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Research Article

Mechanical, Durability, and Microstructure Investigations on High-Strength Concrete Incorporating Nanosilica, Multi-Walled Carbon Nanotubes, and Steel Fibres

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In present research, the strength properties, impact resistance, and durability characteristics of high-strength concrete blended with nanosilica (NS) and reinforced with multi-walled carbon nanotubes (MWCNTs) are discussed. The proportion consists of nanosilica added in a constant addition of 1% and MWCNT added in a varied dosage of 0.025%, 0.05%, 0.1%, 0.15%, and 0.2% by weight of the cement. A total of 11 mixes were made including the control mix having no MWCNT. The other 10 mixes were categorized into two classes with one class having steel fibres incorporated as 1% of the total volume of the concrete along with the other ingredients such as 1% NS and different proportions of MWCNT. The other class was made without steel fibres retaining only the NS and different MWCNT proportions. Besides the standard compression and tension tests, to determine the energy absorbing capacity of the mix specimens, impact test was also performed. The strength tests were carried out for 3, 7, and 28-day curing. Also, durability tests were carried out with sorptivity, porosity, and mass loss of the specimens when exposed to aggressive HCL and H_2SO_4 acid. To validate the experiment results, microstructure studies such as scanning electron microscopy (SEM) were also conducted on the samples. Among all mixes, 28-day compressive strength (CS) of 0.2% MWCNT with 1% NS and 1% steel fibre mix was found to increase by 22% compared to control concrete.

1. Introduction

The use of nanomaterials in the building industry has received substantial attention in recent studies due to various characteristics they possess such as their ability to act as a filler material by filling up the tiny gaps or pores present when utilizing traditional materials in concrete. Materials like nanosilica have the ability to function both as a pozzolanic material and as a filling agent. A thorough review on the use and applications of various nanomaterials in the construction field had been presented by Muhd Norhasri et al. [1]. Numerous research studies have used a variety of nanoparticles as mineral admixtures, and nano-ZnO_2 [2], nano-$CaCO_3$ [3], ultra-fine/nano-TiO_2 [4–7], and nano-SiO_2 are some of the nano-sized mineral admixtures which are frequently used by researchers. However, among all the nanoparticles mentioned, nano-SiO_2 is the one that is most widely used in concrete technology because of its high reactivity and higher specific surface area, which promote better pozzolanic reactivity and result in a denser calcium silicate hydrate (CSH) gel with improved strength and durability. Research works of Zhang et al. [8] and Abhilash et al. [9] dealing with the effects of NS in enhancing the mechanical, durable, and microstructure characteristics in the concrete in fresh state and in hardened state also confirm this. Amin et al. [10] and Ershad et al. [11] in their research works have stated that NS can frequently be employed to increase the slurry's impermeability and the hardened material's mechanical qualities, so it is obvious that starting from the fresh properties such as slump to hardened properties, incorporation of nanosilica has proved its positive impression in the mix properties. Though the high specific surface area can help improving the consistency of the mixes, it may also affect the flow and pore filling effect [12]. Research reports of Supit and Shaikh [13] also confirm this as it is mentioned that incorporation of nano-SiO_2 in 2% and 4% in concrete may have the tendency to reduce the slump by about 40% and 60%. So, El-Gamal et al. [14] had insisted that 1% of NS is the best dose to use in concrete at both normal room air temperature and higher healing temperature. Despite the few problems occurred regarding the flow, reports from earlier research [15–18] have proved that there is indeed an improvement in the compressive strength as the blending is controlled due to SiO_2 nanoparticles.

1.1. Use of CNT and MWCNT. Recent studies strongly advocate the hybridization or combining of components with various qualities. The drawback of one component can be offset by the advantages of another component by using hybridization [19]. Carbon nanotubes have been used in a variety of construction-related applications, and in the current study, a similar attempt has been undertaken employing CNT as the additional material to be combined with nanosilica. Better outcomes can be anticipated from hybridization because CNTs are smaller in size to use such a nano-sized component in a macroproduct like concrete. CNTs are five times less dense and have ten times stronger tensile strength and Young's modulus values than steel. Additionally, CNT can be spread broadly and densely at the microscopic scale, covering larger lengths, and have a very high length to diameter ratio. To fill fractures and stop them from spreading, cement composites can make use of these properties. As a result, CNTs can essentially be employed to create a new generation of cement materials that are crack-free [20]. CNTs are essentially the ideal reinforcing materials owing to their extraordinarily high aspect ratios and capacities. Further possessing excellent soundness and flexibility, CNTs also exhibit large aspect ratios, which are frequently greater than 1000 : 1 and as high as 2.500.000 : 1 [21]. Because of their extremely high aspect ratios [22], compressive strength [23, 24], flexural strength [25], high modulus [26], and elasticity [27], CNTs are considered a fantastic material choice for outstanding composite matrix reinforcing goods. The reason for such excellent properties of CNT may be attributed to their nanostructure [28]. Liew et al. [29] indicated that because of their nanoscale size, CNT and other nanomaterials can impede the spread of microcracks in concrete. In their experimental studies comparing separately the outcomes of concrete prepared with CNT and silica fume, Chukka et al. [30] found that CNT-infused concrete performed better than conventional and silica fume-mixed concrete Additionally, Dietzel et al. [31] demonstrated that single-walled CNTs' mechanical characteristics are remarkable when compared to typical conventional fibres. Similar to single-walled CNTs, MWCNT inclusion to improve the toughness and mechanical properties when combined with cement has been the focus of various studies. Wrapping numerous graphene layers into a series of concentric tubes results in multi-walled nanotubes (MWNTs). Gillani et al. [32] improved the mechanical properties of mixes with cement composites by including MWCNTs. The results showed that the compressive strength was influenced by the amount of MWCNT in the concrete matrix. Fraga et al. [33] have proved that MWCNTs, in concentrations of 0.05 to 0.5 weight percent, can significantly improve the mechanical properties of cement. By functionalizing the multi-walled carbon nanotubes with carboxylic groups using MWCNT, Hu et al. [34] and Mansouri Sarvandani et al. [35] were able to boost the compressive strength, fracture toughness of concrete, and durability of concrete. Other notable works in concrete using MWCNT include those by Li et al. [36], in which it was reported that the flexural and compressive strengths of cement matrix composites containing MWCNT increased by 25 and 19%, respectively, even after exposed to aggressive $H2SO_4$ and HNO_3 environment. The strength characteristics of concrete containing both short and long MWCNT were investigated by Al-Rub et al. [37], and their results showed that 0.2% of short MWCNT displayed a growing trend in flexural strength by 269%, whereas 0.1% of long MWCNT exhibited a 65% improvement in strength. From the above discussed research works, the advantages of using nanomaterials and nanotubes in concrete are understood. In present work, an attempt has been made to utilize the advantages of two nano-sized materials to develop a mix which is denser, strong, and durable.

