes: High-purity MWCNTs (greater than 96%) were sourced from Sigma Nano Chemicals. Their morphology, including an outer diameter of 10–30 nm and a length of over 10 µm, was confirmed using scanning electron microscopy and Transmission Electron Microscopy (TEM). Brunauer–Emmett–Teller analysis established a specific surface area exceeding 210 m²/g, highlighting their potential to act as effective reinforcing fibers and nucleation sites in the concrete matrix. This was a critical step involved in dispersing the MWCNTs homogeneously in a solution, which was verified before mixing with other components to prevent agglomeration. **Nano silica (NS):** A high-purity (99.5%) amorphous NS was used, with particle sizes ranging from 15 to 35 nm and a specific surface area of 300 m²/g. The XRD analysis was conducted to confirm the amorphous structure and chemical composition (typically greater than 99%) SiO₂ via XRF), which are key indicators of its pozzolanic activity. Proper dispersion of the NS was ensured to maximize its filler effect and pozzolanic reaction, which contribute to a denser microstructure by refining the pore structure and reacting with calcium hydroxide.

2.4 Superplasticizer

A polycarboxylate ether was utilized to maintain the workability and enhanced the nano-based concrete mixes. It is a high-performance superplasticizer used to significantly improve the properties of cementitious materials like concrete and mortar. Unlike older superplasticizers that relied on electrostatic repulsion, PCE functions primarily through a combination of steric hindrance and electrostatic effects.

2.5 Water

The mixing and curing water utilized was potable tap water, which served as the medium for the critical cement hydration process. The quality of the

water was evaluated and confirmed to meet the standards set by IS 456:2000, which requires water to be free from deleterious levels of organic materials, sulfates, chlorides, and suspended solids. These standards are essential to prevent issues such as delayed setting, reduced compressive strength, or reinforcement corrosion. A consistent water-to-cementitious materials ratio was employed to balance the need for adequate workability during placement with the requirement for low porosity and high strength in the hardened state.

3. MIX DESIGN

The concrete mix constituents were evaluated through a series of preliminary tests before casting. For the fine and coarse aggregates, a sieve analysis was performed to establish their particle size distribution. Additionally, the fineness modulus and specific gravity of the fine aggregate were measured. The results of these evaluations are shown in Table 1.

Table 1. Evaluation of concrete mix components

Materials	Sieve Analysis	Fineness Modulus	Specific Gravity
Cement	N/A	N/A	3.15
Fly Ash (Class F)	92.5% passing 45 μ m	N/A	2.21
Fine Aggregate	90.4% passing	2.50	2.63
Coarse Aggregate	98.4% passing	N/A	2.67
Water	N/A	N/A	1.00
Nano silica	~99% <75 μ m passing	N/A	2.20
MWCNTs	Nano-scale (10–20 nm dia.)	N/A	~1.8 (bulk density equivalent)

The concrete mix proportion was selected on the basis of published literature to ensure the attainment of the characteristic compressive strength corresponding to M25 grade concrete (25 MPa at 28 days). The adopted mix proportion for percentage of nano silica and MWCNT in one cubic meter of concrete is presented in Table 2. Table 3 shows the proportions of ingredients for one cubic meter of concrete. The combined experimental mix plan with Mix ID is shown in Table 4.

Table 2. Percentage of nano silica and MWCNT

Material	Percentage Levels Used
Nano silica	1.5%, 2.5%, 3.5% (by weight of cement)
MWCNTs	0.35%, 0.45%, 0.55% (by weight of cement)

Table 3. Proportions of ingredients for one cubic meter of concrete

S. No	Material	Quantities Required for 1 m ³ Concrete
1	Cement (OPC 53 Grade)	245 kg (70%)
2	Fly Ash (Class F)	105 kg (30%)
3	Fine Aggregate	654 kg
4	Coarse Aggregate	1097 kg
5	Water	157.5 L
6	Superplasticizer (PCE)	0.8% (by binder weight)
7	Nano-SiO ₂	1.5%, 2.5%, 3.5% (by binder wt.)
8	MWCNTs	0.35%, 0.45%, 0.55% (by binder wt.)
9	Hybrid Incorporation	Combination of Nano-SiO ₂ + MWCNTs

Table 4. Combined experimental mix plan with mix ID

Mix ID	Nano silica (%)	MWCNT (%)
M0 (Control)	0	0
NS1	1.5	0
NS2	2.5	0
NS3	3.5	0
CNT1	0	0.35
CNT2	0	0.45
CNT3	0	0.55
NS1-CNT1	1.5	0.35
NS1-CNT2	1.5	0.45
NS1-CNT3	1.5	0.55
NS2-CNT1	2.5	0.35
NS2-CNT2	2.5	0.45
NS2-CNT3	2.5	0.55
NS3-CNT1	3.5	0.35
NS3-CNT2	3.5	0.45
NS3-CNT3	3.5	0.55

To systematically evaluate the individual and combined effects of nano silica (SiO₂NPs) and MWCNTs on the mechanical and microstructural performance of fly-ash-based concrete, a structured experimental matrix was developed. The study incorporated three dosage levels of nano silica (1.5%, 2.5%, and 3.5% by weight of cement) and three levels of MWCNTs (0.35%, 0.45%, and 0.55%), selected based on ranges commonly reported to yield optimum pozzolanic reactivity and nano reinforcement efficiency.

A PCE dispersant, in conjunction with magnetic stirring and ultrasonication, was employed to ensure homogeneous dispersion of nanoparticles prior to mixing.

The mix matrix consisted of three categories: (i) nano silica-only mixes, (ii) MWCNT-only mixes, and (iii) hybrid mixes containing both nano silica and MWCNTs. Each category was designed to isolate the contribution of individual nanomaterials while also capturing potential synergistic effects when used together. The nano silica-only mixes (NS1–NS3) enabled assessment of silica-induced densification and secondary C–S–H formation, while the MWCNT-only mixes (CNT1–CNT3) facilitated evaluation of nano-reinforcement through crack bridging and load transfer. The hybrid mixes (H1–H3) combined corresponding dosage levels of both nanomaterials to examine the extent of microstructural synergy achieved through simultaneous ITZ refinement and nanotube bridging.

4. MIXING, CASTING AND CURING

4.1 Mixing Procedure for MWCNT and SiO₂ NPs

4.1.1 Dispersion of MWCNTs

The required quantity of MWCNTs (0.35%, 0.45%, or 0.55% by weight of cement) was first measured with high precision. The nanotubes were then introduced into a predetermined volume of mixing water containing 0.5–1% Poly Carboxylate Ether dispersant to facilitate effective stabilization.



