

# Sustainable construction material using nanosilica and multi-walled carbon nanotubes in cement concrete

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**Abstract.** Nanotechnology is a popular field in the construction industry due to its multiple functions. It mitigates CO<sub>2</sub> emissions and enhances the desirable properties of concrete by replacing small amounts of cement with supplements. This study assess the sustainability impact of using two different nanoparticles partially replacing the cement with 0.3%, 0.6%, 1.0% of nano silica (NS) and 0.03%, 0.045%, 0.06% of Multi-Walled Carbon Nanotubes (MWCNT) in the green concrete mix development. Nano-sized fragments at the atomic scale tends to modify the properties of concrete. Concrete may increase its strength, durability by adding nanocomposite materials, which will decrease the amount of nano and micropores in structural parts. The strength of the structural elements can be greatly improved and allowing them to withstand higher loads and resist deformation. It improved durability properties by 64.8% in water absorption, 56.4% in acid attack, 78.1% in sulphate attack, and 53.4% in chloride attack. There was an improvement in compressive strength of 37% and split tensile strength of 90%. SEM, FTIR, and XRD investigations have used to look at the microstructural characteristics of nanoconcrete dictated the micro-structure characteristics may be made more consistent and dense by adding nanocomposite materials.

**Keywords:** carbon nanotubes; microstructure; nano-silica; nanocomposite; sustainability

## 1. Introduction

Concrete is a composite material consist of Portland cement with or without mineral admixture, water, coarse and fine aggregate. They blend, form a paste, and then gradually harden over a time period. Concrete is used in the construction of concrete dams, culverts, and sewers, making it as an essential component in many projects. High mechanical strength and durability may be effectively attained in concrete by substituting nanomaterials, offers low-cost, environmentally friendly concrete construction.

The main novelty of the research lies in the production of high-strength concrete with the addition of reduced percentage of nanomaterials. The addition of nanoparticles as a partial cement substitute is more advantageous in developing environmentally friendly, sustainable building materials. In the adjacent areas, where heavy materials and hazardous synthetic chemicals, including nanomaterials, can be employed in addition to cement could increase concrete strength and durability. The focus of the nano-concrete material was not as fast on the spots utilizing synthetic compounds and robust materials but also on the places where the impact of water bodies is available, the spots where there is successive utilization of water, and the locations where the dampness content is exceptionally high. Carbon nanotubes (CNTs) have self-sensing properties, and

Hence they are instrumental in the structural health monitoring of concrete structures by measuring material resistivity. Adding a small quantity of CNTs to concrete will increase electrical conductivity and produces high-performance concrete. (Suchorzewski *et al.* 2020a, b, Tounsi *et al.* 2013a, b). Nanoparticle has the potential of enhancing concrete strength due to their unique properties, presenting opportunities to improve the performance and durability properties of cement-based materials in various applications (Hany and Ahmed 2023, Zhang 2023). Nanomaterials shows promising application in fortifying the strength and durability of concrete by reducing the micropores, enhancing the microstructure density, and improving the properties such as compressive, tensile, and flexural strength. Additionally, they enhance resistance to various environmental factors, underscoring their versatility in optimizing concrete performance (Allujami *et al.* 2022a, b).

Manufacturing cement-based composites give rise to an undeniable level of CO<sub>2</sub> gas emissions. Nanomaterials can be used in concrete- products, reduces the amount of cement utilised in construction projects. Minimum fluctuation in strength can be noticed in CNT dispersion with and without the addition of plasticizers. Nanotechnology offers several advantages for cement-based products and it improves their bulk properties, enhancing strength, durability, and thermal stability.

Additionally, nanotechnology enables the production of thinner final products, reducing material usage without compromising structural integrity. It allows for precise manipulation of materials at atomic scale, leading to improved homogeneity and customized properties. Moreover, nanotechnology reduces environmental contamination by

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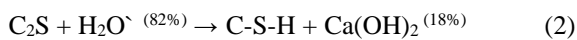
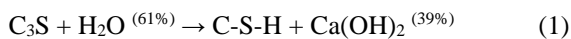
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optimizing resource utilization and minimum waste generation. Finally, the use of nanotechnology in cement-based products can be cost-effective, delivering better performance and sustainability in the long term. Nanotechnology has great potential for enhancing the properties and reducing the environmental impact of cement-based materials.

Fig. 1 shows the concrete comprises of different sizes of particles that generate a well-composed, impermeable, homogeneous medium, and well-graded concrete composite with minimised micro and nano cracks. The CNT diameter normally ranges between 0.4 to 40 nm, and its length varies from 0.14 nm to 55.5 cm. NS comprises tiny particles ranging from 1–100 nm with high amorphous silica content (Singh *et al.* 2015a, b). The calcium silicate hydrate (C-S-H) gel pore size percolates in first three days of hydration, ranging from 2.5 nm to 10 nm, and the interlayer pore size is always measured to be 0.8 nm (Berodier *et al.* 2020a, b). The particle size of Portland cement varies from 7 μm to 200 μm. Acceptable aggregate ranges from 75 μm to 4.75 mm, and coarse aggregate size ranging from 4.75 mm to 80 mm as per Indian standards.

The aggregates, sand, and other particles are called centropiasm, which functions as a skeleton, gel, and transmitter material. According to Wu’s centropiasm hypothesis, the strength of concrete is highly influenced by the binding pressure in between them. Nanoparticles dispersed in the cement paste serve as a sub-centropiasm, closely bonded with the hydrated products in the transition zone, enhancing the interaction between paste and aggregates. Nanomaterials have a filler effect by refining the intersectional region of cement, resulting in denser concrete. Common conflicts in the microstructure of concrete, such as porosity, voids, etc., were eliminated due to the presence of nanoparticles in the concrete. Table 1. shows a brief view of the reaction mechanisms involved in the hydration process of cement.

The reactions influenced by the addition of Nanoparticles are given below:



Eqs. 1 and 2 show excess Ca(OH)<sub>2</sub> is retained in the cement mix. This unused Ca(OH)<sub>2</sub> further reacts with nanoparticles produces C-S-H gel which contributes to the strength development. Thus, more C-S-H gel is generated, eventually leading to a stronger cement composite with denser and compact microstructure.



NS and carbon nanotubes can improve the compatibility between aggregates and cement-based materials. Modern, sustainable building materials can help lessen the built environment’s adverse environmental effects (Sakulich, 2011). Studies on cement-based materials, including NS and CNT, are required to comprehend their impact on various characteristics. Nanoparticles are now being exploited to create novel materials, electronics, and systems at the molecular, nano, and micro scales. NS and CNT exhibit unique physical and chemical features that might make

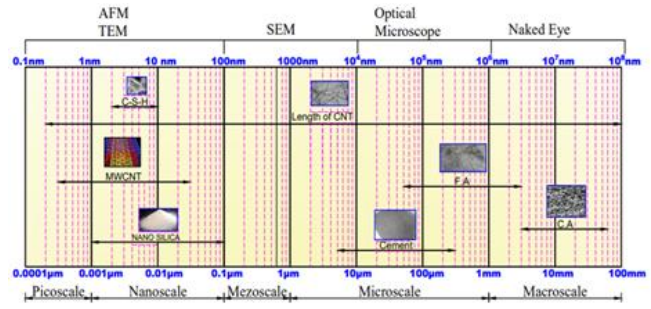


Fig. 1 Size range of Particles used in concrete fabrication

Table 1 Main Products of Hydration

Cement + Water → Main Hydration Products	
Clinker	Ca(OH) <sub>2</sub>
C <sub>3</sub> S	3CaO. 2SiO <sub>2</sub> .
C <sub>2</sub> S	3H <sub>2</sub> O (C-S-H gel)
C <sub>3</sub> A	+H <sub>2</sub> O → 3CaO. Al <sub>2</sub> O <sub>3</sub> . 3CaSO <sub>4</sub> .
C <sub>4</sub> AF	→ 32H <sub>2</sub> O
Gypsum	3CaO. Al <sub>2</sub> O <sub>3</sub> . CaSO <sub>4</sub> .
CaSO <sub>4</sub> .2H <sub>2</sub> O	→ 12H <sub>2</sub> O
	4CaO. Al <sub>2</sub> O <sub>3</sub> . 13H <sub>2</sub> O

more effective materials than those currently visible in the industry (Kumar *et al.* 2014a, b). Their large surface area and good fire-retardant capabilities may help the employment of nanoparticles in buildings in various ways due to their excellent size. Multiple types of research have been explored on the subject of influence, both individually and in combination with NS and CNT in cement-based materials.