1.2. Use of Steel Fibres along with MWCNT. In general, steel fibres are recognized for their good bridging effect, better tensile properties, proven impact resistance, and arresting of crack propagation characteristics [38–40], and so they can be utilized to enhance the durability characteristics as they help in reducing propagation of cracks. The diverse mechanical properties of concrete prepared with NS individually and combined with NS and MWCNTs, for various amounts of nanomaterials, different curing periods, and incorporation of steel fibres, were discussed by Sumathi et al. in their study [41–43], and the authors in their research using steel fibres indicated that incorporation of 1% improved the mechanical and structural properties of high-strength concrete. Murali et al. [44, 45] have tested the impact strength of concrete specimens made with MWCNY and different fibres such as short steel fibre, long steel fibre, and polypropylene fibres and have reported that addition of fibres substantially increased the fracture resistance of the specimens. Hassan et al. [46] examined the impact of adding steel fibres (SFs) and carbon nanotubes (CNTs) to in 0%, 0.025%, 0.050%, and

0.075%, and the authors in their findings reported that 0.05% carbon nanotubes remained the ideal level for mix as the combination provided the highest compressive, tensile, and flexural strengths when compared to other concrete mixtures with different concentrations of CNTs. Additionally, they also suggested that using silica fume can enhance the mix's mechanical qualities and they can increase the mix's postcracking and fatigue behaviour as well as its mechanical qualities.

1.3. Research Significance. The major challenges to the utilization of carbon nanotubes in the cement matrix are their dispersion and their bonding with cement hydration products [32]. Mendoza et al. [47] reported that MWCNT dispersion in water will induce agglomeration and mentioned that a mere combination of two nanomaterials such as NS and MWCNT will not exhibit promising results in strength tests as the agglomeration may affect the reactivity of the NS and suggested that a suitable proportion of the nanomaterials should be properly chosen to avoid the agglomeration. So, in current research, the ingredients for the mixes were chosen such that they properly fill the pores and do not form any agglomeration, and the work was formulated to utilize the advantages of two nano-sized materials of different compositions, namely, MWCNTs and nanosilica, for producing a densely packed concrete. In addition to that, steel fibres which are recognized for their good bridging effect, better tensile properties, proven impact resistance, and arresting of crack propagation characteristics were also used to enhance the durability characteristics by reducing propagation of cracks. Though CNT had been in use in the construction field for quite a long period, most of the research done using them focused on strength improvement and electrical resistivity improvement [48]. Only limited works were available on durability of concrete with CNT and on energy absorbing characteristics of mixes with MWCNT [49]. Present research aims to fill this research gap also by developing a mix using MWCNT to possess the necessary strength to withstand the impact resistance as well as to perform well in aggressive environment. Thus, this work is a next logical step to develop a hybrid fibre-reinforced concrete that incorporates different sizes of fibres ranging from nanometer to millimeter length scales.

2. Materials and Experimental Investigations

2.1. Materials. Ordinary Portland cement of grade 53 with a specific gravity of 3.15 which complies with the IS 12269 (1997) specifications [50] was used throughout the work. The physical properties of the fine and coarse aggregates were also checked, and the fineness modulus was found to be of 2.7 and 6.7, respectively. The fine aggregate passing through a 4.75 mm sieve and coarse aggregate with a range between 12.5 mm and 20 mm were used in present experimental study. Superplasticizer in the range of 2.5 to 5 ml per kg of cement was used. Figure 1 shows NS with a density of 2.4 g/cm^3, a molar mass of 59.96 g/mol, and a colour of white powder procured from ASTRA chemicals which was used in this work. MWCNT was sourced from Go Green Technologies, Guntur. Figure 2 shows the appearance of used MWCNT. The dosage adopted for MWCNT usage was calculated based on the quantity of cement used as reported in various literatures. The properties of MWCNT are presented in Table1. Steel fibres of type long crimped end with length of 5 cm and diameter of 0.1 cm were considered. Aspect ratio of the steel fibres is 50 as shown in Figure 3. The chemical components present in the cement and NS were identified by X-ray fluorescent method and are exhibited in Table 2.

Figure 1: Nanosilica.

2.2. Methodology Adopted. The methodology adopted has been presented graphically as shown in Figure 4.

2.3. Mix Details. M60 with a mix ratio of 1 : 1.08 : 1.86 : 0.38 was done in accordance with IS 10262-2019 [51]. Eleven different mixtures were prepared for the present research. The control concrete is labelled as CC. From CC, other mixes were developed by adding NS in 1% as cement replacement as constant addition and MWCNT in various percentages, such as 0%, 0.025%, 0.05%, 0.1%, 0.15%, and 0.2%. The other 10 mixes were categorized into two classes with one class having steel fibres incorporated as 1% of the total volume of the concrete along with the other ingredients such as 1% NS and different proportions of MWCNT. The other class was made without steel fibres retaining only the NS and different MWCNT proportions. Table 3 shows the details of 11 mixes adopted and their proportions. Superplasticizer and aqueous solution were used as surfactant. The aqueous solution is divided into three parts. MWCNTs are taken in batches and mixed in water thoroughly along with the superplasticizer. The steps involved in mixing MWCNT are as follows: initially, the needed amount of MWCNT was combined with half of the water that included superplasticizer, and the mixture was stirred using a mechanical stirrer for 30 minutes. The remaining water was then added, and mixing was continued slowly for the following 60 seconds to produce a scattered MWCNT. It is then poured into concrete after proper mixing. Again, the whole mixture is continuously mixed in such a way that the nanomaterials avoid agglomeration in the concrete mixture.

2.4. Specimen Details. 100 × 100 × 100 mm cubes were cast and tested for compressive strength at 3, 7, and 28 days of curing. For testing tensile strength, 100 × 200 mm cylinders

Figure 2: Carbon nanotubes.

Table 1: Multi-walled carbon nanotubes.

Multi-walled carbon nanotubes	Description
Purity	~99%
Outer diameter	10–30 nm
Inner diameter	5–10 nm
Length	>10 μm
Surface area	110–350 m^2/g
CNT content	~95–99%
Bulk density	0.14 g/cm^3
Chemical formula	C
Physical form	Fluffy, very light powder
Odour	Odourless
Colour	Black powder

Figure 3: Crimped steel fibres.

Table 2: Chemical components in cement and nanosilica.