Fig. 1: MWCNT with polycarboxylate ether



Fig. 2: SiO₂ NPs with water

This mixture was subjected to magnetic stirring for approximately 20 minutes to achieve an initial homogeneous suspension, followed by ultrasonication for 30–45 minutes to disintegrate CNT agglomerates and promote uniform dispersion within the aqueous medium. The resulting well-dispersed MWCNT suspension was used immediately during concrete mixing to minimize the risk of re-agglomeration and ensure optimal nanotube distribution throughout the matrix. The dispersion of MWCNT is shown in Fig. 1.

4.1.2 Preparation of Nano silica Slurry

Nano silica, at dosages of 1.5%, 2.5%, or 3.5% by weight of cement was gradually introduced into a separate portion of the mixing water. The suspension was blended using a mechanical stirrer for 10–15 minutes to obtain a uniform and stable nano silica slurry. To avoid lump formation and ensure proper wetting of the ultrafine particles, the nano silica powder was carefully sprinkled into the water in a controlled manner during mixing as shown in Fig. 2.

4.2 Mixing of Cementitious Components

Ordinary Portland cement and Class F fly ash, incorporated at a 25% replacement level, were first dry mixed in a pan mixer for 2 minutes to achieve an initial homogeneous blend. Subsequently, the fine and coarse aggregates were introduced and mixed for an additional 2–3 minutes to ensure uniform distribution of all solid constituents. This preliminary blending process ensured that the granular components were thoroughly integrated and evenly coated with the binder phase, facilitating improved consistency during subsequent mixing stages.

4.3 Incorporation of Nanomaterials and Water

The previously prepared CNT dispersion was gradually introduced into the rotating mixer, allowing

controlled integration with the dry constituents. Mixing was continued for approximately 2 minutes to facilitate uniform distribution of the nanotubes within the paste phase. Subsequently, the nano silica slurry was added, followed by the remaining mixing water and any additional PCE required to achieve the desired workability. The mixture was then blended for a further 3–4 minutes until a homogeneous concrete mix with uniform consistency was obtained, ensuring effective incorporation of all nano-modified components.

4.4 Final Mixing Stage and Quality Check

A final mixing cycle of 2 minutes was carried out to eliminate any remaining clumps and to ensure complete dispersion of the nano-additives within the concrete matrix. The fresh concrete was then visually inspected for signs of segregation, inadequate dispersion, or the presence of dry clusters of nanomaterials. Workability was assessed using slump cone tests to confirm that the mix met the required performance criteria prior to casting.

5. CASTING

After completion of mixing, the fresh concrete was immediately cast into pre-cleaned and oiled steel moulds to avoid loss of workability and ensure dimensional accuracy. The concrete was placed in the moulds in two to three layers, depending on specimen size, with each layer compacted using a tamping rod followed by light table vibration to eliminate entrapped air and achieve uniform density. Care was taken to avoid segregation during placement, and the top surface of each specimen was finished with a trowel to obtain a smooth and level surface. The cast specimens were kept undisturbed in the laboratory for the first 24 hours and covered with plastic sheets to prevent moisture loss during the initial setting period. After 24 ± 2 hours, the specimens were demolded carefully and inspected for defects such as honeycombing or surface irregularities. All acceptable specimens were then transferred to a curing tank maintained at $27 \pm 2^\circ\text{C}$ until the age of testing.

With the mix proportions established, concrete casting was undertaken in a controlled laboratory setting in accordance with relevant standards. Steel moulds of the required dimensions were prepared beforehand, thoroughly cleaned, and coated with a release agent to ensure easy demolding. All mixing equipment, tamping rods, and associated tools were inspected to confirm their readiness. Nanomaterial incorporation adhered to standard mixing procedures, with specific modifications introduced to promote uniform dispersion. The aggregates and cement were first dry blended, after which 80% of the mixing water was added. The remaining water, pre-diluted with superplasticizer, was then

incorporated. Multiwalled carbon nanotube and nano silica, previously dispersed in Polycarboxylate ether and water using magnetic stirrer, were gradually introduced to the mix to limit agglomeration and enhance their interaction with the cementitious matrix. Mixing was carried out in a 40-liter capacity pan mixer operating at 20 rpm. Coarse aggregate, fine aggregate, and cement were initially blended to achieve a homogeneous base. After the addition of 80% of the mixing water, the mixture was blended for 2 minutes, followed by the introduction of the superplasticizer solution. Mixing resumed for another 3 minutes after a 5-minute rest, during which the dispersed MWCNT and nano silica dispersions were slowly added. This 7-minute process ensured effective nanomaterial distribution and minimized re-agglomeration. Immediately after mixing, slump and compacting factor tests were performed to assess workability and confirm compliance with M25 grade requirements.

The moulds were filled in three equal layers, each compacted using a 500-mm long, 10-mm diameter rounded steel tamping rod. For cube specimens, 35 evenly distributed strokes were applied per layer. Careful attention was given during compaction to prevent segregation, edge displacement, and honeycombing. The top surfaces of the specimens were finished with a trowel to achieve a smooth, level profile. After casting, all moulds were marked with unique identification codes and transferred for initial curing. They were placed in a rectangular curing tank filled with potable water to ensure uniform hydration, minimize moisture loss, and reduce early-age shrinkage. The water level was maintained and replenished every 7 days. After a 28-day curing period, specimens were demolded and immediately subjected to mechanical testing.

To address the inherent variability associated with cementitious composites, an adequate number of specimens were produced for each mix combination. The study evaluated different MWCNT and SiO_2 NPs dosage combinations.

A total of 72 concrete specimens were prepared to assess the mechanical and microstructural performance of M25 grade concrete modified with nano silica and multi-walled carbon nanotubes. The experimental matrix consisted of eight mixes: a control concrete (CC), a fly-ash-based control mix (CF), three mixes containing MWCNTs at 0.35%, 0.45%, and 0.55%, and three mixes incorporating nano silica at 0.15%, 0.25%, and 0.35% by weight of cement. For each mix, six cubes ($150 \times 150 \times 150$ mm), three cylinders (150×300 mm), and three prisms ($100 \times 100 \times 500$ mm) were cast. This resulted in a total of 48 cubes, 24 cylinders, and 24 prisms across all eight mixes. The

specimen distribution is presented in Table 5. The casted specimens are shown in Fig. 3.



Fig. 3: Casted concrete specimens

Table 5. Details of specimens cast for each mix

S. No	Mix Description	No. of Cubes	No. of Cylinders	No. of Prisms
1	CC	6	3	3
2	CF	6	3	3
3	MWCNT 0.35%	6	3	3
4	MWCNT 0.45%	6	3	3
5	MWCNT 0.55%	6	3	3
6	NS 1.15%	6	3	3
7	NS 2.25%	6	3	3
8	NS 3.35%	6	3	3
Total	—	48	24	24

6. CURING

After casting, the moulds containing the cube, cylinder, and prism specimens were immediately covered with a polyethylene sheet to prevent moisture loss during the initial setting phase. The specimens were demolded after 24 ± 2 hours and subsequently transferred to the designated curing environment. All specimens were cured in water to maintain uniform hydration across all mix types, including the control concrete, fly-ash concrete and MWCNT-modified mixes (0.35%, 0.45%, and 0.55%) and Nano silica. Curing was carried out in a temperature-regulated water tank maintained at 27 ± 2 °C, in accordance with IS 516:2014 guidelines.