In the investigation conducted by Elkady and Hassan (2018), the enhancement of concrete ductility using Carbon Nanotubes was explored to maintain other mechanical properties unaffected even under extreme temperature conditions. Mechanical and durability characteristics of concrete were evaluated by replacing varying proportions of cement, ranging from 0% to 4%, with nanosilica (NS). The study findings revealed that the inclusion of 2% NS in the concrete substantially improved the compressive strength, tensile strength, and flexural strength by 40%, 15%, and 35%, respectively. This study effectively demonstrated that incorporating NS into the concrete mixture successfully enhanced the overall performance and structural integrity of the material.

CNTs typically serve as addition element to reinforce the different structural materials because of its exceptional tensile strength (Hu *et al.* 2018a, b). Adding NS improves resistance to sulphate, acid, and chloride attacks by about 10% (Atahan and Arslan 2016, Saloma *et al.* 2015a, b). The primary role of NS is to fill voids and make the cement structure denser. It also has a pozzolanic impact, which helps to improve rheology and improve the strength qualities (Vera-Agullo *et al.* 2009a, b). Carbon nanotube-reinforced polymer composites improve strength but its high manufacturing costs along with their distinctive advantage of being piezo-resistivity for real-time monitoring, is especially beneficial in automated processes (Tapeinos *et al.* 2012a, b). The degree of hydration is enhanced with the incorporation of nanosilica.

Additional amount of C-S-H gel is generated, aided in filling the pores inside the concrete mixture. The accelerated hydration reaction is attributed to the high reactivity of NS, leading to a reduction in the setting time, is noted in the study conducted by Zhuang and Chen (2019). Furthermore, adding NS and hybrid nanoparticles contributes to the improved compressive strength in cured concrete, that tends to coincide with increased drying shrinkage.

The alterations in the mechanical properties of CNTs and Portland cement-based mortars were reported by (Camacho *et al.* 2014a, b). It was found that, after the curing period of 28 days, the incorporation of CNTs in Portland cement mortars does not produce any noteworthy impact on the concrete bending or compressive strength. The approach for dispersing CNTs within the concrete structure involved sonication with water and a polycarboxylate-based superplasticizer. The method achieved a thorough and effective dispersion of CNTs within the concrete. MWCNT-reinforced composites outperform the conventional composites in compression and flexure, according to Manzur and Yazdani (2015). In the study conducted by (Adhikary *et al.* 2020a, b), determined that the optimal fraction of plasticizer ranged from 0.1% to 0.3% weight of cement for Multi-Walled Carbon Nanotubes. It was found that the most effective plasticizer fraction was 0.008. When concrete features long CNTs in its microstructure, it increases fracture bridging effect and reduces the CNT dispersion within the composite structure (Shariati *et al.* 2019a, b).

Additionally, CNT-enhanced cementitious composites with low water-to-cement ratio exhibited enhanced long-term durability, creep and shrinkage. Moreover, in the research conducted by (Nguyen *et al.* 2020a, b), it was noted that SiO<sub>2</sub> serves not only as a filler to enhance the microstructure of mortar cement but also promotes the pozzolanic reaction in the production of high-strength concrete. Furthermore, the inclusion of CNTs and Nylon 66 nanofibers increases the bridging effect and mechanical strength of the concrete. The study done by Mishra (2022) examined the combined impact of carbon nanotubes and nano-sized silica on cement matrices, demonstrating a notable improvement upto 12 to 15% in compressive strength compared to control cement paste.

Compared to conventional concrete, incorporating nanoparticles has increased compressive strength by approximately 38%. This finding highlights the significant positive impact that nanoparticles kept on enhancing the compressive strength of concrete materials (Qian *et al.* 2022a, b). The study (Musso *et al.* 2009a, b) concludes that as-grown and annealed MWCNTs significantly enhance the mechanical properties of cement matrix composites, while carboxyl functionalized MWCNTs have a detrimental effect. The physical adsorption of NS on the CNT surface might speed up cement hydration and improve the strength and durability of concrete (Li *et al.* 2017a, b). A feasible method of improving the mechanical behaviour of concrete is the addition of nanoparticles and the nanoparticles are the suitable fillers and also promote the hydration process (Li *et al.* 2004a, b, Madenci (2021), Raheem *et al.* 2021a, b, Wang

*et al.* 2020a, b). Concrete can become more resilient and environmentally sustainable by incorporating nano-silica, offering a promising avenue for advancing construction materials towards excellent durability and reduced environmental impact (Abilash *et al.* 2021a, b).

The experimental study done by (Saleh *et al.* 2021a, b) explores the impact of Nano-silica on concrete mechanical properties, revealing a remarkable increase in compressive strength up to 32.45%. These findings highlight the potential abilities of nanosilica material in enhancing the concrete performance significantly in various construction applications. Adding MWCNT significantly shoots up the compressive strength of recycled aggregate concrete by 70%. Additionally, MWCNT inclusion enhances impact resistance, and 0.1% dosage shows the highest improvement (Allujami *et al.* 2022a, b).

The comparative analysis of various nanomaterials on concrete, Nano Silica demonstrated their superior performance, as noted in the study reported by Amin and Abu El-Hassan (2015). Furthermore, Mohamed's (2016) research revealed that incorporating 3% nanoparticles (comprising 25% NS and 75% NC) into concrete-mechanical properties at different ages yielded best mechanical qualities, particularly in compressive and flexural strength. NS-incorporated concrete was seen as statistically more significant than conventional concrete, accelerating hydration and achieve the maximum strength of concrete within a shorter period. Adding CNT and titanium dioxide (TiO<sub>2</sub>) in concrete provides better resistance to corrosion (Yan *et al.* 2020a, b). IS 516:1959 (2004) codes help the perception of this technique to assure the strength of cement. Examination and preparation of materials, proportioning, weighing, mixing concrete, workability, size of test samples, moulds, compaction, capping of the specimens, and curing are portrayed in the laboratory making and curing of compression test specimens. Indian Standard Code IS 5816 (1999), deals with the testing of splitting tensile strength of concrete portrays the positioning of specimen and loading rate.

Nanoconcrete offers various applications in industries that demand robust construction materials for heavy instrumentation and materials. It is beneficial in sectors that utilize different types of chemicals, and the structures required to be built with chemical-resistant and durable materials. By incorporating nanomaterials, this nanoconcrete can be engineered to possess high mechanical strength, chemical resistivity, and long-lasting durability. Furthermore, nanoparticle infusion can enhance the properties of UHPC, making it even more versatile and effective in demanding construction projects. As the applications of nanomaterial-based concrete are large, this research aims to find a sensible solution to improve concrete mechanical and durability properties for its efficient application in industries employing heavy materials and chemicals. Nanoconcrete aims to produce strong, durable, and less porous concrete with denser and more homogeneous matrices. Hence, extensive research has yet to be carried out to develop a sustainable hybrid nanomaterial-based concrete. The proposed research aims to experimentally analyze the suitability of hybrid nanomaterial-based concrete for construction industries.

Table 2 Physical Properties of NS

Surface area	200-250m <sup>2</sup> /g
Specific gravity	1.36
Purity	99%
Bulk Density	0.06g/cm <sup>3</sup>
Physical form	Fine powder
Colour	White

Table 3 Properties of MWCNT

Outer diameter	10-20nm
Inner diameter	5-10nm
Length	>10 $\mu$ m
Surface area	110-350m <sup>2</sup> /g
CNT content	95-99%
Bulk Density	0.14g/cm <sup>3</sup>
Specific gravity	0.14
Purity	99%
Physical form	Very fine powder
Colour	Black powder
Odour	Odourless

### 1.1 Research significance

The significance of the research lies in the exploration of innovative solutions to address critical challenges in the construction of industrial structures. The study aims to offer tangible benefits to the construction industry by investigating the sustainability and feasibility of hybrid nanomaterial-based concrete. This research examines the improvement in mechanical and durability properties and resistance to environmental factors of concrete structures by incorporating nanosilica and Multi-Walled Carbon Nanotubes in concrete mixes. This has made significant implications for industries requiring robust and long-lasting durability, particularly those exposed to carry heavy loads and harsh environmental conditions. By filling a critical gap in existing knowledge and focusing on sustainable solutions, this research contributes advanced solutions in the field of construction materials. It offers practical insights for the development of more efficient and durable infrastructure.