Component	Concentration in percentage	
	Cement	NS
SiO_2	19.5	99.81
Al_2O_3	3.83	0.06
CaO	65.13	0.03
Fe_2O_3	5.86	73 PPM
MgO	0.77	—
TiO_2	0.25	—
SO_3	3.62	0.05
K_2O	0.8	—
Na_2O	0.19	—
P_2O_5	0.16	—
PbO	—	0.03
CeO_2	—	—
V_2O_5	—	—
Cl	0.08	—
Cr_2O_3	0.03	—
ZrO_2	—	—
Pd	—	71 PPM
NiO	—	—
ZnO	0.03	96 PPM
CuO	—	—
MnO	0.08	—
SrO	0.05	—

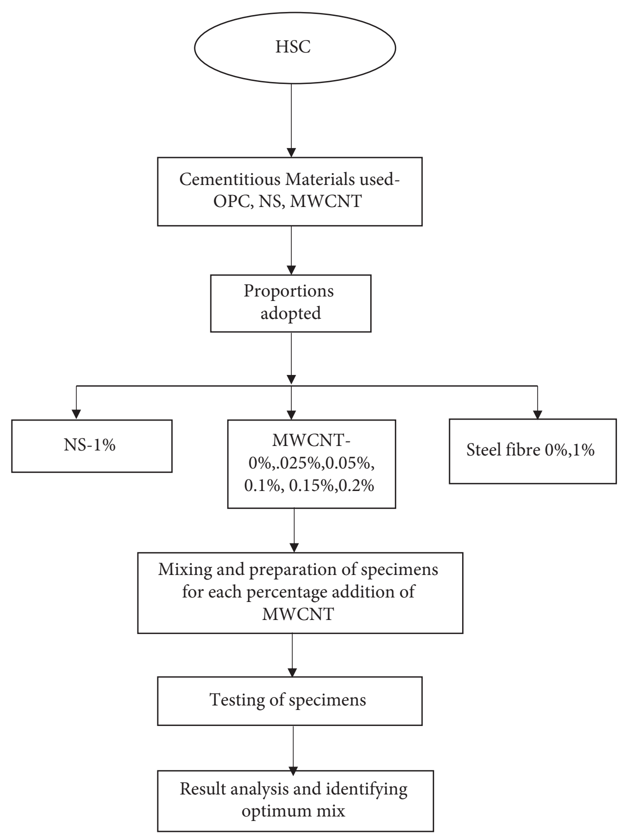


Figure 4: Methodology.

were prepared and tested at 28 days as per ASTM C496 [52]. In accordance with ACI 544 [53], cylindrical discs of 150 mm in diameter and 64 mm in height were made and were tested after 28 days. 100 mm diameter and 50 mm height cylindrical discs were cast for the sorptivity and porosity studies and were tested as per ASTM 1585-20 [54] and ASTM C642 [55], respectively. 100 × 50 mm discs were cast to test acid and sulphate resistance. For water absorption test, 100 mm cube specimens were used.

2.5. Curing Environment. Sulphate attack is a familiar factor which is encountered by concrete buildings frequently when exposed to aggressive environment. To evaluate its severity, the specimens were initially cured in normal water for 28 days and after completing their prescribed curing period in normal water they were cured in water diluted with 1 percent H_2SO_4 and were cured in that state for a duration of two months to initiate accelerated degradation and deterioration. Since sulphate attack is one of the most prevalent causes of concrete deterioration [56] and causes serious

Table 3: Mix proportions.

S. No.	Cement (kg/m^3)	Nanosilica (kg/m^3)	w/b ratio	Fine aggregate (kg/m^3)	Coarse aggregate (kg/m^3)	MWCNT (%)	Steel fibre (kg/m^3)
1	554.4	5.6	0.38	604.8	1041.6	0	—
2	554.4	5.6	0.38	604.8	1041.6	0.025	—
3	554.4	5.6	0.38	604.8	1041.6	0.05	—
4	554.4	5.6	0.38	604.8	1041.6	0.1	—
5	554.4	5.6	0.38	604.8	1041.6	0.15	—
6	554.4	5.6	0.38	604.8	1041.6	0.2	—
7	554.4	5.6	0.38	604.8	1041.6	0	7850
8	554.4	5.6	0.38	604.8	1041.6	0.025	7850
9	554.4	5.6	0.38	604.8	1041.6	0.05	7850
10	554.4	5.6	0.38	604.8	1041.6	0.1	7850
11	554.4	5.6	0.38	604.8	1041.6	0.15	7850

damage including the spreading of concrete cracks and dissolution of cement paste, it is crucial to research how high-strength concrete responds to any new mineral additive or fibre. Additionally, the specimens were exposed to 1 percent HCl for a duration of two months to evaluate the characteristics when the specimens confronted chloride attack because chloride attack on specimens creates major durability concerns [57]. Therefore, they were tested in this research work to check whether the specimens well-equipped with NS and CNT fill the voids in concrete and thereby help to prevent deterioration leading to strength in concrete. The solution was changed on a regular basis to maintain a steady concentration throughout the experiment. The sample's percentage weight loss was calculated. Figure 5 shows the deteriorated specimens because of exposing to aggressive acid environment.

Figure 5: Specimens after exposed to acid and sulphate.

3. Experimental Investigations

3.1. Strength Tests and Impact Resistance. To evaluate the capability of the specimens in resisting the applied loads, in a typical setting, compression and splitting tensile strength tests were carried out. 150 mm × 64 mm cylinder specimens were used to test the impact resistance in accordance with ACI 544 [58] which involved repeated striking by a 64 mm hardened steel ball on top and at the centre of the cylindrical specimen with a hammer that weighed 44.7 N from a height of 1120 mm. For each specimen, the number of strikes necessary for the creation of the first crack and the number of blows necessary for the specimen's failure were recorded, and the related impact energy was determined.

3.2. Sorptivity. Finding the percentage, finding the rate, and finding the coefficient of water absorption are a few techniques that are widely used to assess the water permeation in concrete. Water is absorbed through sorption when capillary suction is present. The water permeability of the specimens in this study was determined using the techniques outlined in ASTM C1585 [54] and ASTM C642 [55]. Cylindrical specimens of 100 mm in diameter and 50 mm in height were employed in the current study. The specimens were dried in an oven at roughly 110°C after completing their 28-day period of standard curing. The specimens that had been baked in the oven were then kept in a sealed container to cool to room temperature. Except for the bottom surface, the specimen surfaces were coated with nonporous insulation tape, as illustrated in Figures 6(a) and 6(b). To guarantee that water entered the specimens solely through the bottom, they were submerged barely 3 to 5 mm above the surface. Periodic weights of the specimen were taken as per ASTM C1585 guidelines. A graph was plotted between the mass per unit area of the specimen and the square root of time, and the sorptivity was obtained from the slope.

3.3. Porosity Test. By completing a porosity test, the specimen's durability was evaluated. This test covers the density, percentage of absorption, and pores present in the different concrete mixes. ASTM C642 [55] was followed in performing the test. Cylindrical discs of size 100 mm × 50 mm were made for a range of cement replacement percentages and maintained for 28 days to cure. The porosity of the specimens was computed after the weights of all these specimens were arrived at under various conditions, like oven dried weight and saturated weight after being kept in water for 24 hours, as indicated in the codal regulations.