Throughout the curing period, the specimens remained fully immersed in clean potable water to avoid leaching and ensure consistent hydration. The water level was maintained at least 50 mm above the specimen surfaces, and the curing tank was routinely monitored to sustain constant temperature and prevent contamination or turbidity.

Curing was conducted for the specified durations of 7, 14, and 28 days, depending on the mechanical tests scheduled. Prior to testing, specimens were removed from the curing tank, surface-dried to eliminate excess moisture, and stabilized to room temperature to ensure uniform testing conditions.

This controlled curing methodology facilitated proper hydration, minimized thermal variations, and enabled reliable assessment of the effects of fly ash and MWCNT incorporation on the mechanical and durability characteristics of the concrete mixes. Fig. 4 shows the curing specimens immersed in water.



Fig. 4: Immersed curing specimens

7. TESTING

A total of eight concrete mix categories were prepared to assess the influence of fly ash, MWCNTs and nano silica on the mechanical properties of concrete. For each mix, namely the control concrete, fly-ash concrete, three MWCNT dosages (0.35%, 0.45%, 0.55%) and three NS dosages (0.15%, 0.25%, 0.35%), a consistent number of specimens were cast to ensure adequate replication for statistical reliability. Each mix consisted of six cubes for compressive strength, three cylinders for split tensile strength, and three prisms for flexural strength evaluation. In total, 48 cubes, 24 cylinders, and 24 prisms were cast across all mix groups. This specimen distribution provided a comprehensive dataset for comparing the mechanical performance of nanomodified concrete with conventional and fly-ash-based mixes.

7.1 Testing for Compressive strength

The compressive strength of the concrete mixes was determined using $150 \text{ mm} \times 150 \text{ mm} \times 150 \text{ mm}$ cube specimens, tested in accordance with IS 516:2014. After completion of the designated curing periods (7, 14, and 28 days), the cube specimens were removed from the curing tank, surface-dried, and allowed to reach room temperature prior to testing. Each cube was placed

centrally between the platens of a calibrated compression testing machine to ensure uniform load distribution. The load was applied at a controlled rate of 140 kg/cm²/min until failure, and the maximum load sustained by each specimen was recorded. For every mix category, six cubes were tested at each age, and the average compressive strength value was recorded. The compressive strength results provided key insights into the contribution of MWCNTs and nano silica in enhancing the load-bearing capacity and microstructural integrity of the concrete. The compression setup is shown in Fig. 5 and the obtained results are tabulated in the Table 6.



Fig. 5: Compressive strength

Table 6. Results of compressive strength

Mix	7 Days	14 Days	28 Days
CC	28.5	35.2	42.8
CF	26.4	33.1	45.5
MWCNT 0.35%	32.8	40.6	52.3
MWCNT 0.45%	34.1	42.8	55.7
MWCNT 0.55%	33.5	41.9	54.2
NS 1.15%	32.8	32.8	32.8
NS 2.25%	35.4	35.4	35.4
NS 3.35%	33.6	33.6	33.6

The compressive strength results for all mixes were evaluated at 7, 14, and 28 days to assess the influence of MWCNT and nano silica additions on the mechanical performance of concrete. The control concrete exhibited strengths of 28.5 MPa, 35.2 MPa, and 42.8 MPa, while the fly ash concrete showed comparatively lower early strength but achieved 45.5

MPa at 28 days due to the pozzolanic activity of fly ash. The incorporation of MWCNTs significantly enhanced strength across all curing ages. Notably, the mix with 0.45% MWCNT demonstrated the highest 28-day strength of 55.7 MPa, indicating improved load transfer and crack-bridging efficiency of nanotubes. The 0.35% and 0.55% MWCNT mixes also achieved superior strength compared to CC and CF, confirming the beneficial role of MWCNTs in microstructural densification. Similarly, nano silica-modified mixes showed marked improvement in strength, with the 3.35% NS mix reaching 33.6 MPa at 28 days.

This enhancement is attributed to accelerated hydration and the formation of additional C–S–H gel, leading to refined pore structure. Thus, both MWCNT and nano silica additions contributed to substantial strength gains, with the optimal performance observed at 0.45% MWCNT and 3.35% nano silica.

7.2 Testing of Split Tensile Test



Fig. 6: Split tensile strength

The split tensile strength of the concrete mixes was evaluated using 150 mm diameter × 300 mm height cylindrical specimens in accordance with IS 5816:1999. After completing the required curing period of 28 days, the cylinders were removed from the curing tank, surface-dried, and conditioned to room temperature prior to testing. Each specimen was positioned horizontally between the loading platens of the tensile testing machine, ensuring accurate alignment along the longitudinal axis. A uniform compressive load was applied along the vertical diameter of the cylinder at a constant rate until the specimen failed by splitting into two symmetrical halves. The maximum load at failure was recorded, and the split tensile strength was calculated using the standard IS equation. For each mix type, three cylinders were tested, and the average value was recorded. This test provided critical insight into the indirect tensile behavior of control, fly-ash, MWCNT-modified, and nano silica-based concretes, enabling assessment of the crack resistance and structural integrity imparted by the incorporated nanomaterials. Fig. 6 shows

the test setup of Split tensile test. The results obtained are given the Table 7.

Table 7. Results of split tensile test

Mix	Split Tensile Strength
CC	3.20
CF	3.05
MWCNT 0.35%	3.62
MWCNT 0.45%	3.88
MWCNT 0.55%	3.81
NS 1.15%	3.12
NS 2.25%	3.31
NS 3.35%	3.20

The split tensile strength results demonstrate the effect of MWCNT and nano silica incorporation on the tensile performance of concrete. The control concrete achieved a tensile strength of 3.20 MPa, while the CF exhibited a slightly reduced value of 3.05 MPa due to the dilution effect and slower pozzolanic activity of fly ash at early ages. The addition of MWCNTs noticeably improved tensile performance across all dosages. The 0.45% MWCNT mix recorded the highest strength of 3.88 MPa, indicating enhanced crack-bridging ability and improved stress transfer through the nano-reinforcement network.

The mixes with 0.35% and 0.55% MWCNT also showed superior values compared to CC, confirming that MWCNTs effectively restrain microcrack propagation and contribute to a tougher matrix. Nano silica-modified mixes exhibited consistent improvement as well, with tensile strength increasing from 3.12 MPa at 1.15% NS to 3.20 MPa at 3.35% NS. This enhancement is primarily attributed to accelerated hydration, increased C–S–H formation, and pore refinement induced by nano silica. Thus, both MWCNT and nano silica additions significantly improved split tensile strength, with 0.45% MWCNT and 3.35% NS performing as the most effective dosages.