## 2. Materials and methods

Cement is a substance that binds other materials together, forming a solid mass. This experimental study has used ordinary Portland cement (OPC 53 grades). The initial and final setting times of cement is 29 minutes, 603 minutes respectively. The normal consistency value is 32%, and the specific gravity of the cement is 3.15. Fine aggregates particles that almost pass through the 4.75 mm sieve and completely retained on 75  $\mu$ m sieve are used in concrete as a filler material. M-sand that conformed to IS 383-1970 Grading Zone II was used in the experiments. The specific

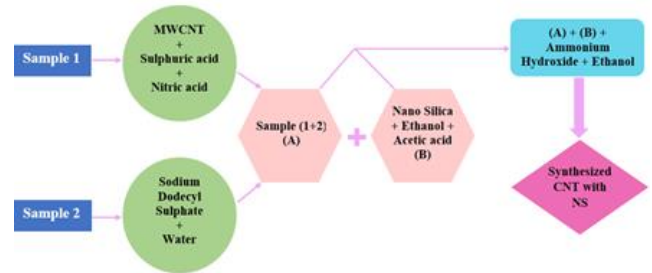


Fig. 2 Dispersion Process

gravity value of fine aggregate was found to be 2.66. The water absorption and moisture content values obtained in the experimental studies are 0.8%, 4.6%, respectively. Coarse aggregates particles retained on the 4.75-mm sieve with nominal sizes 20mm and 10mm, as defined by IS 383: 1970, are utilised. Specific gravity of coarse aggregate is measured to be 2.69. The water absorption and moisture content values of coarser particles obtained are 0.6% and 1.5%, respectively.

NS is a pre-eminent product of microsilica and recent advances in nanoscale concrete research have shown NS is most preferable than silica used in conventional concrete. Table 2 presents the physical properties of NS used in the experimental study.

MWCNTs are elongated, hollow nanocylinders formed by sp<sup>2</sup> carbon with a 3 to 30 nm diameter. The outer and inner diameters of MWCNT were measured by TEM analysis. Their length-to-diameter proportion ranges from 10 to 10 million. In this experimental study, the length of MWCNT used was greater than 10  $\mu$ m (104 nm), and its inner and outer diameters were 5–10 nm and 10–20 nm, respectively. The physical properties of MWCNT examined by SEM and TEM analysis are presented in Table 3.

### 2.1 Dispersion of CNT

The MWCNT and nano-silica are chemically synthesized by adding concrete to increase strength. Fig. 2 shows the synthesis process of CNTs and NS. Multi-walled carbon nanotubes are weighed at 200 mg and dissolved in the ratio of 30 ml of sulfuric acid to 10 ml of nitric acid. They are treated separately for 6 hours and thoroughly stirred. Then it is followed by, washing, drying and then the weight of dry sample is taken and denoted as sample 1. Then sodium dodecyl sulfate is weighed separately, dissolved with water, and stirred till it dissolves and considered as Sample 2. Sample 1 is combined with sample 2 is considered as A. Then 100 mg of NS is dissolved in 20 ml of ethanol and acetic acid is stirred thoroughly, and labelled as B. Now add B and A in the process, drop by drop. Now the sample in acid medium is added to ammonium hydroxide drop by drop is converted into to a primary medium. Then 10 ml of ethanol is added and, stirred for 30 minutes, and centrifuge until the precipitate gets separated. It is dried for 8 hours at 80 degrees Celsius produces a dry yield synthesized MWCNT with NS. With this procedure, we ensures the utilisation of minimum product for large quantities; and some extra quantity is required if the process repeated several times.

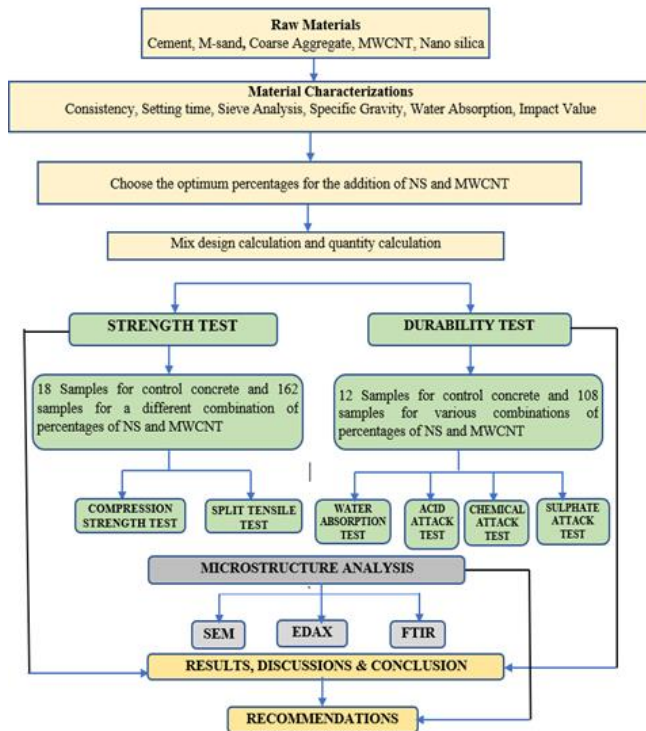


Fig. 3 Comprehensive framework of the experimental program

Table 4 Various percentages of NS & MWCNT Replacement

Nanomaterials	Percentage of Replacement
NS replacement	0.3, 0.6 and 1.0
MWCNT replacement	0.03, 0.045 and 0.06
NS+MWCNT replacement	0.3 NS+0.03MWCNT, 0.6 NS+0.045MWCNT and 1.0 NS+0.06MWCNT

## 2.2 Experimental program

The proposed detailed framework of the research is presented in Fig. 3 for a clear understanding. The knowledge from earlier related works shows that nanomaterials can be used in concrete for strength and durability. NS and MWCNT were chosen, and the optimum dosage of such materials was also determined in this study. The experimental plan was initiated by selecting the probable combination of nanomaterial percentages used and then calculating the number of samples to be cast for testing and analysis. The concrete grade M20 was used in the experiment is designed as per IS 10262: 2009 procedure. The water-cement ratio was kept constant at 0.50 and the sample specimens were cast for 7 days, 14 days, and 28 days of curing for compressive and split tensile strength tests. Durability tests include a water absorption test, acid attack, sulfate attack, and chloride attack etc and the sample specimens were cast for 42 days. Three samples and the percentage of NS and MWCNT for each test were released and the average test results of three samples were taken as the final reading, and the results are presented.

The samples were cast for combinations of different percentages of NS & MWCNT, as given in Table 4.

Cubical specimens for compressive strength tests 150mm x 150mm x 150mm, and durability tests were conducted on 50mm x 50mm x 50mm size specimens. Cylindrical test specimens for the split tensile strength test were 150mm diameter and length 300mm. Concrete batches were mixed and filled in the moulds. After  $24 \pm \frac{1}{2}$  hours, the samples were marked, removed from the moulds, and immersed in clean water. The materials used in concrete were cement, fine aggregates, coarse aggregates, NS, MWCNT, and water. Proportioned quantities of these materials for each batch were determined by weight, with an accuracy of 0.1% of the total weight. After the curing period the specimen was positioned in the compression testing machine (CTM) for the compressive strength test. The axial load was applied and increased at  $140 \text{ kg/cm}^2$  ( $13729.31 \text{ KN/m}^2$ ) per minute until the specimen collapsed. The maximum load carried was noted. Then split tensile strength test was conducted on the lower plate of the CTM; a plywood strip was held, and the specimen was placed on it, on which the other plywood was placed. The upper plate was lowered. The load was applied at a constant rate of  $1372.9$  to  $2059.4 \text{ kN/m}^2$  per minute until the specimen failed. Fig. 4(a) shows the casted cubes and cylinders with various percentages of nanocomposites.

Durability is the most critical parameter in nano-concrete and it can be achieved using different methods, including acid attack, sulphate attack, water absorption, and chloride attack tests. Fig. 4(b) shows the cast specimens in curing process for durability tests. For acid attack test, the specimens were immersed in 20% hydrochloric acid (HCl), and for the chloride attack test, the samples were immersed in 20% magnesium chloride ( $\text{MgCl}_2$ ). A sulphate attack test of concrete was done by engaging the samples in 20% magnesium sulfate ( $\text{MgSO}_4$ ) for about 28 days. Fig. 4(c) shows the cube specimens prepared for durability tests. The specimens were removed from curing tank after the curing period. It was dried, weighed as dry weight (**W1**), and then immersed in water for a specific period of time for water absorption test. The weight of the specimen was noted as (**W2**). In the acid attack test, the samples were cured for seven days, taken out, kept in the atmosphere for 24 hours, and weighed (**W1**). They were immersed in a solution containing 20% hydrochloric acid (HCl) for 42 days. The specimens were taken out and weighed as **W2**. The loss in weight due to the acid attack was found. Fig. 4(d) shows the effect of the specimen after 42 days of the acid attack test. The specimens were dried and weighed for the sulfate attack test after seven days of curing (**W1**). They were immersed in 20% magnesium sulphate ( $\text{MgSO}_4$ ) solution for 42 days and then taken out and weighed. The corresponding weights were tabulated as **W2**. Concrete showed a loss in weight due to the sulphate attack. Fig. 4(e) shows the effect of the specimen after the sulphate attack test.

In the chloride attack test, the samples were cured for seven days were removed, dried, and weighed (**W1**). It is then immersed in a 20% magnesium chloride ( $\text{MgCl}_2$ ) solution for 42 days and the weights were tabulated as **W2**.