4. Results and Discussion

4.1. Compressive Strength. Figures 7 and 8 depict the 3, 7, and 28-day average compressive strength of concrete made with and without steel fibres, respectively. For both the categories, a gradual increase with the increase in MWCNT content was noticed. It is also clear that despite the small addition of MWCNT in 0.025%, 0.05%, 0.1%, 0.15%, and 0.2%, the gradual increase in strength indicates the influence of MWCNT in strength improvement. Aydın et al. [59] in their results indicated that mixes with CNT yielded better strength improvement even for a small addition of 0.08% in concrete. From Figure 8, it is obvious that even without steel fibres, the NS and CNT combination showed an increase in strength by 5.54%, 12.03%, 15.04%, 16.93%, and 18.2% more than the control mix (CC). The higher compressive strength may be attributed to the presence of the NS and the CNT. The main reason for the drastic strength increase was due to the high reactivity property and nucleation effect of NS [60]. The observations are analogous to those of Lee et al. [61], in which the authors reported that CNT and NS were used with NS contributing to pozzolanic activity and filling effect and CNT improving the strength by controlling the crystal growth. Figure 9 displays the strength of the specimens with steel fibres incorporated along with the NS and CNT combinations. The specimens showed a much higher strength of necting the tiny gaps present in the speci 7.34%, 12.25%, 16.4%, 18.86%, and 21.78% than the control concrete. The higher strength was possible due to the combined bridging effect of steel fibres and MWCNT in connecting the tiny gaps present in the specimen. In addition, the pozzolanic behaviour and filling effect shown by the fine particles of NS were also responsible for the increase in strength.

4.2. Splitting Tensile Strength. Figure 9 illustrates the 28-day average splitting tensile strength of concrete made with and without steel fibres, respectively. All the mixes incorporated

(a) (b)

Figure 6: (a, b) Specimens for sorptivity test.

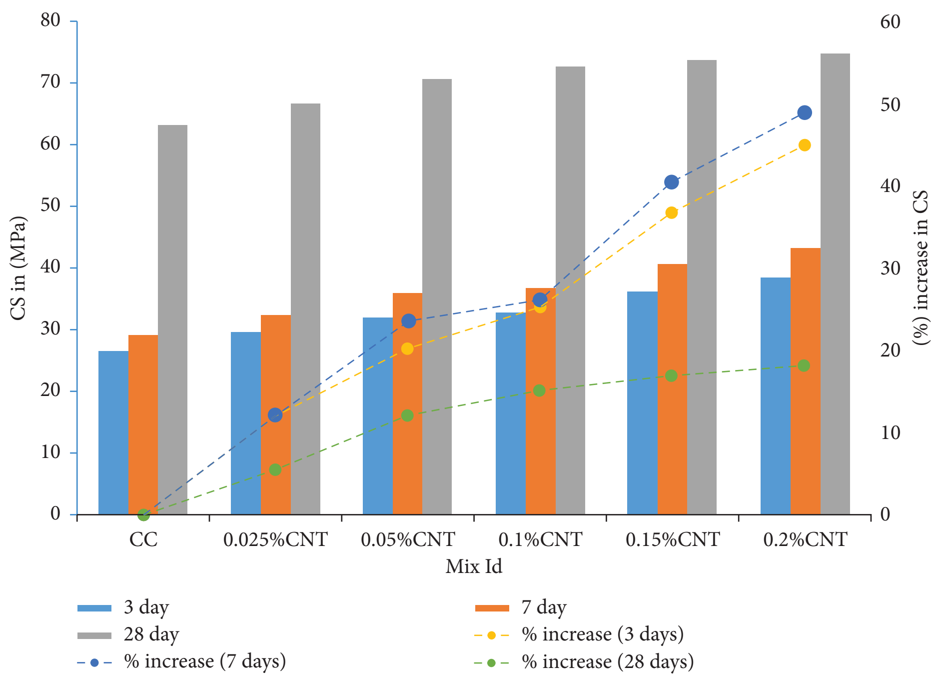


Figure 7: CS of specimens without steel fibre.

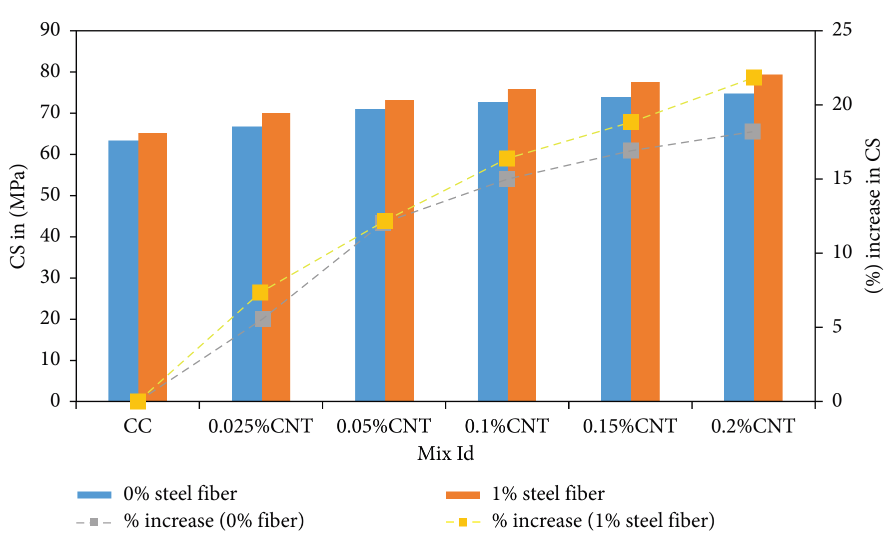


Figure 8: Comparison of compressive strength with and without fibre for 28 days.