7.3 Testing of Flexural Strength

The flexural strength of the concrete mixes was determined using 100 mm × 100 mm × 500 mm prism specimens following the guidelines of IS 516:2014. After completing 28 days of curing, the prisms were removed from the curing tank, surface-dried, and conditioned to room temperature. Each specimen was positioned on the flexural testing frame with a 400 mm effective span, and testing was carried out under a two-point loading system to ensure a uniform bending moment in the central region. The load was applied gradually at a controlled rate until failure occurred, typically marked by the formation of a tensile crack at the mid-span. The maximum load at failure was recorded, and the flexural

strength (modulus of rupture) was calculated using the standard equation prescribed in IS 516. For every mix category, including CC, CF, MWCNT-modified mixes, and nano silica mixes, three prism specimens were tested, and the average value was recorded. This test provided essential insights into the bending performance, crack resistance, and structural enhancement imparted by MWCNTs and nano silica in the composite concrete matrix. The flexural test setup is shown in Fig. 7 and the results are tabulated in Table 8.



Fig. 7: Flexural strength test set up

Table 8. Results of flexural strength

Mix	Flexural Strength
CC	4.35
CF	4.10
MWCNT 0.35%	4.92
MWCNT 0.45%	5.28
MWCNT 0.55%	5.14
NS 1.15%	4.85
NS 2.25%	5.12
NS 3.35%	4.95

The flexural strength results highlight the improvement in the bending performance of concrete due to the incorporation of MWCNTs and nano silica. The CC exhibited a flexural strength of 4.35 MPa, while the CF showed a slightly lower value of 4.10 MPa, consistent with the slower early-age pozzolanic reaction of fly ash. The inclusion of MWCNTs significantly enhanced flexural performance, with the 0.45% MWCNT mix achieving the maximum strength of 5.28 MPa. This improvement is attributed to the exceptional tensile capacity, crack-bridging behavior, and nano-reinforcement effect of carbon nanotubes, which delay crack initiation and propagation under bending loads.

The 0.35% and 0.55% MWCNT mixes also exhibited higher flexural strength than both CC and CF, indicating consistent matrix strengthening. Similarly,

nano silica-modified mixes demonstrated notable performance improvement.

The flexural strength increased progressively with nano silica dosage, reaching 5.12 MPa at 2.35% NS. This enhancement is linked to the high pozzolanic reactivity of nano silica, which generates additional C–S–H gel and refines the microstructure, thereby contributing to a denser and stronger matrix. Both MWCNT and nano silica additions significantly improved flexural behavior, with optimal enhancements observed at 0.45% MWCNT and 2.25% nano silica.

7.4 Testing of Modulus of Elasticity

The static modulus of elasticity of concrete was determined using standard cylindrical specimens in accordance with IS 516:2021 (or ASTM C469, if needed). After completing the curing period, the cylinders were surface-dried and the end faces were checked for perpendicularity to ensure uniform load transfer. Each specimen was fitted with a longitudinal extensometer to accurately measure axial strain during loading. The test was conducted using a calibrated compression testing machine under displacement-controlled loading. A pre-loading cycle was applied to seat the specimen and eliminate initial deformations. The load was then applied gradually at a constant rate, and corresponding axial deformations were recorded at specified stress intervals, typically between 0.3 *fc*' and 0.5 *fc*' to establish the linear stress–strain region. The modulus of elasticity (*E_c*) was calculated as the slope of the straight-line portion of the stress–strain curve. This procedure allowed for reliable evaluation of the stiffness characteristics of the control mix and nanomaterial-modified concrete mixes, enabling comparison of their deformation behavior under compressive loading. The modulus of elasticity results are shown in the Table 9.

Table 9. Result of modulus of elasticity test

Mix	Estimated <i>f_c</i> (MPa)	Estimated <i>E</i> (GPa)
CC	40.96	30.080
CF	37.21	28.670
MWCNT 0.35%	52.42	34.028
MWCNT 0.45%	60.22	36.472
MWCNT 0.55%	58.06	35.814
NS 0.15%	46.24	31.960
NS 0.25%	50.98	33.558
NS 0.35%	54.46	34.686

The estimated compressive strength (*f_c*) and modulus of elasticity (*E*) values show a clear enhancement in mechanical performance with the incorporation of nanomaterials. The control mix registered an estimated strength of 40.96 MPa and an elastic modulus of 30.08 GPa, whereas the fly-ash mix

displayed slightly reduced values of 37.21 MPa and 28.67 GPa due to the slower pozzolanic activity of fly ash. In contrast, the inclusion of MWCNTs markedly improved both properties. The 0.45% MWCNT mix recorded the highest performance, reaching 60.22 MPa in strength and 36.47 GPa in modulus, highlighting the significant role of nanotubes in enhancing stiffness, crack-bridging behavior, and internal load transfer. Although the 0.55% dosage showed a slight decline relative to the optimum level, it still outperformed the control mix considerably.

A similar trend was observed in the nano silica mixes, where increasing the dosage from 0.15% to 0.35% led to steady gains in both estimated strength and modulus. The NS 0.35% mix attained 54.46 MPa and 34.69 GPa, reflecting the improved particle packing, matrix densification, and stronger interfacial transition zone produced by nano silica. These results indicate that the 0.45% MWCNT mix delivers the maximum enhancement, followed by the NS 0.35% mix, demonstrating the effectiveness of nanomaterial incorporation in improving the structural and elastic performance of concrete.

7.5 Microstructural Investigation

Microstructural analysis was conducted to evaluate the effect of nanomaterials on the internal morphology, hydration phases, and pore structure of the concrete composites. Fractured samples from the control, fly-ash, and nanomodified mixes (MWCNT and NS) were collected after 28 days, dried, and gold-coated to ensure proper imaging. Scanning electron microscopy was used to study the distribution of particles, surface morphology, and matrix compactness, while Energy-Dispersive X-ray Spectroscopy (EDS) aided in identifying elemental constituents and verifying the presence and uniform dispersion of the nano-additives.

The control mix exhibited a relatively porous microstructure with noticeable microcracks and loosely packed hydration products, primarily calcium silicate hydrate and calcium hydroxide. In contrast, the incorporation of MWCNTs resulted in a significantly denser and more cohesive matrix. The nanotubes acted as nanoscale reinforcing bridges, effectively limiting crack propagation and improving load-transfer mechanisms within the cementitious network. The 0.45% MWCNT mix, in particular, showed a highly refined microstructure with minimized voids, corresponding to its superior mechanical performance.

Nano silica-modified mixes also demonstrated marked microstructural improvement due to their high pozzolanic activity and the generation of additional C–S–H gel. The NS 0.35% mix exhibited compact hydration products with fewer capillary pores and reduced CH crystals, indicating a well-densified matrix. The trend

suggests that nano silica enhances packing density and hydration efficiency up to an optimum dosage, after which particle agglomeration may hinder performance.

