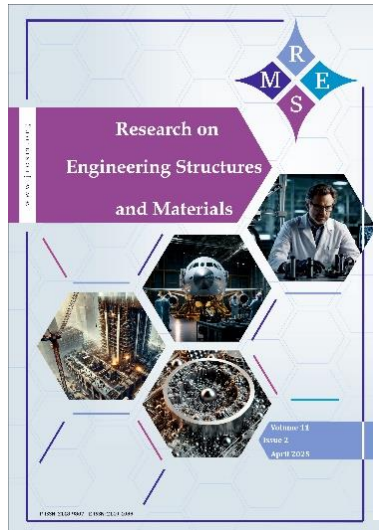




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Effect of Nano-TiO₂ at macro and micro level of concrete by partial substitution of cement

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Article Info	Abstract
<p>Article History:</p> <p>Received 17 Aug 20204</p> <p>Accepted 04 Oct 2024</p> <p>Keywords:</p> <p>Nano-TiO₂; Workability; Strength; Variations; Macro level; Micro level</p>	<p>At macro level the strength of the concrete will decline due to several reasons like high water-cement ratio, improper compaction and improper curing. At micro level concrete strength will reduce due to poor pore structure. Since the cement acts a binding agent upon reacting with water, the fineness of the cement influences the strength. Higher water cement ratio leads to higher porosity. Fine particles of very high specific surface area may affect the pores of concrete leading to strength enhancement rather than decline. In this study is aimed to study the effects of partial substitution of nano-TiO₂ for cement into concrete, on mechanical strength and microstructure. Fresh and hard macro level properties are investigated for M40 and M60 with cement replacement 0% to 2% by nano-TiO₂. SEM images were examined for studying dispersion of particles and XRD for products of hydration at micro level. It is found that at 1% of nano-TiO₂ strength for M40 and M60 were higher with other percentages replacements.</p>

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1. Introduction

Most highly used man-made material is concrete heterogeneous rigid at macro and porous at micro with varied sizes of pores effecting mechanical properties significantly. Many key attributes, including strength and permeability, are directly or indirectly related to the characteristics of these pores [1,2]. It is studied that mechanical and durability properties are highly affected by pore structure of concrete [3]. Using very fine particles could fill voids or pores of concrete, the mechanical properties would improve. Addition of nano particles of higher surface energy into cement concrete, acts as nucleus and grow to form conglomeration for hydrated products of cement. Dispersion of the nano particles leads to form strong microstructure by the conglomeration, when quantity of nano particles is small for higher dispersion [4]. Some nano fillers can react with hydration products in cementitious materials, acting as nano-cores. This enables unique structures around the fillers that differ from those in traditional cement. These distinct areas significantly influence the overall behavior of the cementitious composites. [5].

Cement mortars with nano-FeO₃ and nano-SiO₂ had reduced Ca(OH)₂ and pores being filled up when compared with plain cement mortars. Furthermore, nano particles strengthened concrete at paste phase and paste and aggregates phase [4]. The mechanical strengths in compression and flexural of concrete with nano-TiO₂ are higher than concrete with nano SiO₂, which is mainly dispersion of particles having lower specific surface area of nano-TiO₂(240m²/g) than nano-SiO₂(640m²/g) [6].

Nano clays, Nano carbon tubes, mineral oxides like alumina, silica, metallic oxides like iron, titanium, zinc, zirconium etc are the various nano materials used in building materials. Presently

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usage of nano-TiO₂ has gained lot of interest as construction materials because of low in cost, high chemical activity and stable [7-9]. The workability of concrete is affected when nano particles are added in high quantity due to their larger specific surface area [10]. The raise in water quantity is noticed for concrete mixtures due to lower size and larger surface area in absorbing water in high [11, 12].