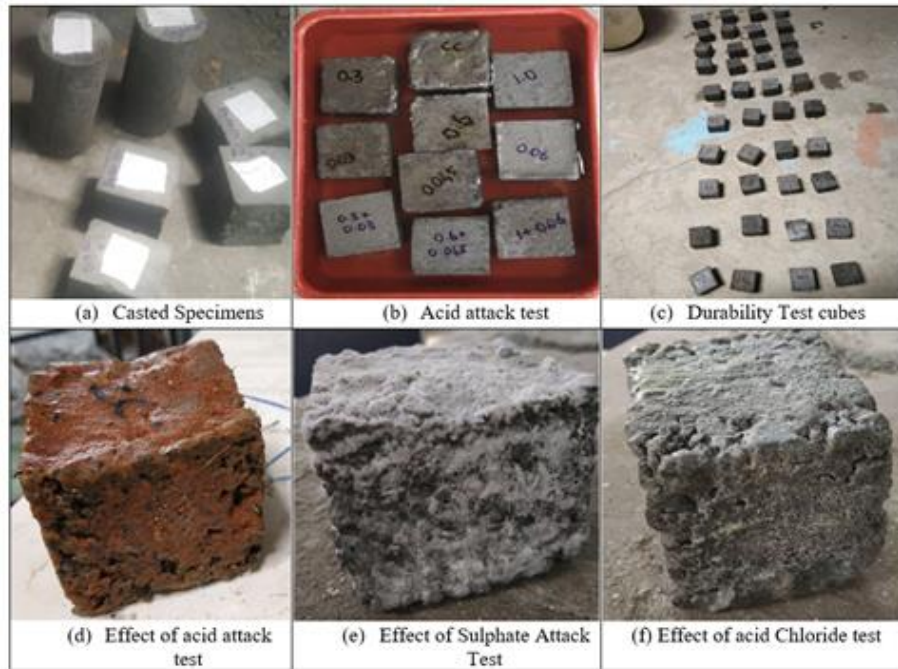


Fig. 4 Experimental work

The loss in weight due to the chloride attack was calculated. Fig. 4(f) shows the effect of the specimen after the chloride attack test.

Microstructure analysis is necessary to confirm nano-materials reaction, cracking pattern, structural arrangement, and dispersion properties in concrete. Scanning Electron Microscope (SEM), Fourier transform Infrared Spectroscopy (FTIR), and Energy Dispersive X-ray spectroscopy (EDX) analyses were conducted. The infrared absorption spectra of synthesized samples were investigated using the FTIR instrument Shimadzu Intracrer-100 with a scanning range of 400-4000  $\text{cm}^{-1}$ . A study of the surface morphology of the developed material was made using FE-SEM (S-3400 N Hitachi). PANalytical Xpert-PRO XRD identified the phase identification on the developed nanocomposites with Cu Ku radiation ( $\lambda = 1.541 \text{ \AA}$ ) in the  $2\theta$  range from 10 to 80.

### 3. Results and siscussion

#### 3.1 compressive strength test

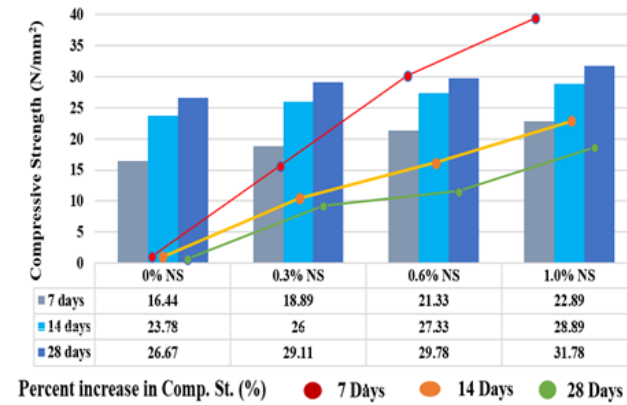
The compressive strength of sample cubes was determined by testing the specimen in a compression testing machine, is presented in Fig. 5(a). Adding 0.3% of NS improved the compressive strength by 14.9% at 7 days, 29.74% at 14 days, and 39.23% at 28 days. An increase in the quantity of NS of 0.6% increases the compressive strength by 9.3%, 14.9%, and 21.48% at 7, 14, and 28 days respectively. Adding 1% of NS improved the strength characteristics by 9%, 11.6%, and 19.1% after curing period of 7 days, 14 days, and 28 days, respectively. Research indicates that while adding more significant quantities of NS to concrete results in a lower percentage gain compressive strength and adding smaller amounts of NS to

concrete gives higher improvement in compressive strength.

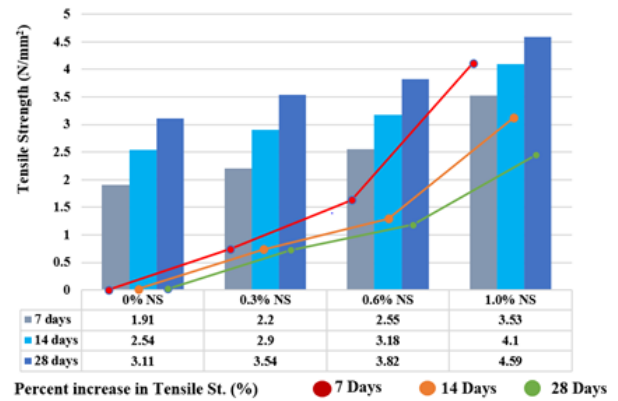
The CNTs with percentages of 0.03%, 0.045%, and 0.06% were added to the cement. The addition of 0.03% CNT shows a compressive strength of 16.88  $\text{N/mm}^2$  in 7 days, 25.05  $\text{N/mm}^2$  in 14 days, and 29.53  $\text{N/mm}^2$  in 28 days. The addition of 0.045% CNT causes an increase in strength from 18.66  $\text{N/mm}^2$  to 31.55  $\text{N/mm}^2$  at 28 days. The addition of 0.06% CNT caused an improvement in the strength ranging from 19.55  $\text{N/mm}^2$  to 33.78  $\text{N/mm}^2$ , respectively, as represented in Fig. 5(c). Fig. 5(e) shows variations in strength with the addition of NS and CNT. Adding 0.03% of CNT and 0.3% of NS causes an increase in strength ranging from 19.78  $\text{N/mm}^2$  to 30  $\text{N/mm}^2$  at 28 days of curing. The strength rises from 22.44  $\text{N/mm}^2$  to 33.11  $\text{N/mm}^2$  with 0.6% NS and 0.045% CNT. Adding 1% of NS and 0.06% of CNT causes an increase in strength from 23.55  $\text{N/mm}^2$  to 36.67  $\text{N/mm}^2$ .

After comparing the data presented in Fig. 5(a),(c), and (e), it was observed that the early age (7 days) strength exhibited a more significant increase when 1.0% of NS was used as a replacement, as opposed to any MWCNT replacement. However, it is essential to note that the increase in strength was not more significant than the combined replacement of NS and MWCNT. Compared to the results of (Chandramouli *et al.* 2019a, b) and (Dheeraj Swamy *et al.* 2017a, b) in Fig. 5e, the combined replacement of 1.0% NS and 0.06% MWCNT is the best way to replace nanomaterials in concrete to elevate the compressive strength.

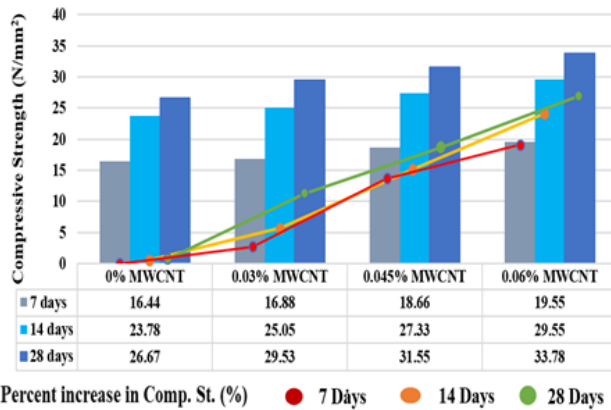
The compressive strength improved with increasing CNT and NS content by nearly 40%, but the results (Chandramouli *et al.* 2019a, b) show a increase about 37.85%. The improvement in the strength of concrete was brought by the fact that nanocement serves as a filler material and a catalyst due to its larger specific surface area.