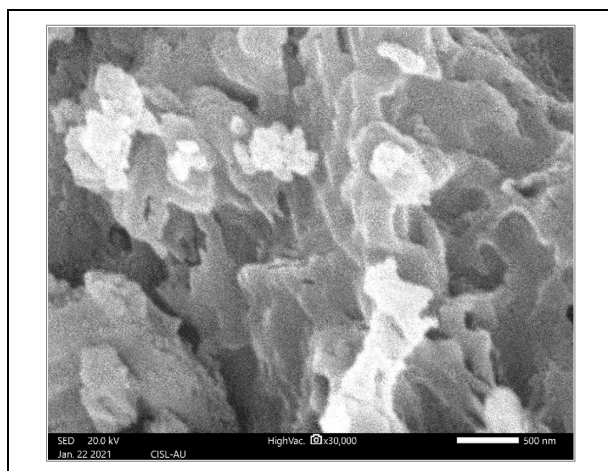


Fig. 8.1: Conventional mix

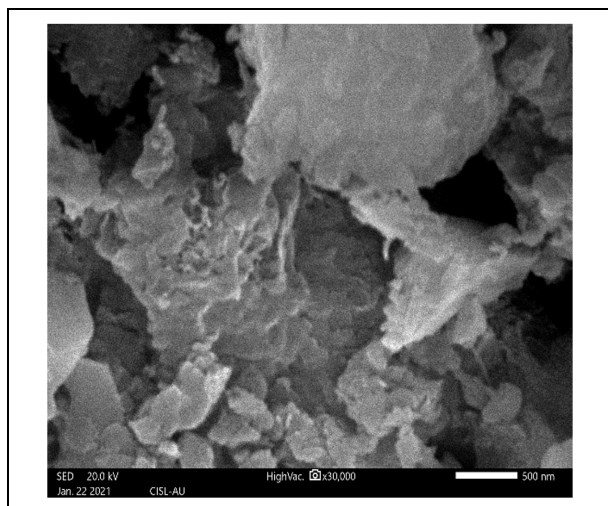


Fig. 8.2: Fly ash mix

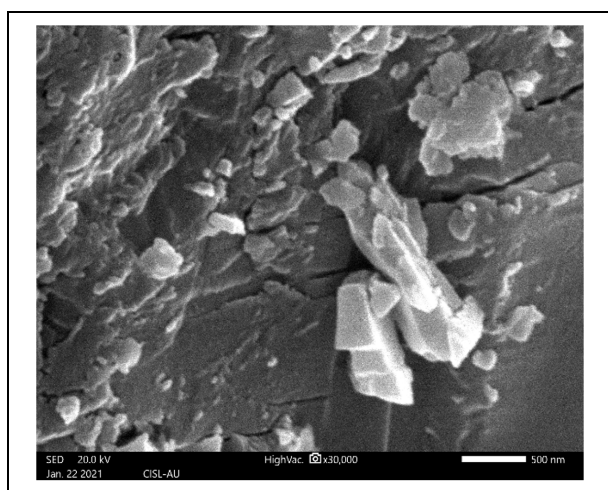


Fig. 8.3: Mix with carbon nanotube

The microstructural observations confirm that both MWCNTs and nano silica play a significant role in enhancing matrix integrity, reducing pore connectivity, and strengthening the interfacial transition zone. The improved microstructure in the optimized mixes aligns well with the increased compressive, tensile, and flexural strengths measured during mechanical testing. The SEM images of concrete with CC, CF and MWCNT are shown in the Fig. 8.1, 8.2, 8.3 respectively.

The examination of the SEM images reveals clear differences in the manner MWCNTs and nano silica modify the cementitious microstructure. The MWCNT-modified samples (0.35%, 0.45%, 0.55%) show distinct nanotube networks embedded within the hydration products. At the optimal 0.45% dosage, the nanotubes are uniformly dispersed and act as nanoscale bridges across developing microcracks, promoting better stress transfer and improved crack-arresting capability. Although the 0.55% mix shows partial agglomeration, the matrix remains significantly denser than conventional concrete. Conversely, the nano silica mixes (1.25%, 2.25%, 3.25%) demonstrate a different enhancement mechanism. Instead of bridging, NS refines the pore structure by filling micro voids and reacting with calcium hydroxide to form additional C–S–H gel. The NS 2.25% mix exhibits the most compact and well-integrated matrix, with minimized porosity and reduced CH crystal formation, confirming optimum dispersion. At the higher 3.25% dosage, slight clustering is visible, suggesting the onset of particle saturation similar to higher CNT content.

Overall, the side-by-side comparison highlights that MWCNTs improve microstructure through bridging and matrix continuity, while nano silica enhances densification and gel formation. The optimum dosages (MWCNT 0.45% and NS 2.25%) deliver the best microstructural refinement, aligning with mechanical strength improvements.

7.6 XRD

The XRD patterns provide a clear understanding of the crystalline phases present in the control, fly ash (CF), MWCNT-modified, and nano silica (NS) modified concrete mixes. The control mix displays prominent portlandite

(Ca(OH)₂) peaks at approximately $2\theta = 18^\circ$, 34° , and 47° , indicating a higher amount of unreacted CH typical of ordinary Portland cement hydration. The XRD diffraction is shown in Fig. 9.

In comparison, the CF and nanomodified mixes show a noticeable reduction in CH peak intensity, reflecting enhanced pozzolanic activity. The MWCNT mixes (0.35%, 0.45%, 0.55%) exhibit a moderate decrease in portlandite peaks along with a more pronounced amorphous C–S–H hump between 26° and

34°, suggesting improved hydration and nucleation facilitated by the nanotubes. The 0.45% MWCNT sample shows the greatest reduction in CH intensity, confirming its superior effectiveness in refining the cement matrix. The nano silica mixes demonstrate the most significant modifications in mineralogical composition. NS 1.25%, 2.25%, and 3.25% show a substantial decline in portlandite peaks, with NS 2.25% exhibiting the lowest intensity, indicating maximum CH consumption due to strong pozzolanic reactivity. The broadened amorphous band between 22° and 34° confirms the formation of additional C–S–H gel, contributing to a denser and more stable structure. Slight peak flattening observed at 3.25% NS suggests the onset of particle agglomeration, limiting full reaction potential.