Incorporation of nano-TiO₂ to concrete was carried out first from the available research was by Li in Harbin Institute of Technology, China [13]. Nano-TiO₂ particles are non-pozzolanic materials which do not react, but they are refill the pore structures. Nano-TiO₂ accelerates hydration of cement, significant peak during heat of hydration takes place early with improved potential, reduced occurrence and lower setting time duration compared to the conventional specimen providing additional nucleation sites and raise in water demand. [14]. Nano-TiO₂ being fine in particle size the strength of concrete is enhanced by accelerating hydration and pore filling. Drying Shrinkage minimized with addition of nano-TiO₂ and hydrophilicity of paste is increased [15]. Addition of nano-TiO₂ modified the microstructural mechanisms filling pores by producing homogeneous dense needle shaped calcium silicate hydrated gel hydration products with sustained deposition on improving properties of concrete [16].

In the early stages of hydration, porosity increases due to the formation of voids. However, as hydration continues, the growth of hydrates fills these voids, leading to a subsequent reduction in porosity. Significant changes in porosity observed over time, reflecting the transition from initial expansion to eventual densification as the cement paste matures [17]. During the hydration crystallization of Ca(OH)₂ reduced at aggregate and cement paste interface enhancing the structure and mechanical properties. Increase in nano-TiO₂ quantity in concrete limits the space required for gel growth and reduces Ca(OH)₂ in cement matrix.[18,19]. The performance of concrete can be optimized by the nano-TiO₂ photocatalytic activity decomposition of gaseous pollutants [20]. The surface hardness of concrete that possess 1% nano-TiO₂ exhibited significant betterment than nano-silica and polypropylene. Hydrophilicity of nano-TiO₂ provides zero contact and reflectance in high level with 5% content in concrete mixtures [21]. Nanofillers migrate with water into interfacial zone of aggregates and cement mortar by the nano-core effect enhances bond strength. Beyond the substitution of 1% of nano fillers to cement in concrete the bond strength between aggregates and cement mortars turns to decline [22].

The suitability of nano-TiO₂ as cementitious materials is identified in XRD diffraction with smooth hump establishing amorphous phase. Rutile and anatase phases are noticed in the peaks of XRD diffraction. [23]. Nano-TiO₂ renders cementitious composites through its excellent properties and by optimizing hydration, which improves strength and reduces shrinkage. This allows for tailored performance, lower production costs, and longer-lasting infrastructure. Future advancements promise even more efficient and multifunctional applications in construction [24].

The quality of concrete mainly influenced by materials properties, preparation and workmanship. Due to various reasons the quality of concrete pertaining to strength is declined. The inclusion of nano-TiO₂ into concrete has been done extensively and observed considerable improvement in the strength of concrete. The longevity of concrete will also enhance on improved strength by partial substitution of nano-TiO₂. In addition to the enhancement of strength and longevity, the maintenance cost of concrete structures with nano TiO₂ can be less than conventional concrete.

In this paper, experimental study on concrete with partial substitution of cement by nano-TiO₂ varying partially with an interval of 0.5%, up to 2% for two grades M40 and M60, is presented. Two grades of concretes from Table no. 2 of IS 456, M40 and M60 were selected, and their mix designs are done following IS 10262:2009 guidelines. Workability and strength properties at macro and SEM and XRD analysis at micro level, with and without substitution of Nano Titanium Dioxide for both M40 and M60 grade concretes are investigated.

2. Materials

Nano TiO₂, ordinary Portland cement (OPC 53 grade), fine aggregate (river sand), coarse aggregate (natural crushed aggregates), water and super plasticizer are used for preparing of both M40 and M60 grade concretes to observe the effect of nano-TiO₂ by cement partial replacement.

2.1. Cement

In the present experimental investigation for lower pore size in cement paste phase OPC 53 grade Ordinary Portland cement is utilized throughout the work. In accordance with IS: 12269-1987 cement procured is tested for the basic physical properties and are shown in Table 1.