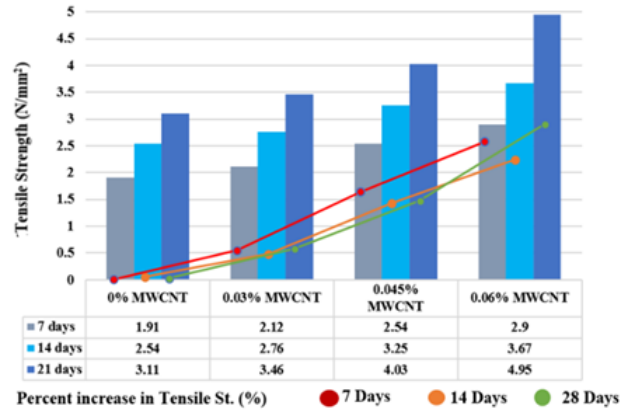
(a) Compressive Strength with NS Replacement



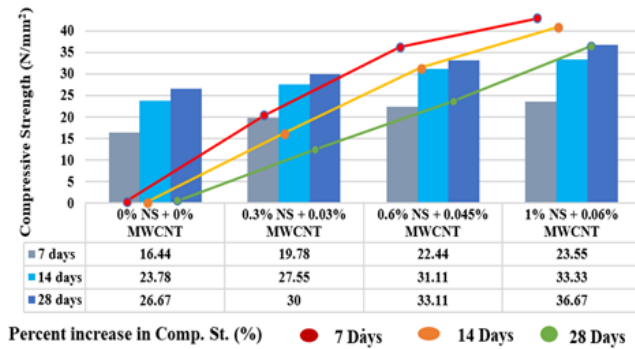
(b) Tensile Strength with NS Replacement



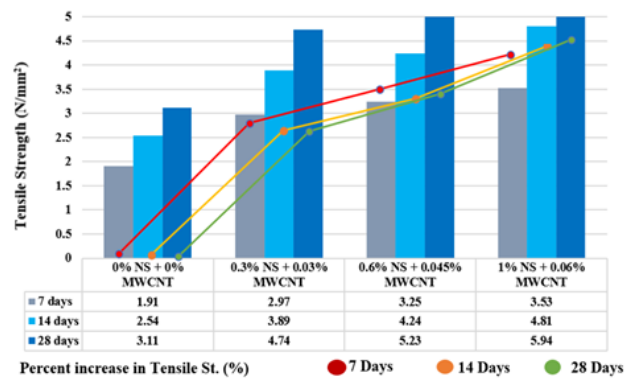
(c) Compressive Strength with MWCNT Replacement



(d) Tensile Strength with MWCNT Replacement



(e) Compressive Strength with NS &amp; MWCNT Replacement



(f) Tensile Strength with NS &amp; MWCNT Replacement

Fig. 5 Mechanical strength of hybrid nanomaterials

Nanomaterials specific surface area and particle size distribution are more important in improving concrete compressive strength. The 28-day compressive strength of concrete helped to achieve a 19.16% increase after replacing 1.0% NS in the concrete. The 28-day compressive strength of concrete is increased to 26.65% by replacing 0.06% MWCNT in the concrete. The maximum increase in compressive strength at 28 days was observed in the specimens with 1.0% NS and 0.06% MWCNT, combinations. On replacing 1.0% NS with 0.06% MWCNT in the concrete, gives a 37.49% improvement in 28-day compressive strength. When compared to the results of (Narasimman *et al.* 2020a, b), the addition of 1% of NS and

2% of CNT in cement resulted in 38.7% increase in compressive strength, indicating that the inclusion of only 0.06% CNT is sufficient for a significant increase in strength.

### 3.2 Split tensile strength test

NS in various ratios was added to the cement to improve the strength characteristics of the concrete. In 28 days, 84.8% of strength was attained while adding 0.3% of NS, 61.41% was achieved with 0.6% of NS, and 47.5% of strength was reached by adding 1% of NS, as shown in Fig. 5(b). Fig. 5(d) shows the tensile strength variations in

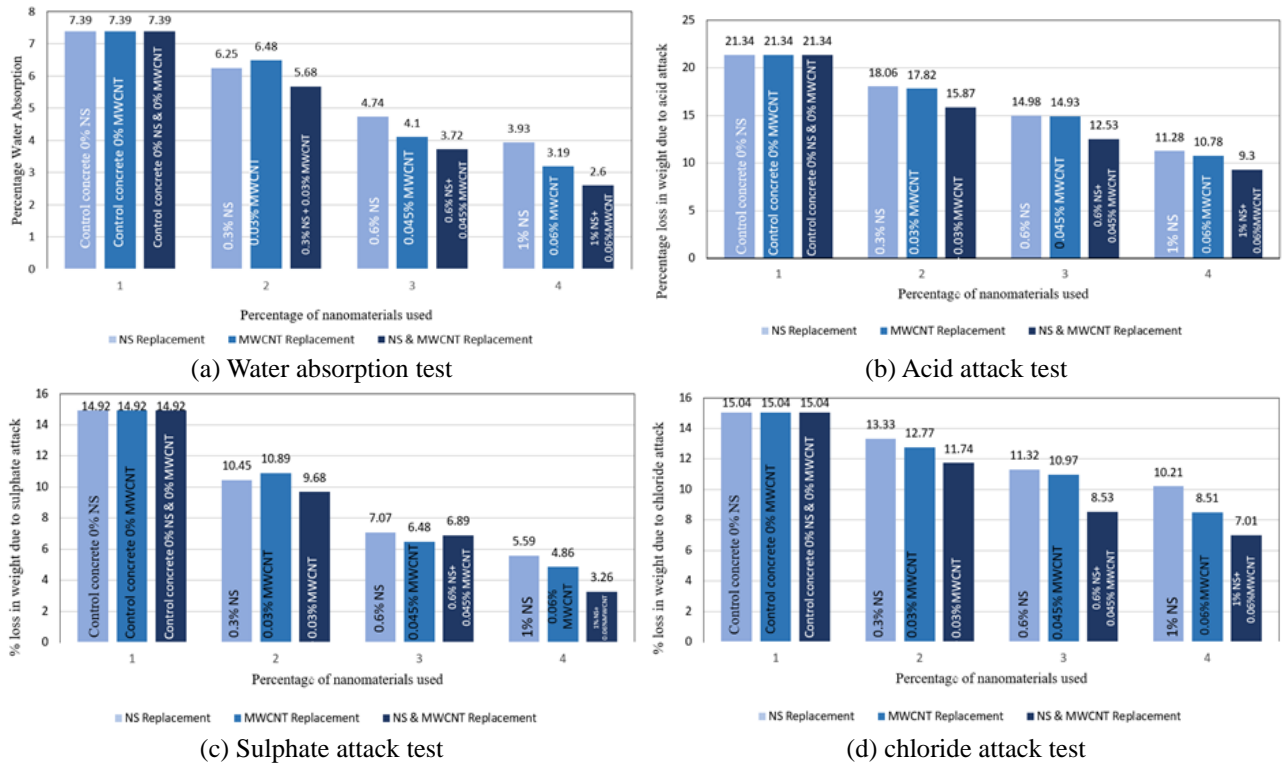


Fig. 6 Comparison of durability test

MWCNT incorporated in the concrete specimen. MWCNT was added to cement in different ratios to improve the split tensile strength characteristics of the concrete. With the addition of 0.03% of MWCNT 51.8% of strength was attained with the, 44.48% with 0.045% of MWCNT, and 47.5% of strength with the addition of 0.06% of MWCNT. With the addition of 0.03% CNT and 0.3% NS, the strength increased from 2.97 N/mm<sup>2</sup> to 4.74 N/mm<sup>2</sup> at 28 days of curing. The strength rises from 3.25 N/mm<sup>2</sup> to 5.23 N/mm<sup>2</sup>, with 0.6% NS and 0.045% CNT. Adding 1% NS and 0.06% CNT helped increase the strength from 3.53 N/mm<sup>2</sup> to 5.94 N/mm<sup>2</sup>. The strength variations are presented in Fig. 5(f).

The comparison graphs from Fig. 5(b), (d), and (f) show a maximum increase in the split tensile strength when the combination of 1.0% NS and 0.06% MWCNT is used. This replacement of nanomaterials enhances the split tensile strength by nearly 90% compared with a standard concrete mix. In the previous research carried out by (Chandramouli *et al.* 2019a, b) with the combined percentage of NS and CNTs, the flexural strength value after 28 days of curing was found to be 6.41 N/mm<sup>2</sup>, and the flexural strength value decreased to 5.94 N/mm<sup>2</sup>. The presence of individual CNTs acted as additional nucleation sites for hydrates and increased the reactivity of the NS. Combining NS with modest amounts of CNT positively made impact on the hydration process. As the quantity of nanomaterials increases, the improved tensile strength of concrete decreases. More nanoparticles in the mixture are necessary to combine with the liberated lime during the process of hydration, which causes excess silica to leach out and weaken the material. After completing all the reactions, it only replaces a portion of the cementitious material without

contributing to its strength. Due to the strong reactivity of nanoparticles at early ages, the quick consumption of Ca(OH)<sub>2</sub>, was generated during the hydration of Portland cement, influences the great splittensile strength of concrete containing nanoparticles. The 28-day strength of concrete rise by almost 47.58% after replacing 1.0% NS in the concrete. The 28-day split tensile strength helps to elevate by 59.16% after returning 0.06% MWCNT in the concrete.