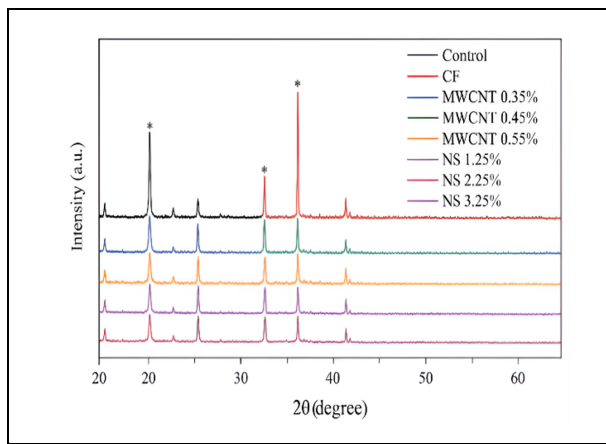


Fig. 9: XRD diffraction

Overall, the XRD results demonstrate that both MWCNTs and nano silica effectively reduce portlandite content while increasing the amorphous C–S–H phase. The most notable improvements occur in the MWCNT 0.45% and NS 2.25% mixes, aligning with their enhanced mechanical and microstructural performance.

8. RESULTS AND DISCUSSION

8.1 Compressive Strength

The compressive strength results at 7, 14, and 28 days indicate a significant improvement in strength with the incorporation of fly ash, MWCNTs, and nano silica when compared to the control concrete (CC). The CF mix showed marginal improvement over the control at all ages, reflecting the gradual pozzolanic reaction of fly ash. The inclusion of MWCNTs produced a noticeable increase in strength, with the 0.45% dosage achieving the highest 28-day strength among the CNT-modified mixes. This enhancement is attributed to improved crack-bridging capability and better load transfer through well-dispersed CNT networks. Although the 0.55% MWCNT mix showed good performance, the slight reduction compared to 0.45% suggests the possibility of CNT agglomeration at higher dosages, reducing efficiency.

The nano silica mixes (NS 1.15%, NS 2.25%, and NS 3.35%) showed uniform strength values across all curing ages. While these mixes displayed consistent results, the improvements were moderate when compared to MWCNT-based mixes. This behaviour is associated with the high pozzolanic reactivity of nano silica, which enhances C–S–H gel formation and refines the microstructure, but the impact tends to stabilize beyond a certain dosage due to particle saturation. The graphical representation of the compressive strength is shown in the Fig. 10 and Fig. 11.

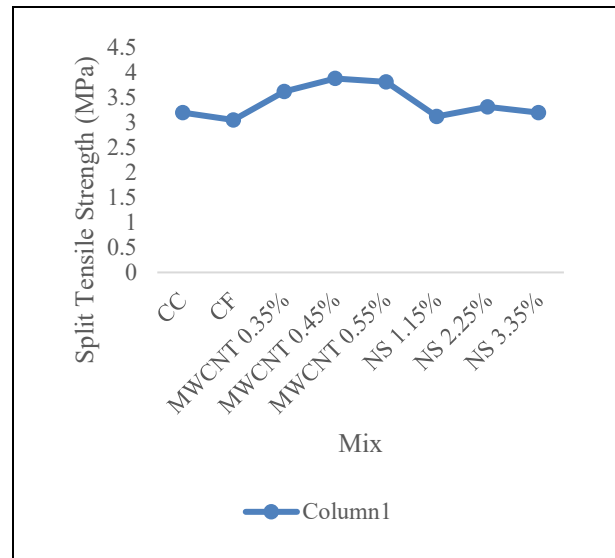


Fig. 10: Compressive strength results

Overall, the 28-day results confirm that the MWCNT 0.45% mix delivered the highest compressive strength, followed by MWCNT 0.55% and the nano silica mixes. The findings highlight that MWCNTs, when used at an optimal dosage, provide superior improvement in mechanical performance, whereas nano silica contributes to matrix densification but shows limited incremental gains at higher percentages.

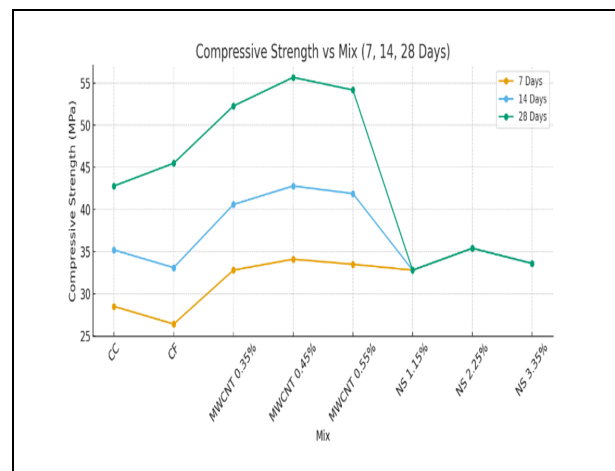


Fig. 11: Graphical presentation for compressive strength

8.2 Split Tensile Strength

The 28-day split tensile strength results indicate a notable improvement in tensile performance with the addition of MWCNTs and nano silica compared to the control concrete (CC). The control mix achieved a strength of 3.20 MPa, while the fly ash mix (CF) exhibited a slightly lower value of 3.05 MPa, highlighting the limited early-age tensile enhancement due to the slower pozzolanic reaction of fly ash. The MWCNT-modified mixes demonstrated a significant rise in tensile capacity. The 0.35% MWCNT mix reached 3.62 MPa, confirming the beneficial role of CNTs in improving crack resistance. The 0.45% MWCNT mix recorded the highest value of 3.88 MPa, indicating optimal CNT dispersion and effective microcrack bridging, which enhances stress transfer within the matrix. The 0.55% mix also showed improved performance at 3.81 MPa, although slightly lower than the optimum, likely due to CNT agglomeration at higher dosages. The graphical representation of the split tensile strength is shown in the Fig. 12 and Fig.13.

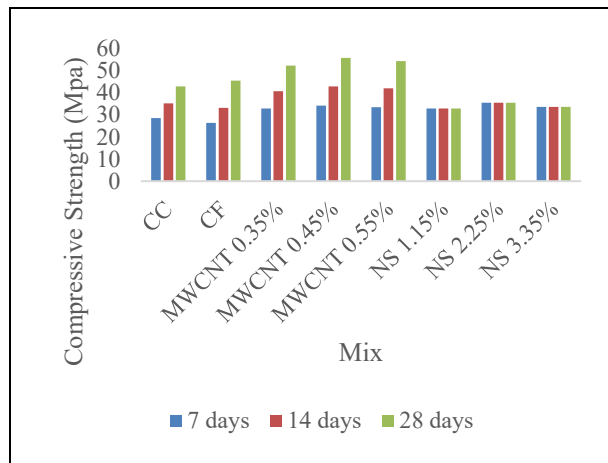


Fig. 12: Split tensile strength results

The nano silica mixes displayed moderate gains in tensile strength. The 1.15% NS mix achieved 3.12 MPa, while the 2.25% NS mix attained 3.31 MPa, suggesting enhanced particle packing and a denser matrix. However, the 3.35% NS mix, with a value of 3.20 MPa, showed that excessive nano silica may lead to particle clustering and reduced overall efficiency. In summary, the results clearly show that the use of MWCNTs markedly improves split tensile strength, with 0.45% MWCNT emerging as the most effective dosage. Although nano silica contributes to matrix densification, its tensile enhancement remains lower than that achieved by CNT-modified mixes.