Table 1. Basic physical properties of OPC 53

Property	Standard Consistency	Setting time		Specific gravity	Compressive strength at 28 days
		Initial	Final		
Test results	32%	130 minutes	270 minutes	3..15	57.30 N/mm ²

2.2 Nano-TiO₂

Nano-TiO₂ is purchased at a cost of INR 375/- per kilogram from local paint shop. In general, it is mixed in paints as a pigment. From the literature is found that particles ranging from 50 – 200nm and density about 3.9 g/cm³. Titanium hardness improves the strength and durability of cement concrete.

2.3 Fine Aggregate

Godavari river sand clear from clay, slit and organic impurities is procured from local supplier. The laboratory tested physical properties are shown in the Table 2.

Table 2. Laboratory tested physical properties of fine aggregate

Property	Specific gravity	Unit weight (kg/m ³)		Fineness Modulus	Zone
Test results	2.58	1620 (loose)	1750 (dense)	2.74	II

2.4 Coarse Aggregate

Maximum size of 20mm with 67% and 10 mm with 33% of total coarse aggregate mass are used throughout the research work. Test results for physical properties are tabulated in Table 3.

Table 3: Laboratory tested physical properties of coarse aggregate

Property	Specific gravity	Unit weight (kg/m ³)	Fineness Modulus
Test results	2.72	1400 (loose)	2.74

2.5 Water

Potable fresh water available in the laboratory, which was clean without any organic substances is used in preparing the wet mix of concrete.

2.6 Super Plasticizer (Fosroc Conplast SP430)

Concrete with lower water-cement ratio yields poor workability like low slump and low compaction factor values. But, to achieve high strength water-cement ratio has kept low for all the variations. So, for the same water-cement ratio using chemical admixtures like Conplast SP430 is

used better slump and compaction factor values have yielded. Since the superplasticizer releases water-logged between cement grains during mixing, the same water-cement ratio adopted to keep its effect similar.

3. Experimental Investigation

In the experimental study mix proportions were obtained as per IS 10262: 2009 mix guidelines for both M40 and M60 grade concrete having water-cement ratios 0.40 and 0.38 respectively. Primarily trial concrete mixes without superplasticizer yielded low slump stiff concrete and low compaction factor values. Based on trials superplasticizer added 0.8% of mass of cement for better workability. Mix proportions for both M40 and M60 concrete grades are shown in Table 4.

Table 4. M40 and M60 grades mix proportions

Constituents	Cement	Fine aggregate	Coarse aggregate	w/c ratio
For M40	1	1.825	3.420	0.40
For M60	1	1.516	2.966	0.38

The effect of nano-TiO₂ on concrete workability and strength were studied experimentally by partial substitution of cement with nano-TiO₂ content varying from 0% to 2% for both grades of concretes. Test methods executed on concretes in fresh state as per IS codes, listed in Table 5 for assessing workability. After casting the specimens for each mix of each grade, left for a period of 24 hours and then extracted out of moulds to place in curing tanks. Specimens were tested for carrying strength developed after the curing periods of 3, 7 and 28 days. Test methods performed on concretes in fresh and hardened state are listed in Table 5 for assessing compressive strength, split tensile strength and flexural strength.

Table 5. Test methods for assessing properties of concrete

Property	Test Method	Specimen Shape	Specimen Size, mm	Guidelines
Slump	Slump cone test	-	-	IS 1199 : 1959
Compaction Factor	Compaction apparatus test	-	-	IS 1199 : 1959
Compressive strength	Compression test	Cube	100x100x100	IS 516 : 1959
Split tensile strength	Split tensile test	Cylinder	100x 200	IS 5816 : 1999
Flexural strength	Flexural test	Beams	500x100x100	IS 516 : 1959
Microstructure	SEM	powder	75µ Passing	Nishka Labs,HYD
Hydrate Products	XRD	Powder	75µ Passing	Nishka Labs,HYD

4. Results and Discussions

4.1 Workability Properties of Concretes M40 and M60

In the fresh state of concrete workability was assessed by performing tests slump cone and compaction factor apparatus, the test results were tabulated in Table 6. The influence of nano-TiO₂ on concrete strengths like compressive, split tensile and flexural are graphically represented with obtained values at 3, 7 and 28 days of curing for both grades M40 and M60 in chart 1 to 6.