### 3.3 Durability Tests

Fig. 6(a) shows the water absorption value of different combinations of NS and MWCNT. The value obtained for control concrete was 7.39%. Adding 0.3% of NS shows a water absorption value of 6.25%, and it decrease by 15.42%. The water absorption value obtained was 6.48% with the addition of 0.03% of MWCNT, and the reduction in water absorption value was 12.31%. The combined percentages of 0.3% NS and 0.03% MWCNT produced 5.68%, which is 23.1% lower compared to control concrete. The three different percentage combinations of 0.6% of NS, 0.045% of MWCNT, and 0.6% NS + 0.045% MWCNT provided water absorption values of 4.74%, 4.1%, and 3.72%, respectively. The percentage of water absorption decreased from 35.8% to 49.6%. Adding 1% of NS and 0.06% of MWCNT causes a reduction of 46.8% from 64.8%. A combination of both NS and MWCNT paved the way for obtaining a good performance in all the combinations.

Fig. 6(b) shows the acid attack value of different combinations of NS and MWCNT. The acid attack value obtained for control concrete was 21.34%. The percentage

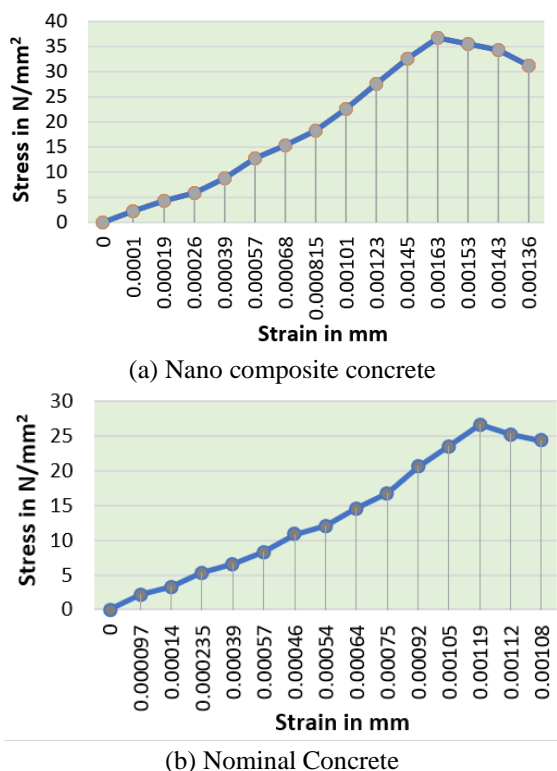


Fig. 7 Stress-strain curve of nominal and Nanocomposite concrete

loss in the weight of concrete was noted as 18.06% with the addition of 0.3% of NS, while the decrease in acid attack was 15.37%. The loss in weight of concrete was 6.48% with the addition of 0.03% MWCNT, which was 16.49% lesser compared to control concrete. Combination of 0.3% NS and 0.03% MWCNT resulted in 15.87% loss in concrete and 25.63% decrease in acid attack. Three different percentage combinations of 0.6% of NS, 0.045% of MWCNT, and 0.6% NS + 0.045% MWCNT infer the loss in weight of concrete by 14.98%, 14.93%, and 12.53%, respectively. The decrease in the percentage of acid attacks was observed from 29.8% to 41.28%. The addition of various combinations of 1% NS and 0.06% MWCNT cause a loss in the weight of concrete from 47.14% to 56.4%. A variety of both NS and MWCNT gave better resistance to the acid attack in all the varieties.

Fig. 6(c) shows the sulphate attack value of different combinations of NS and MWCNT. The sulphate attack value of control concrete was found to be 14.92%. Adding different varieties of 0.3% NS and 0.03% MWCNT caused a loss in the weight of concrete from 10.45% to 9.68%. The 0.6% NS and 0.045% MWCNT combinations decreased the sulphate attacks from 52.61% to 53.82%. The three different percentage combinations of 0.6% of NS, 0.045% of MWCNT, and 0.6% NS + 0.045% MWCNT resulted in a loss by 5.59%, 4.86%, and 3.26%, respectively. The decrease in the percentage of sulphate attacks varies from 62.5% to 78.1%. The combined proportions of NS and MWCNT helps to get a good performance in sulfate attacks. Fig. 6(d) shows the chloride attack value of various combinations of nanoparticles. Adding 0.3% NS and

0.03% MWCNT caused a reduction in the sulfate attack from 11.3% to 21.9%. Adding 0.6% NS and 0.045% MWCNT cause a reduction from 24.7% to 43.2%, while adding 1% NS and 0.06% MWCNT tends to decrease the sulphate attack from 32.11% to 53.3%.

Fig. 6 shows the replacement of NS in concrete resulted in a significant increase in durability, with a reduction in water absorption by 46.8%, acid attack by 47.1%, sulphate attack by 62.4%, and chloride attack by 32.1% when 1.0% of NS was replaced. When solely replacing NS in concrete, the maximum increase in durability is observed when 0.06% of MWCNT is replaced, resulting in significant improvements. The water absorption is reduced by 56.8%, acid attack by 48.6%, sulphate attack by 67.4%, and the chloride attack by 43.4%. Through the combined replacement of 1.0% NS and 0.06% MWCNT in concrete, a significant enhancement in durability is achieved, when the water absorption is reduced to 64.8%, acid attack decreased to 56.4%, sulphate attack decreased to 78.1%, and chloride attack lowered to 53.4%.

CNTs have a pore-filling and nucleation effect, which improves the durability properties and lowers the number of micropores and nanopores in concrete. Nanomaterials improve concrete resistance to freezing and thawing, reducing water permeability, sorptivity, chloride migration, and infiltration of harmful agents, thereby increasing the density and crack resistance. Hence, the durability of concrete improves when nanomaterials are used and the concrete should be free from chemical attacks to ensure the structure durability. Nanocomposite concrete enhances durability by reducing water and chemical ingress, thus minimizing the corrosion and deterioration of structural elements over time.

### 3.4 Stress-strain characteristics

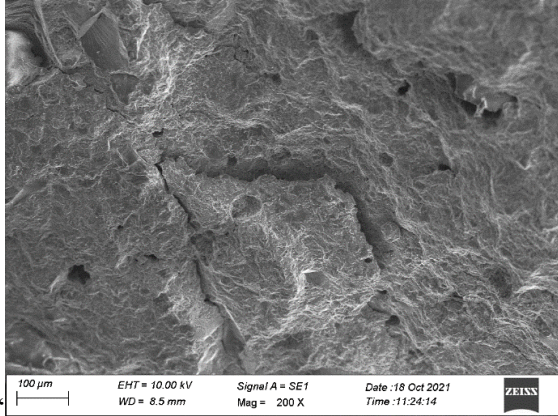
The stress-strain curves depicted in Fig. 7(a)-(b) indicate that the behavior of nominal and hybrid nanocomposite concrete mixes under stress and strain appears similar. The main difference is that nanocomposite concrete mixes have shown improved stress values for the same strain levels compared to control concrete mixes. The nominal concrete stress-strain curve has a slightly linear and steeper ascending portion. The strain at peak stress is somewhat higher, and the falling portion's slope is more steeper than nominal concrete. This is due to the reduction in internal cracking of nanocomposite concrete.

### 3.5 Microstructure analysis results

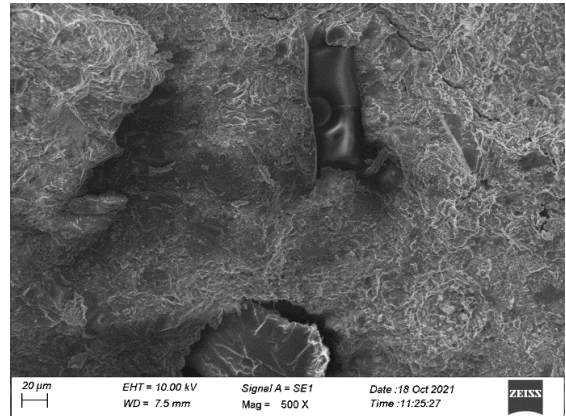
#### 3.5.1 SEM analysis results

Microstructural analysis of the prepared cement slurries performed by a scanning electron microscopic technique showed the modified cement matrix in terms of carbon nanotubes along with nanosilica. Qualification of the micromechanical level of 0.06% carbon nanotubes with 1% nano-silica materials examination was done with a resolution of micron range of matrix with the new products of SEM images shown in Fig. 8(a)-(g).