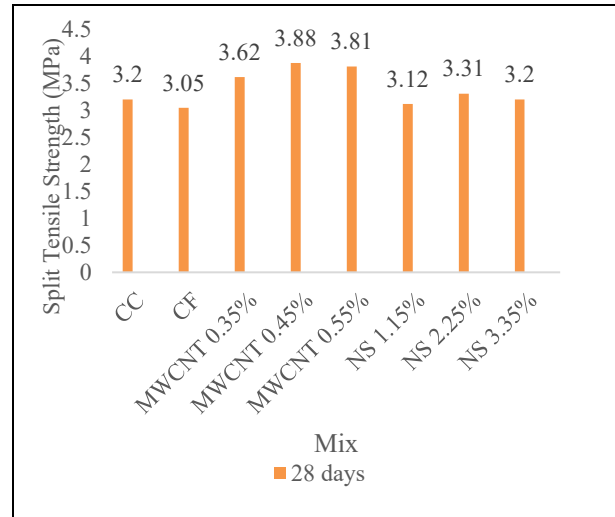


Fig. 13: Graphical presentation for split tensile strength

8.3 Flexural Strength Result

The flexural strength results indicate a clear improvement in performance with the addition of MWCNTs and nano silica when compared to the control concrete (CC). The control mix exhibited the baseline strength, while the fly ash mix (CF) showed a slightly reduced value, suggesting that fly ash alone offers limited enhancement in early-age flexural behaviour. A pronounced increase in flexural capacity was observed in the MWCNT-modified mixes. The 0.35% MWCNT mix demonstrated a substantial improvement, whereas the 0.45% MWCNT mix recorded the highest flexural strength, confirming that this dosage ensures optimal CNT dispersion and effective crack-bridging. The 0.55% MWCNT mix also achieved notable strength, though slightly below the optimum level, likely due to CNT clustering at higher concentrations. The flexural strength results are shown in Fig. 13 and Fig. 14.

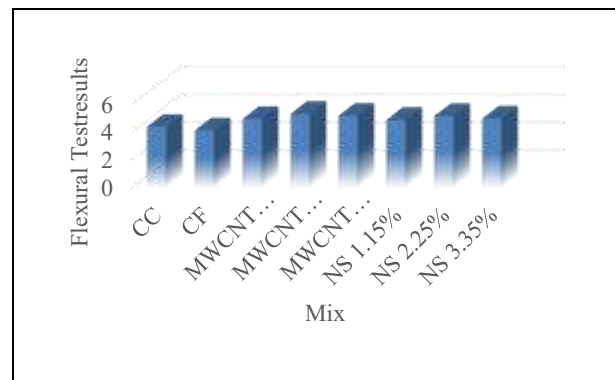


Fig. 14: Flexural strength result

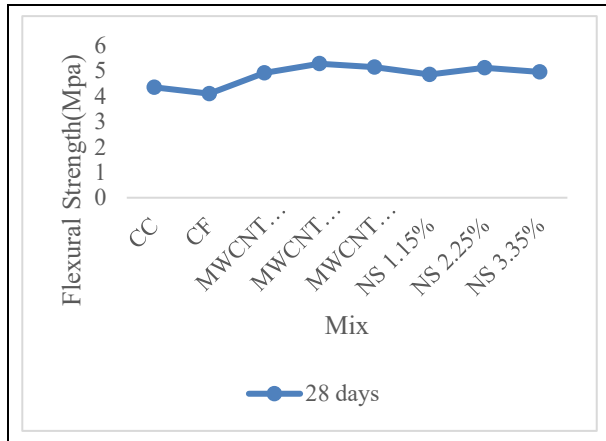


Fig. 15: Graphical presentation for flexural strength

The nano silica mixes also exhibited an upward trend in flexural strength relative to the control. The 1.15% NS mix showed moderate enhancement, while both 2.25% NS and 3.35% NS achieved further improvement, reflecting the benefits of improved particle packing, matrix densification, and refinement of the microstructure. The consistent increase with higher nano silica content highlights its role in enhancing bond strength and reducing pore volume. In summary, the findings clearly show that MWCNTs provide a superior enhancement in flexural performance, with 0.45% MWCNT identified as the most effective dosage. Although nano silica also contributes positively to flexural strength, its impact is comparatively moderate and highly dependent on dosage.

8.4 Modulus of Elasticity Results

The results show that the addition of fly ash, multi-walled carbon nanotubes (MWCNTs), and nano silica (NS) has a substantial impact on both the estimated compressive strength (f_c) and the modulus of elasticity (E) of the concrete mixtures. The control concrete (CC) achieved an estimated compressive strength of 40.96 MPa with an elastic modulus of 30.08 GPa, serving as the benchmark for comparison. In contrast, the fly ash mix (CF) exhibited a reduction in strength to 37.21 MPa and a corresponding decrease in modulus to 28.67 GPa, which can be linked to the delayed pozzolanic activity and lower early-age stiffness associated with fly ash. Significant improvement was observed in the mixes incorporating MWCNTs. The mix containing 0.35% MWCNTs recorded 52.42 MPa strength and 34.03 GPa modulus, demonstrating the beneficial role of nanotubes in enhancing stress transfer and bridging micro-cracks. The most notable enhancement occurred at 0.45% MWCNTs, where the compressive strength reached 60.22 MPa and the modulus increased to 36.47 GPa. This reflects the superior dispersion and reinforcing capability of the nanotubes, resulting in a denser microstructure. However, increasing the dosage to 0.55% caused a slight decline in strength (58.06 MPa) and modulus (35.81

GPa), likely due to agglomeration at higher concentrations, which creates weak points within the matrix. The modulus of elasticity and Flexural test results are shown in Fig. 15 and Fig. 16 respectively. The nano silica-modified mixes also exhibited improved performance.