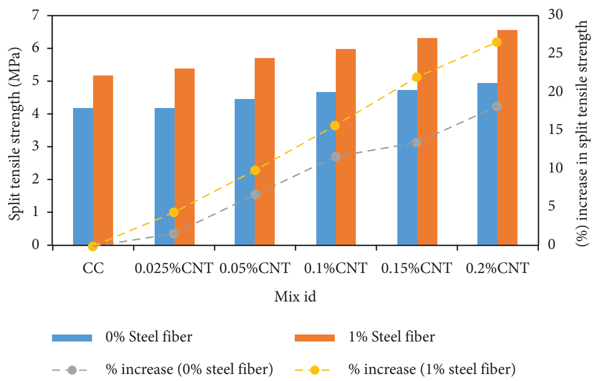


Figure 9: Splitting tensile strength for 28 days.

with nano silica and MWCNT possessed higher strength than the control irrespective of fiber incorporation. Their presence filled the micro cavities in the structure and accelerated the hydration process due to which additional C-S-H gels have developed. So, they possess dual role as fillers and accelerating agents which had helped in boosting up the tensile strength. The strength increase observed was 1.65%, 6.78%, 11.64%, 13.51%, and 18.2% compared to the control mix. Those incorporated with steel fibres had shown a massive increase than those without fibres, and it was found to be 4.45%, 9.86%, 15.76%, 22%, and 26.5% more than the control mix. Karthikeyan et al. [6] on using steel fibres in concrete along with $UFTiO_2$ had mentioned that steel fibres, by bridging the gaps developed and resisting crack propagations, had helped the specimen to sustain the load to a maximum limit by resisting crack propagation. In this research, the effect of MWCNT as a filler element helped the specimens to attain the higher tensile strength by filling the pores developed in the structure, bridging the gaps and thus reducing the continuity of gaps which had improved the tensile strength. Thus, the strength enhancement is attributed to the combined action of NS and CNT in acting as filler and activator with the support from the steel fibres.

4.3. Impact Strength Test. The impact strength of the control and mix with NS, MWCNT, and steel fibre was estimated after 28 days of normal water curing, and the findings are displayed in Figures 10–13. Figures 10 and 11 show the number of impacts required by the specimen for the development of first crack. It is observed from the figures that the mixes with 1% NS and 0.2% MWCNT both with and without steel fibres have taken more numbers of impact shots indicating that a longer time was required for them for the appearance of the first crack and for failure. The maximum number of hits required for first crack and final failure for mixes with 0.2% CNT + 0% steel fibre was 21 and 36, respectively, and for mixes with 0.2% CNT + 1% steel fibre, it was found to be 40 and 101, respectively. From the reports of Murali et al. [44], which discusses the impact resistance specimens made with 0.2%MWCNT incorporated with various other fibres such as steel, polypropylene, it is evident that addition of fibres, whether short-steel, long-steel, or polypropylene, when incorporated in normal mixes, would show a greater resistance to impact than the control mixes. In present work also it was observed that the fibre incorporated specimens exhibited a massive resistance to impact than the control mix and other mixes without steel fibres.

Figures 12 and 13 illustrate the impact energy of the specimens at their first crack and at failure state. Impact energy increases with the amount of CNT and the addition of steel fibre. It is well established that adding steel fibres to concrete mixtures significantly improves their impact resistance and fracture behaviour. Research works by Murali et al. [44, 45] state that the use of MECNT in 0.2% had increased the resistance to crack by 23% and failure resistance by 66%, and it was also mentioned that the action of MWCNT as discrete nanoscale reinforcement is the main cause for the delay in the crack initiation. This was found to be true for present case also in which it was observed that the maximum impact energy at first crack and at failure for the 0.2% MWCNT + 1% NS combination without steel fibres was 423.228 J and 725.533 J, respectively, and the percentage improvement in impact energy at first crack was 250% and 176.92%, respectively. For the mixes with 1% steel fibre incorporated, the same 0.2% MWCNT + 1% NS combination showed better improvement, and the impact energy at first crack and at failure was 806.148 J and 2035.524 J, respectively. The energy improvement was found to be more by 53.84% and 98.04%, respectively, than the control mix. There was a decrease in the percentage increase in energy at first and final failure for 0.2% CNT + 1% steel fibre compared to 0.2% CNT + 0% steel fibre due to improper bonding of nanomaterials and steel fibre. The materials might have agglomerated to produce such a decrease in the result. Figure 14 shows the failure pattern of different mixes. The number of cracks and crack width were greater in mixes with MWCNT and no steel fibres, whereas crack width was less in mixes with steel fibres, so it is obvious that due to the crack bridging potential of the steel fibres, the impact resistance had improved.

4.4. Sorptivity Test. The variation of sorption coefficients for control and MWCNT in different percentages with and without steel fibres is depicted in Figure 15. The adsorption rate (I) which was the ratio of amount of water adsorbed to the area of cross section of the specimen that was in contact with water was computed for all mixtures. Later, it was plotted with the square root of time to obtain the sorptivity values. The primary sorption was done for six hours, and the secondary sorption readings were noted after six days, and the process was continued for one week as specified in ASTM C1585-13; since much variation was not achieved in secondary sorption, it was not included in the results, and Figure 16 shows the primary sorption values only. The observed sorptivity values were inversely proportional to the strength values indicating that the mix which had permitted less water permeation possessed high strength. Compared with control mix, the mixes with NS and MWCNT combination showed decreased sorptivity values, and a much more decrease in values was observed for the same combinations with steel fibres incorporated. Carrico et al. [17]

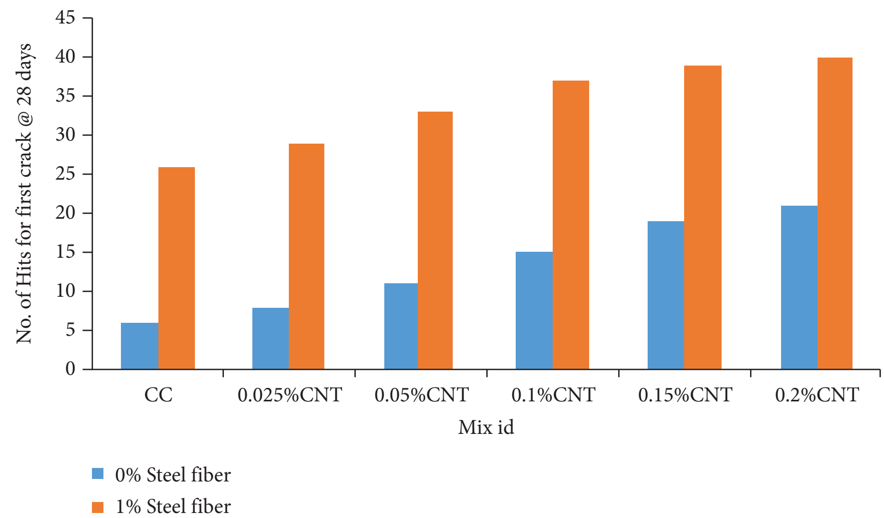


Figure 10: Number of impacts taken by specimens for first crack development.