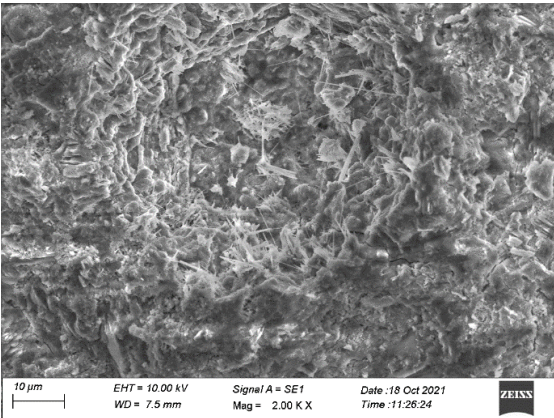
It examined in turns to determine their strength characteristics of applied concrete materials withstanding



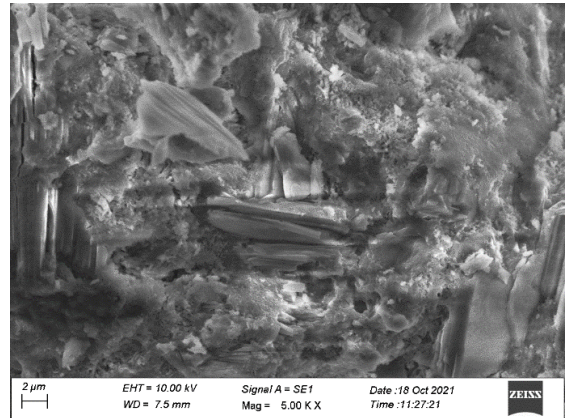
(a) Magnification image 200X



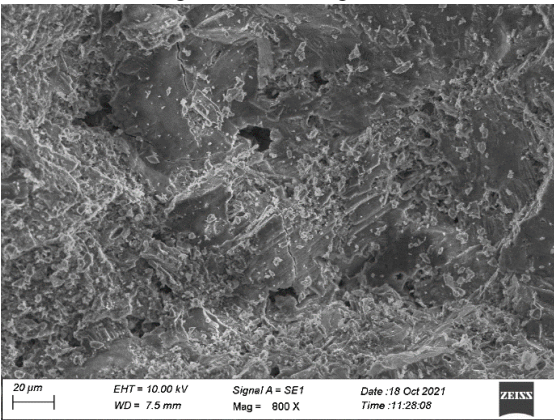
(b) Magnification image 500X, WD 7.5mm



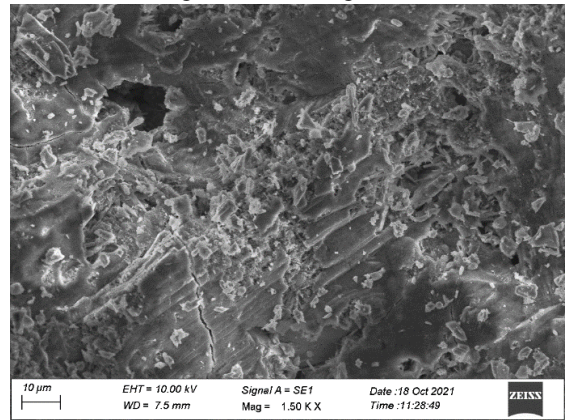
(c) Magnification image 2.00KX



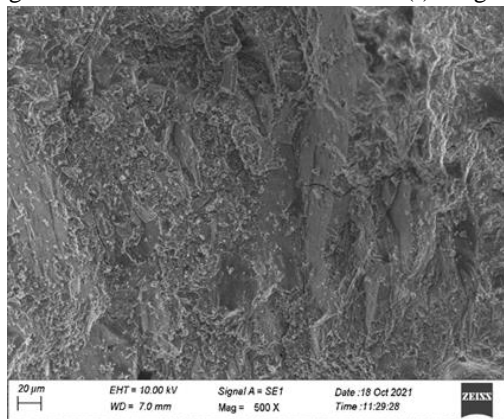
(d) Magnification image 5.00 KX



(e) Magnification image 800X



(f) Magnification image 1.50 KX



(g) Magnification image 500X, WD 7.0mm

Fig. 8 SEM analysis of 0.06% CNT and 1% NS

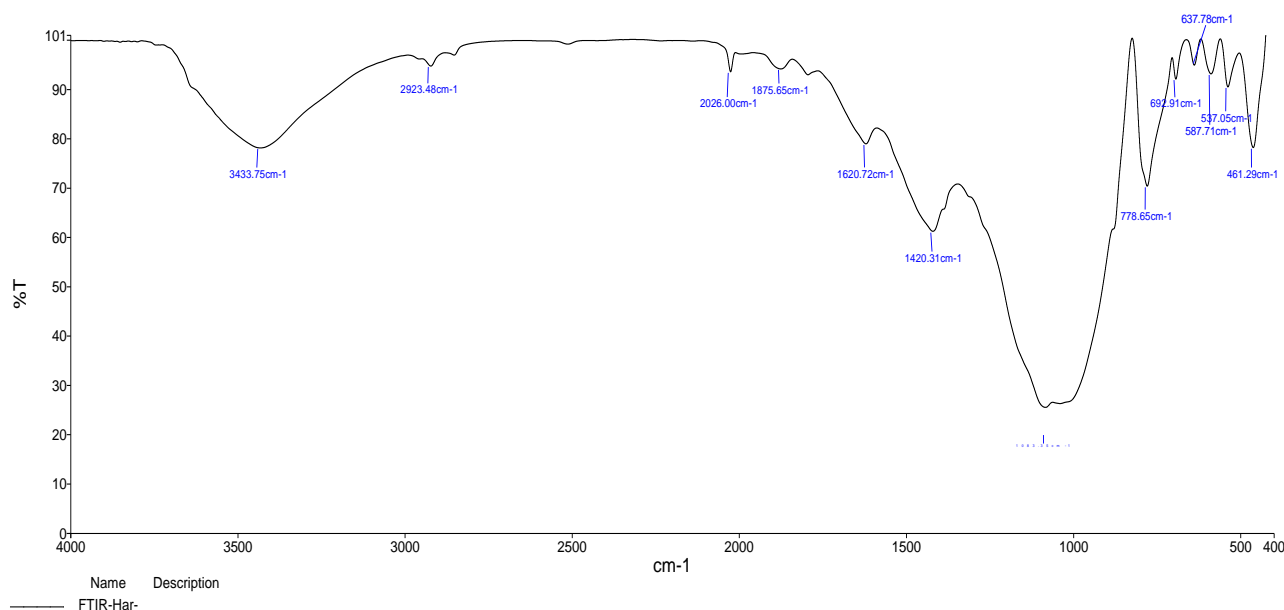


Fig. 9 FTIR analysis of hybrid CNT and NS-based concrete

stability. The hardened specimens based on 0.06% MWCNTs and 1% NS were investigated after 14 days of hydration at 90°C. The prepared composition of the cement matrix of these specimens provided information relating to the formation of needle-like, freshly formed crystals along with carbon-hydrogen linkage. The pores in cement paste can be filled with tiny particles of nanomaterials, improving the microstructural packing and making the cement matrix more impermeable. Single-walled carbon nanotubes are rope-like bundles and in contrast, Multi-walled carbon nanotubes are agglomerated and needle-like structures.

The evaluation of the degree of hydration of calcium hydroxide components within the prepared matrix involved the utilization of two forms of calcium silicate: one hydrated in a gel-like state with three molecules of water and the other with two molecules of water. This assessment was conducted by observing the enthalpy-changing process as the water molecules present on the surface of the modified matrix evaporated, leaving behind tiny holes.

The deposit of the C-S-H phase was seen with pores, and silica's crystalline nature provides high compressive strength. The hardened oil well cement (OWC) pastes mixed with 0.06% carbon nanotubes after hydration of 14 days at 90°C indicated the uniform disposal of carbon nanotubes in the OWC pasted aggregation. The resulting carboxyl group improved the bond strength and the degree of crystallinity between MWCNTs and silica-modified cement matrix due to the aromatic ring and hydrophilic group being connected by long carbon chains, according to this aromatic ring attached to the surface of MWCNT. In contrast, the hydrophilic group increased dispersibility in water. During the cement hardening process, carbon nanotubes and nano-silica composition in the concrete form crystalline hydrates. These hydrates create a thin layer of dense new formations with high-strength shell-like components, which envelop the surface of the solid phase. The morphological images depicted in Fig. 8(e)-(f) showcase

the presence of needle-like crystalline structures, which exhibit agglomeration. In the modified concrete, the direct crystallization of new mineralogical composition changes, as well as the alkalinity of calcium hydrosilicates, helped to predict the packing effect of Vanderwall force between small carbon nanotubes with nano-silica as fillers, filling the interstitial spaces in the hydration products with increased cement intensity. The role of the inorganic nanosilica material lies in improving the morphological and structural properties and enhancing the rate of hydration process and microstructure. NS refined the pore nature of the material and reduced the pore volume of cement pastes during the exhaust process of fly ash to improve the cement durability and mechanical strength. NS can prevent chloride ion concentrations and reduce the alkaline nature of the environment, acting as the corrosion-resistant material of reinforced concrete.