Table 6. Test results for assessing workability

S. No	% TiO ₂	Slump (mm)		Compaction	Factor
		For M40	For M60	For M40	For M60
1	0	44	37	0.882	0.868
2	0.5	35	33	0.854	0.844
3	1.0	32	27	0.846	0.842
4	1.5	26	24	0.842	0.838
5	2.0	21	18	0.821	0.826

4.2 Strength Properties of Concretes M40 and M60

The influence of nano-TiO₂ on strength development is examined at macro level and micro level. Investigation results at macro level strength properties like compressive, split tensile and flexural strengths are presented from chart no1 to 3 for M40 grade concrete and from chart no. 4 to 6 for M60 grade concrete. Micro level study is presented by SEM and XRD analysis by figures 8, 9 and 10.

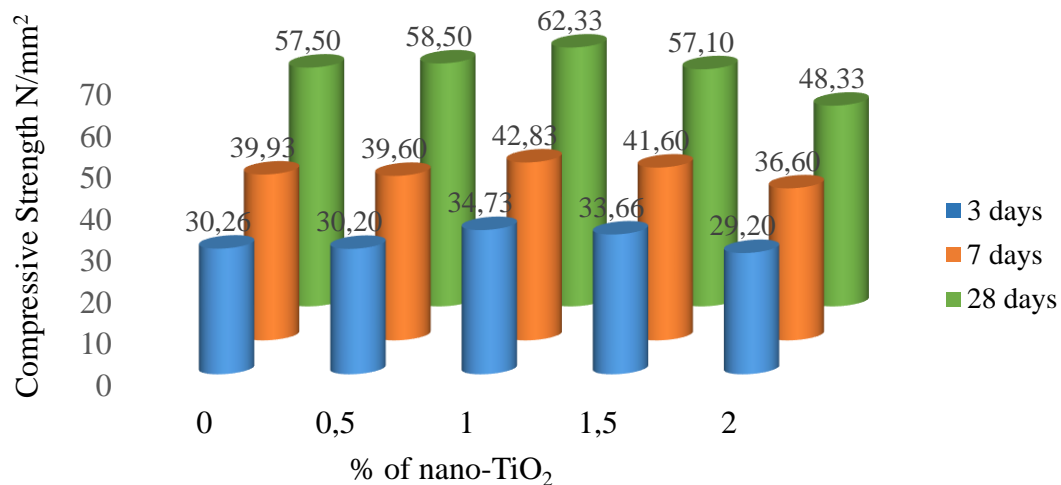


Fig. 1. Compressive strength for concrete M40 grade

From the Figure 1, it can be noticed that as days of curing increased 3 days to 28 days the strength is increased as in the case of conventional concrete. As the partial replacement increasing from 0.5 to 1.0% the compressive strength is increased and thereafter declined. It can be observed that nano-TiO₂ as 1% partial substitution for cement has exhibited higher strength than all other percentages of substitutions for all curing periods. The maximum compressive strengths obtained are 34.33N/mm² at 3days, 42.83N/mm² at 7 days and 62.33N/mm² at 1% of nano TiO₂.

From the Figure 2, it can be identified that increase in period of curing 3 days to 28 days there is increase in split tensile strength similarly as in the case of conventional concrete. It can be noticed that nano-TiO₂ as 1% partial substitution for cement has shown more split tensile strength than all other percentages of replacements for all curing periods. As the partial replacement increasing from 0.5 to 1.0% the split tensile strength is increased and thereafter decreased. The maximum split tensile strengths obtained are 2.29N/mm² at 3days, 3.29N/mm² at 7 days and 3.67N/mm² at 1% of nano-TiO₂.