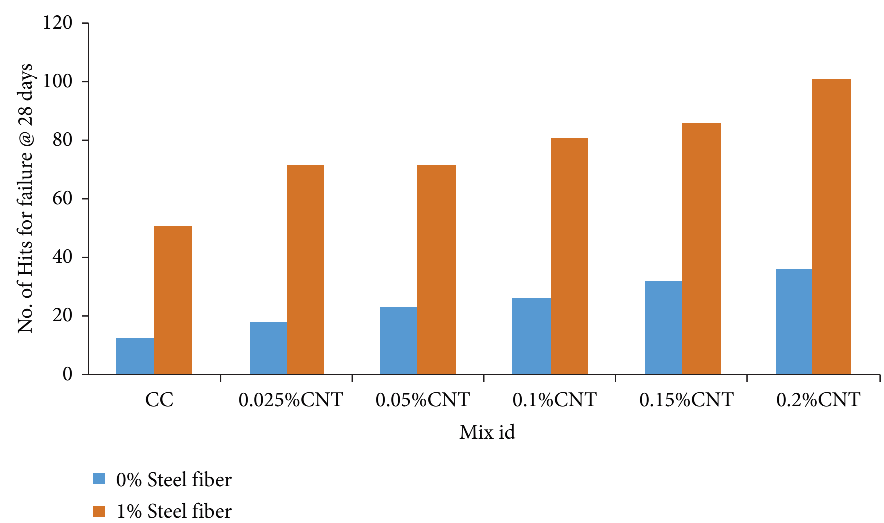


Figure 11: Number of hits for failure.

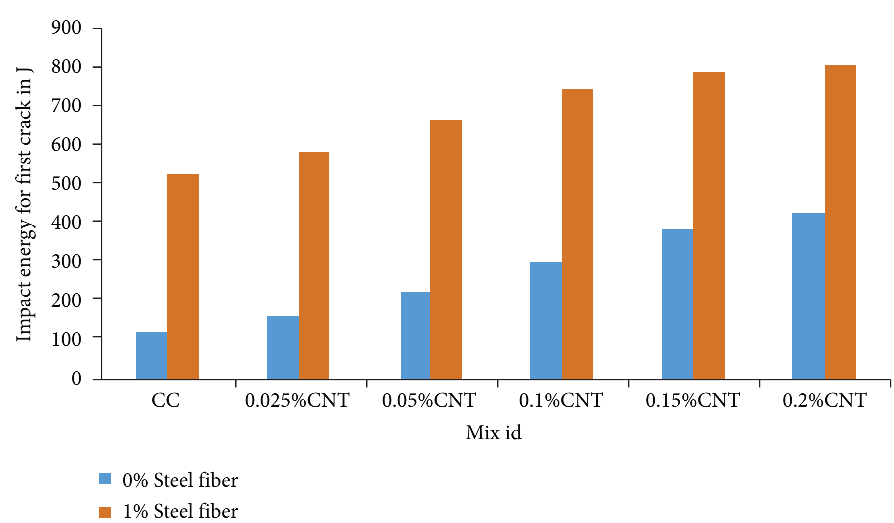


Figure 12: Impact energy for first crack at 28 days.

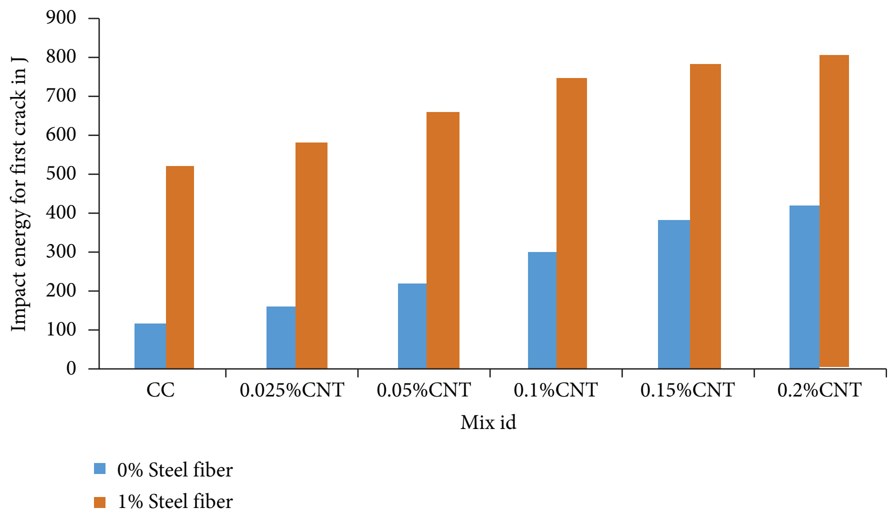


Figure 13: Impact energy for failure at 28 days.

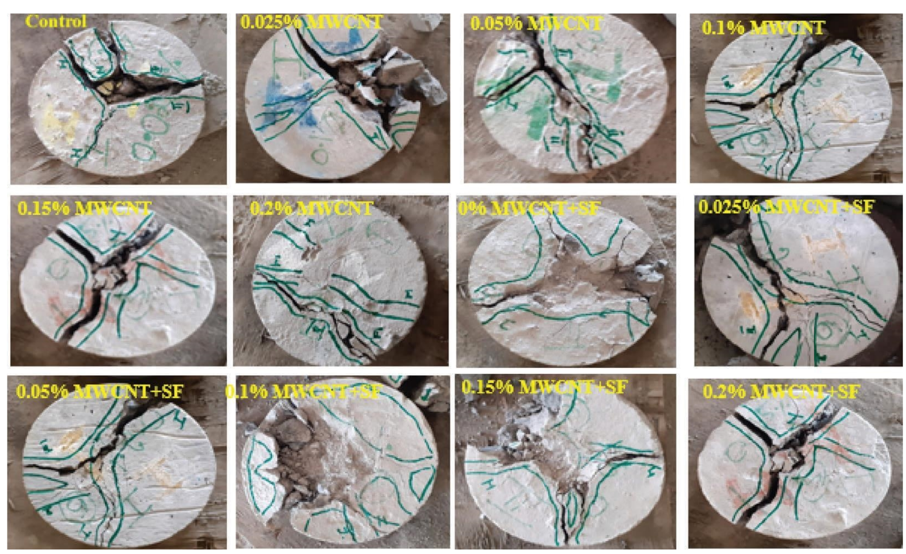


Figure 14: Crack pattern of the failed specimens.

in their results indicated that incorporation of CNT can resist capillary action by acting as a filling agent and by nucleation effects and mentioned that best performance can be obtained by using CNT in larger amounts, which was found to be true for present research as the values showed a decreasing trend as the addition of CNT was increased. Among all the mixes, a minimum sorptivity was found in specimens with 1% NS + 0.2% MWCNT with and without steel fibres. In addition to filling up of pores, the accelerated pozzolanic activity by nanosilica helping in forming C-S-H gel at earlier stages and bridging of the gaps developed by MWCNT and steel fibres has made the specimens attain a dense microstructure resisting water permeation. The presence of nanomaterials and steel fibres attributes to dense and compact microstructure with perfectly filled voids. Previous works done by the authors [62] using ultra-fine-sized silica fume and metakaolin had reported that use of finer-sized materials helped filling up the pores resulting in better resistance to water permeation. The results were analogous to those of Praseeda and Srinivasa Rao [49] where the authors stated that the incorporation of MWCNT in concrete helped the mix to attain decreased sorptivity values as the MWCNT induced accelerated pozzolanic activity and also made the microstructure denser.