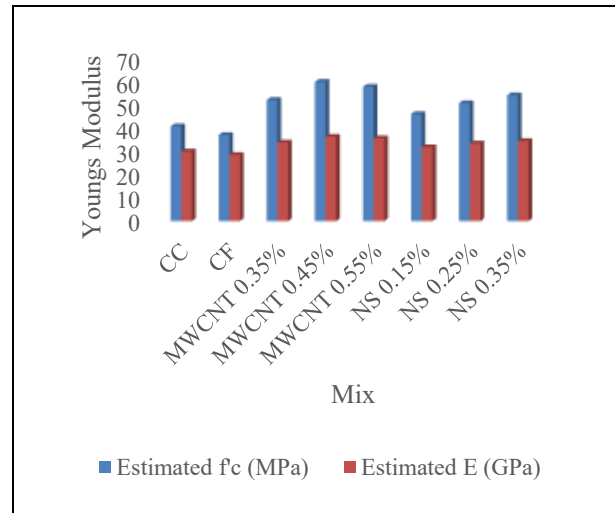


Fig. 16: Modulus of elasticity result

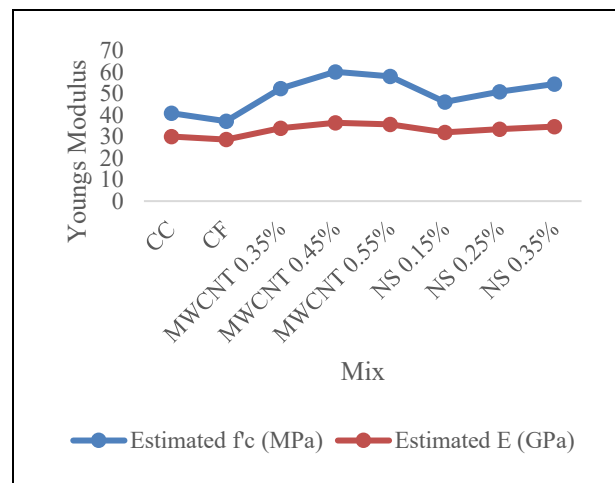


Fig. 17: Graphical presentation for flexural strength

The 0.15% NS mix achieved 46.24 MPa strength and 31.96 GPa modulus, while increasing the dosage to 0.25% NS further enhanced these properties to 50.98 MPa and 33.56 GPa, respectively. The 0.35% NS mix showed the highest performance among the nano silica series, with a strength of 54.46 MPa and an elastic modulus of 34.69 GPa, confirming the effectiveness of NS in refining pore structure and strengthening the interfacial transition zone (ITZ) through accelerated pozzolanic reactions. The results demonstrate that MWCNT incorporation yields more pronounced improvements in both strength and elastic modulus compared to fly ash and nano silica alone. Among all mixes, 0.45% MWCNT emerges as the optimal dosage,

offering the most significant enhancement in mechanical performance for the concrete system studied.

8.5 SEM Result

The microstructural investigation using SEM and X-ray diffraction reveals distinct differences in morphology and phase development among the control concrete (CC), fly ash concrete (CF), MWCNT-modified concrete, and nano silica-modified concrete. The SEM image of the control mix (Fig. 8.1) shows a relatively porous and loosely packed matrix, dominated by unreacted cement particles and microcracks, indicating weaker interfacial bonding. In contrast, the fly ash mix (Fig. 8.2) exhibits smoother spherical particles and improved packing, confirming the presence of unreacted fly ash along with additional C–S–H gel formed from pozzolanic reaction.

The MWCNT-modified specimen (Fig. 8.3) demonstrates a highly refined microstructure, where the CNTs appear embedded within the hydration products, acting as crack-bridging reinforcements and contributing to densification of the matrix. This indicates enhanced load-transfer capability and reduced microcracking. The nano silica mix (Fig. 8.4) shows a dense and compact microstructure with fine C–S–H gel formation due to the nucleation effect of nano silica, resulting in reduced pore spaces and improved matrix continuity.

The XRD results further support these observations, with the control mix showing prominent portlandite peaks, indicating incomplete hydration. The fly ash and nano silica mixes display reduced CH peak intensity and increased formation of secondary C–S–H, confirming effective pozzolanic activity. The MWCNT-modified mix shows improved crystalline distribution with suppressed CH peaks and enhanced hydration phases, indicating accelerated hydration and better structural organization. Overall, both SEM and XRD analyses confirm that MWCNT and nano silica significantly improve microstructural densification, with MWCNT offering superior crack resistance and nano silica contributing primarily to matrix refinement.

8.6 Results of X-Ray Diffraction

The X-ray diffraction (XRD) analysis clearly highlights the variation in phase composition caused by the inclusion of fly ash, MWCNTs, and nano silica in the concrete system. The control concrete (CC) displays sharp and distinct portlandite ($\text{Ca}(\text{OH})_2$) peaks at approximately $2\theta \approx 18^\circ, 34^\circ$, and 47° , indicating a higher presence of unreacted calcium hydroxide and a comparatively less compact microstructure. In contrast, the fly ash mix (CF) shows a noticeable reduction in portlandite peak intensity, confirming the pozzolanic reaction between fly ash and CH, which contributes to the formation of additional C–S–H gel. The mixes

incorporating MWCNTs exhibit an even greater decrease in CH peaks and display broader C–S–H peaks, reflecting enhanced hydration kinetics and improved matrix structuring due to the nucleation and crack-bridging capabilities of CNTs. Similarly, the nano silica mixes demonstrate a significant drop in CH peak intensity along with increased amorphous C–S–H formation, indicating effective secondary hydration and improved matrix densification. Overall, the XRD patterns show that although fly ash reduces CH content through pozzolanic activity, the combined action of MWCNTs and nano silica provides superior microstructural refinement, lower crystalline CH levels, and greater development of strength-enhancing hydration products.

9. CONCLUSION

The comprehensive experimental study clearly demonstrates that the integration of fly ash, multi-walled carbon nanotubes (MWCNTs), and nano silica significantly modifies the mechanical behaviour and microstructural characteristics of concrete. The use of fly ash contributes to improved sustainability and promotes secondary hydration by reacting with calcium hydroxide; however, its impact on enhancing mechanical properties remains relatively modest when used alone. A remarkable improvement in strength characteristics was achieved with the incorporation of MWCNTs. Among all the mixes, the 0.45% MWCNT dosage consistently delivered the highest compressive, split tensile, and flexural strengths, confirming it as the optimal concentration. This enhancement is attributed to the exceptional ability of CNTs to act as micro-reinforcing agents, effectively bridging microcracks, improving stress transfer, and accelerating hydration through their nucleation effect. Even though higher dosages such as 0.55% MWCNT showed strength improvement, slight reductions were observed due to probable CNT agglomeration, which hinders uniform dispersion. Nano silica also contributed significantly to strength enhancement by refining pore structure, filling micro-voids, and increasing the production of amorphous C–S–H gel. The progressive improvement in strength with increasing nano silica content highlights its effectiveness in densifying the cementitious matrix, though its performance remained slightly below that of the CNT-modified mixes. Microstructural investigations strongly support the mechanical test results. The SEM images revealed more compact, homogenous, and dense structures in CNT- and NS-modified mixes compared to the control mix. The XRD analysis further confirmed a substantial reduction in the intensity of portlandite peaks and a corresponding increase in the formation of C–S–H phases, indicating improved matrix quality and enhanced hydration reactions.

These findings confirm that MWCNTs offer the most significant enhancement in terms of strength and microstructural refinement, followed closely by nano

silica, while fly ash provides supplementary pozzolanic benefits. The combined mechanical and microstructural outcomes establish that nanomaterial modification, particularly using CNTs, offers a highly promising pathway for engineering high-performance, durable, and structurally superior concrete composites.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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