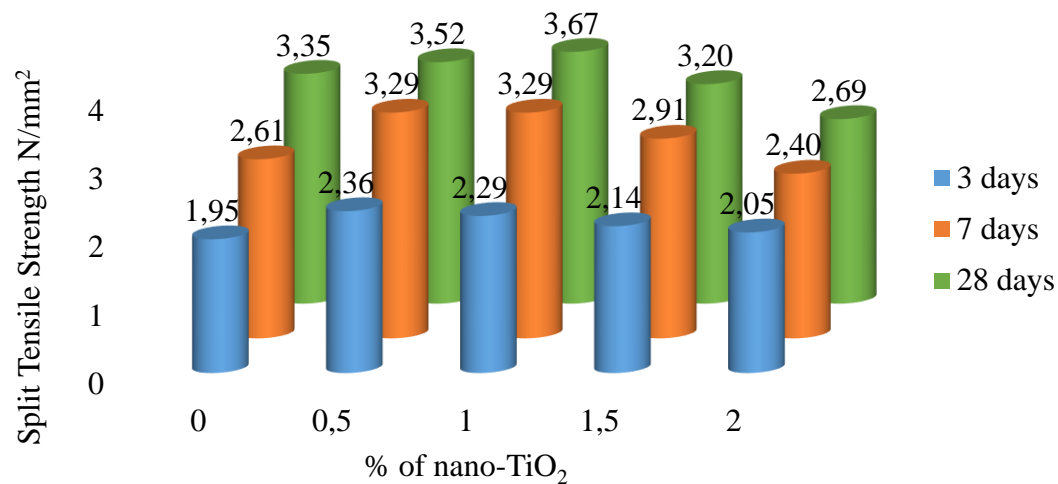


Fig. 2. Split tensile strength for concrete M40 grade

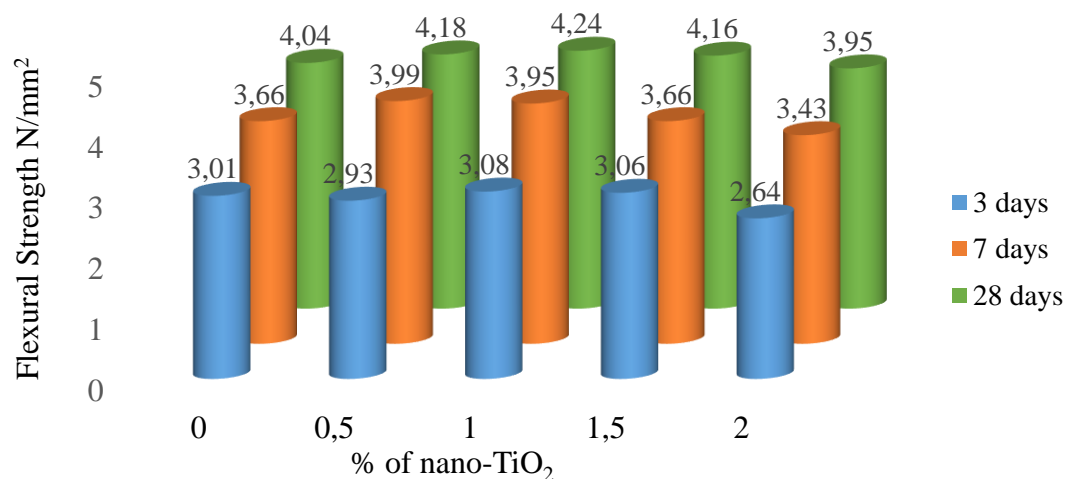


Fig. 3. Flexural strength for concrete M40 grade

From the Figure 3, it can be observed that period of curing 3 days to 28 days shows increase in flexural tensile strength similarly as in the case of conventional concrete. As the partial replacement increasing from 0.5 to 1.0% the flexural tensile strength is increased and thereafter declined. Also, it can be noticed that nano-TiO₂ as 1% partial substitution for cement has exhibited higher flexural strength than all other percentages of replacements for all curing periods. The maximum flexural strengths obtained are 3.08 N/mm² at 3 days, 3.95 N/mm² at 7 days and 4.24 N/mm² at 1% of nano-TiO₂.