4.5. Porosity Test. Figure 16 displays the obtained results of the specimens for the tests conducted to check the porosity. The results convey that the control concrete (CC) has absorbed more water than other mixes indicating that the presence of voids is more in CC. Mixes incorporated with 1% nanosilica and for various proportions of CNT have shown less percentage of voids, and among them, the mix with 0.2% CNT with and without steel fibre addition shows a lesser void ratio than the other mixes. It is because of the pore size refinement and pore filling capability of the NS and CNT nanoparticles blended with the mix. As already mentioned in the strength properties, here too NS and MWCNT

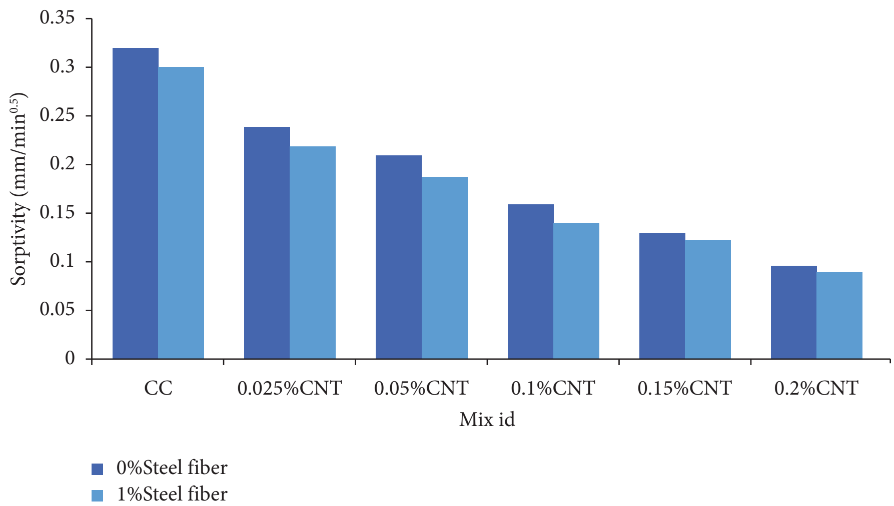


Figure 15: Sorptivity results (specimens subjected to primary sorption).

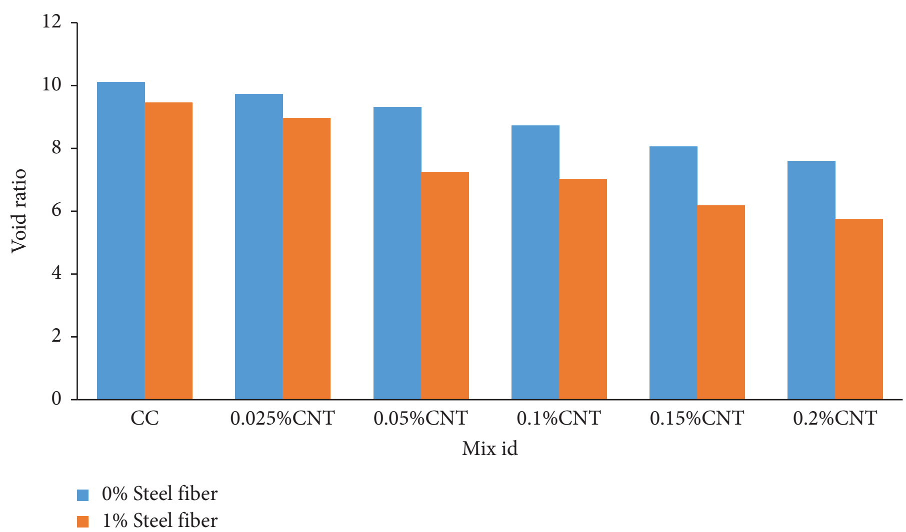


Figure 16: Void ratio for specimens with and without steel fibres.

particles engaged in the multiple actions such as filling the micropores, improving the density of the specimen, initiating the early hydration process, and helping to form earlier C-S-H all leading to less porosity. The observations were confirmed with those of the reports by Xu et al. [63] in which it was mentioned that the MWCNTs participate in refining the pores by filling the internal pores. The steel fibres played their part by reducing the propagation of cracks, thereby leading to void control. Comparing the specimens with steel and without steel, those with steel fibres added once again proved to be better than the others as presence of steel in addition to the nanoparticles had boosted the resisting capacities of the specimens. Among all mixes, it was noted that the porosity was lower in the mixture with 1% NS, 0.2% CNT, and 1% steel fibre. The dense microstructure of HSC and the enhanced interfacial region between the cementitious matrix and aggregates are credited with this. In fibre-reinforced concrete, the decreased capillary pore capacity can reduce capillary suction and the entry of aggressive solutions, improving the durability qualities [64], which hold good for the present work where the incorporation of steel fibre reduces the number of permeable spaces and disrupts the continuity of capillary pores.

4.6. Acid and Sulphate Attack Test. The results of mass loss of specimens exposed to aggressive HCl and H_2SO_4 acid environment are presented in Figure 17. Compared to the exposure environment, specimens exposed to H2SO4 suffered severe mass loss. Sulphur compounds play a significant role in deteriorating the concrete specimens as they form $CaSO_4$ and ettringite on reacting with C3A and both are potentially dangerous to concrete as they occupy a substantial volume than their parent compounds, and as a result, the concrete becomes weak, porous, and finally disrupts [7]. The results clearly indicate that the control specimens