CNT-integrated specimens with the optimum amount of 0.1% samples increased the compressive strength and improved the crystalline nature of hydrated products, as clearly shown from images of Fig. 8(a)-(g) compared to SEM images of CNT-integrated specimens with NS evaluated by Mishra (2022). The addition of NS in cement enhances the pozzolanic activity and forms additional hydration products. It has been proven to have a homogenous, compact, denser, and glassy appearance with a needle-shaped microstructure compared with the investigation done by (Narasimman *et al.* 2020a, b). SEM and XRD analyses verified the development of more hydrated products in the presence of nanosilica and CNT. Additionally, adding nanomaterials to concrete mixes enhances the binding strength at the cement paste and aggregate interface in the hardened cement paste.

Additionally, the use of nanomaterials results in a cement matrix that is more impermeable and has superior microstructural packing. Indirectly, it will strengthen the surfaces made of hardened, porous concrete. Compared to

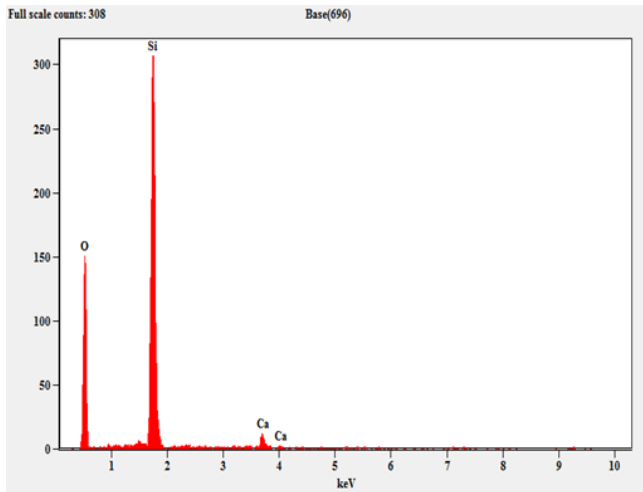


Fig. 10 EDX analysis

Table 5 Results of EDX analysis

Element Line	Weight %	Weight % Error	Atom %
O K	57.41	± 1.52	70.79
Si K	39.24	± 0.78	27.56
Si L	---	---	---
Ca K	3.35	± 0.36	1.65
Ca L	---	---	---
Total	100.00		100.00

the control specimen, the CNT specimen is highly structured and dispersed, which confirms that CNTs perform as bridges across the microcracks, which helps to increase the bondability of concrete.

### 3.5.2 FTIR analysis results

Cement composite applied materials like CNTs with NS did not indicate hydrogen bonding between oxygen and hydrogen at  $3600\text{ cm}^{-1}$ . The FTIR spectrum obtained for hybrid nanomaterial-based concrete is shown in Fig. 9. The intermolecular hydrogen bonding formation was shifted to  $3433\text{ cm}^{-1}$ , which can be attributed to hydroxyl stretching vibration. The bands around  $2923$  and  $2026\text{ cm}^{-1}$  indicated the symmetric and asymmetric stretching of C-H vibrations. Vibrational bands like  $1875$  and  $1620\text{ cm}^{-1}$  could be attributed to MWCNTs in carboxylic acid groups' IR active stretching mode. The  $3300\text{--}3500\text{ cm}^{-1}$  also indicated hydrogen bonding of the OH group on the surface of NS and peaks between  $1000\text{--}1200\text{ cm}^{-1}$ , confirming the formation of Si-O-Si bond. The rise at  $1620\text{ cm}^{-1}$  predicted the stretching vibration of the carbon-carbon double bond mode of MWCNT. This elucidated how the adhesion of NS undergoes SP3 hybridization on the surfaces of carbon nanotubes in modified concrete. The  $2923$  and  $2853\text{ cm}^{-1}$  peaks could be attributed to the formation of MWCNT with NS composite, which corresponding to the stretching vibrations of carbon and hydrogen single-bond modes and methylene groups. NS adhered to the surface of CNTs, resulting in weak defects. The peaks observed in the range of  $600\text{--}700\text{ cm}^{-1}$  could be attributed to the C-H deformation

of modified concrete materials.

The FTIR spectra of the cement samples with and without CNTs were investigated, as well as the shifting process of the characteristic peak from  $921\text{ cm}^{-1}$  and  $983\text{ cm}^{-1}$ . The wave number shifting values of the NS and carbon nanotube samples exhibited high consistency. It did not affect the polymerization made by C-S-H gel at an early stage by Ylmen and Jaglid (2013). Calcium hydroxide and calcium silicate hydrate in cement were carbonated, enhancing the carbon dioxide uptake investigated through FTIR spectroscopy (Shi *et al.* 2019a, b). Accordingly, the modification of carbon nanotubes with NS functionalization in cement, including exposed and unexposed carbonation, has been evaluated.

### 3.5.3 EDX analysis

The nano-silicon dioxide film on the MWCNTs-modified cement materials can consume calcium hydroxide to increase C-S-H gel content at the interface between carbon nanotubes and cement matrix composite materials, as shown in Fig. 10. Immersion tests in saturated calcium hydroxide solution-modified samples were carried out to verify the reactivity of nano-silicon dioxide in the stimulated hydration environment after immersion for 3 days. The Ca/Si ratio weight in the percentage modified MWCNT surface was 1.65. Gelatinous substances were seen following immersion for 7 days. An interconnected three-dimensional network structure increased the average ratio of Ca/Si on the surface of the modified surface of carbon nanotubes to 3.35. The EDX spectrum of modified cement materials showed a sharp peak of silicon and oxygen atoms, confirming the high chemical purity of nano-silica with MWCNTs in the modified cement matrix.

Needle-like nanostructures were grown by the thermal annealing process, as indicated by the elemental analysis (Duraia *et al.* 2012a, b). The molar ratio of calcium and silicon and the effect of functionalized carbon nanotubes were investigated by (Li *et al.* 2020a, b), so the presence of the molar ratio of elements like Ca, C, O, and Si has been predicted from the EDX analysis simultaneously.

## 4. Conclusions

Small amounts of nanomaterials can enhance the properties of concrete by densifying the cement structure by filling voids, speeding up the hydration process, and enhances the compressive strength. At an early stage, compressive strength is improved by the addition of NS. Initially, nanoparticles promote the degree of hydration, making the concrete matrix to produce more C-S-H gel. On the other hand, inactive nanomaterials play a filling role, make the concrete more compact, and exhibit superior durability. Based on the experimental findings and analysis conducted in the study, it is evident that nanotechnology presents a promising avenue for enhancing the sustainability and performance of concrete in the construction industry. Incorporating nanoparticles such as nano silica and Multi-Walled Carbon Nanotubes in green concrete mixes has shown notable improvements across critical parameters like

strength, durability, and micro-structural analysis. The following conclusions have been made regarding nanotechnology influence on concrete properties in the construction industry.

- The study found that replacing 1.0% NS with 0.06% MWCNT in concrete resulted in a 37.49% increase in 28-day compressive strength. Compared with earlier research, adding 1% NS and 2% CNT with cement leads to 38.7% increase, suggesting that a smaller proportion of about 0.06% CNT was sufficient for significant strength gain.

- The specimens with 1.0% NS and 0.06% MWCNT showed the highest improvement 90.99% of split tensile strength at 28 days.

- The maximum reduction in the percentage of water absorption was observed in the specimens with 1.0% NS and 0.06% MWCNT, which was 64.81% better than control concrete.

- When subjected to acid, sulphate, and chloride attacks, concrete with 1.0% NS and 0.06% MWCNT experienced the least percentage weight loss compared to control concrete. The weight loss percentages were 9.30% for acid attack, 3.26% for sulphate attack, and 7.01% for chloride attack, demonstrating the superior resistance to environmental deterioration. SEM analysis revealed the presence of the C-S-H phase with pores, indicating the crystalline nature of silica and its role in enhancing the compressive strength when 0.1% NS and 0.06% CNT were added. NS was found to adhere with the surface of carbon nanotubes, leading to identified weak defects in FTIR analysis. Additionally, the EDX spectrum displayed prominent peaks of silicon and oxygen atoms, highlighting the chemical purity of nanosilica with MWCNT in modified cement materials.

The investigation has highlighted several areas requiring further research. Future studies should explore the effects of higher dosages of NS and CNTs on the mechanical and durability properties of concrete or cement composites. Additionally, partial substitution of cement with both NS and MWCNT could lead to the development of sustainable construction materials with reduced CO<sub>2</sub> emissions. Despite being costly, nanomaterials offer the significant advantage of providing long-lasting durability and high strength with minimal supplementation to cementitious materials in concrete.

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