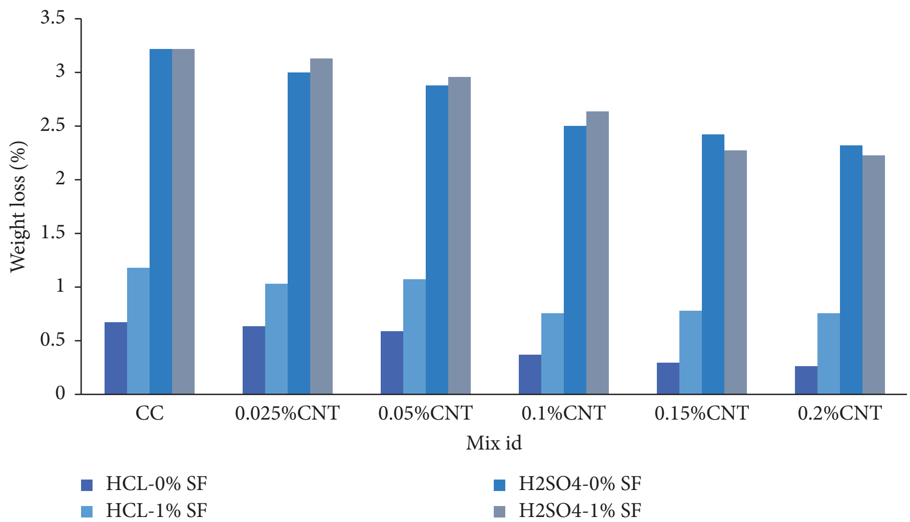
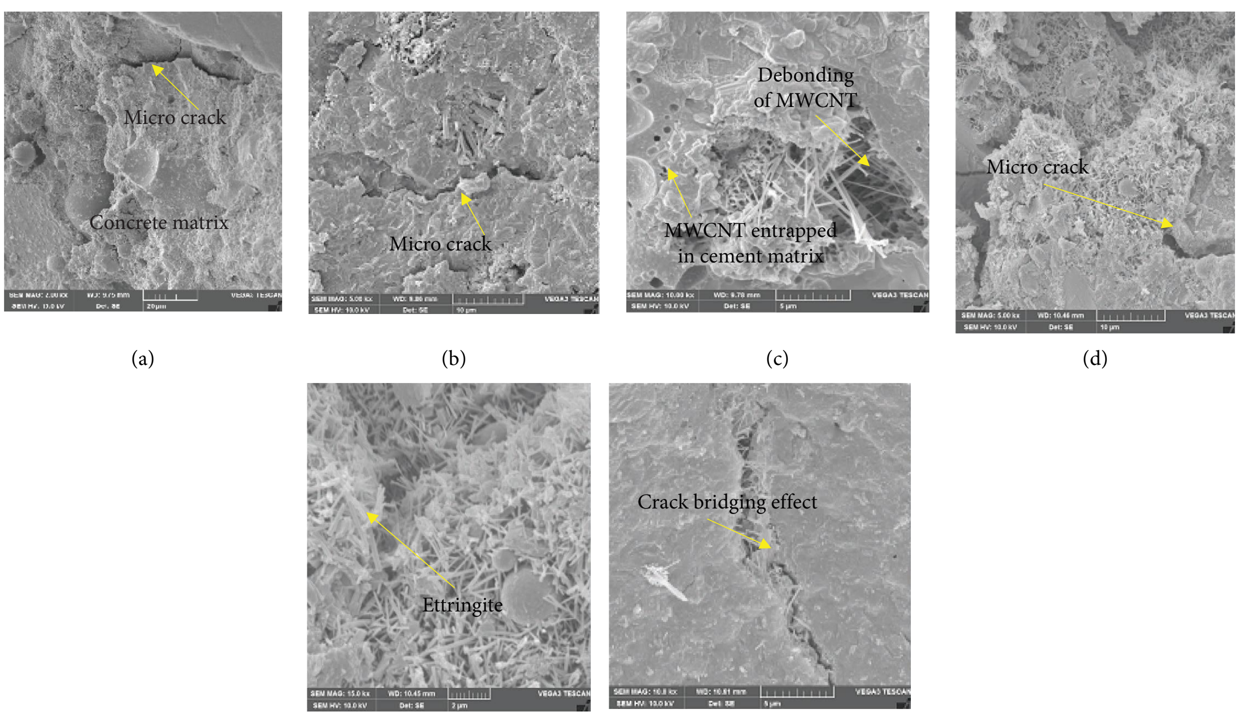


FIGURE 17: Weight loss % with steel fibres.



(a) (b) (c) (d) (e) (f)

FIGURE 18: (a) SEM images of control. (b) MWCNT without steel fibres. (c–f) MWCNT with steel fibres.

possess a severe mass loss than the other mixes made with NS and MWCNT combinations. Praseed and Srinivasa Rao [65] reported that mixes with 0.05% MWCNT along with other supplementary materials such as fly ash and silica fume have showed negligible mass loss due to the better pore filling activity by MWCNT and the active participation of other supplementary material to form additional required C-S-H gel. In present work, the mix with 1% NS + 0.2% MWCNT + 1% steel fibre outperformed other mixes as its mass loss is only 2.25% which is much lower than the other mixes. Since other supplementary materials were not used, it was possible to achieve better performance only with 0.2% CNT with additional support from 1% NS for the filling effect and pozzolanic activity and from the 1% steel in reducing the continuity of pores formed.

4.7. SEM Analysis. The failure plane of the specimens evaluated under compression served as the source for the sample collection for the SEM. Through SEM analysis, the MWCNT's interaction and dispersion properties with the cement matrix were evaluated. The figure demonstrates that different percentages of MWCNT were effectively diffused with the matrix and an adequate dispersion quality was attained for all the MWCNT containing test specimens in several areas without obvious clumping. The inclusion of MWCNT improves the hydration products and fibre adhesion, which increases the mechanical strength. MWCNT in the cracking region was able to be extended due to a strong enough contact with the cement matrix, which may operate as nano to microreinforcement [66]. The presence of MWCNT and NS reduced the number of pores as they filled the microcavities. They also play dual role by acting as a filler, increasing the density and engaging in enhancing the strength properties by initiating early hydration process and formation of extra CSH gel. Also, incorporation of steel fibres has bridged the microcracks and restricted further propagation of the cracks. Figure 18 illustrates the details explained above.

5. Conclusions

The conclusions arrived from the experimental investigations and comprehensive discussion are summarized and listed as follows:

(1) The compression and strength results exhibited a gradual increase in strength for both the mix categories, namely, with and without steel fibres.

(2) Among all the mixes, the combination with 0.2% MWCNT + 1% NS showed higher strength for both the categories, i.e., with and without steel fibres, and maximum increase by 21.78% was exhibited by the combination 0.2% MWCNT + 1% NS + 1% steel fibre.

(3) Impact strength test showed all fibrous mixes possessing better cracking impact resistance than mixes without fibres. Specimens without steel fibre failed suddenly after initial cracking leading to brittle failure. Specimens with steel fibres incorporated experienced multiple number of cracks there by delaying the sudden failure. In addition, they also improved the shock absorbing capability and the ductile behaviour of the specimens. 0.2% MWCNT + 1% NS + 1% steel fibre registered a highest energy absorbing capacity of 806.148 J and 2035.524 J, respectively, which was more by 53.84% and 98.04%, respectively, than the control mix.

(4) In durability tests, the mix with 1% NS + 0.2% MWCNT + 1% steel fibre performed better than other mixes with a very less mass loss by 2.25% than the control mix and with less sorption values and percentage of voids than the other mixes.

(5) From the microstructure results, it is clear that the details obtained from experimental results are true as the gaps are bridged by the MWCNT and steel fibre making the mix denser with less voids.

(6) Finally, the mix with 1% NS + 0.2% MWCNT + 1% steel fibre outperformed all other mixes with higher strength, better durability characteristics, and high resistance to impacts.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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