From the Figure 4, it can be noticed that as days of curing increased 3 days to 28 days the compressive strength is also increased as in the case of conventional concrete. As the partial replacement increasing from 0.5 to 1.0% the compressive strength is increased and thereafter decreased. It can be observed that 1% of nano-TiO₂ as partial substitution for cement has exhibited maximum strength values than all other percentages of replacements for all curing periods. The maximum compressive strength values obtained are 46 N/mm² at 3 days, 62 N/mm² at 7 days and 74.66 N/mm² at 1% of nano-TiO₂.

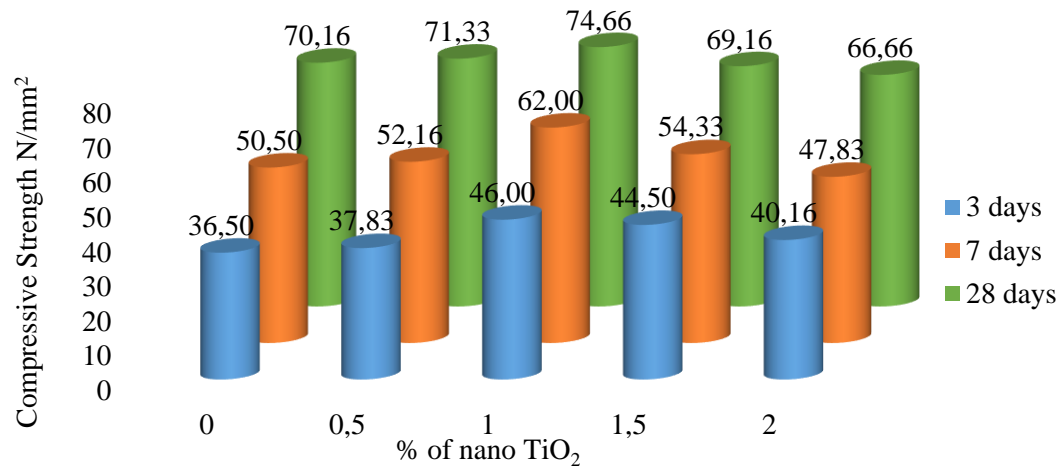


Fig. 4. Compressive strength for concrete M60 grade

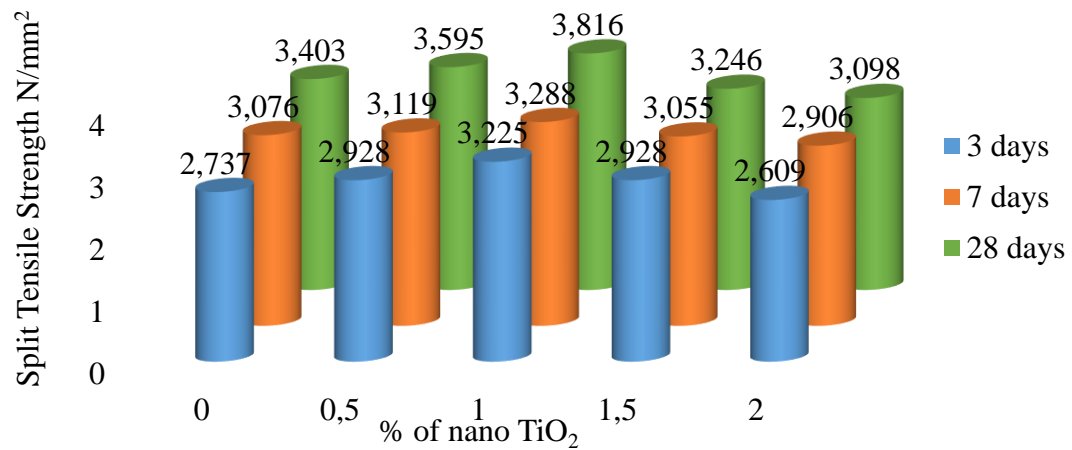


Fig. 5. Split tensile strength for concrete M60 grade

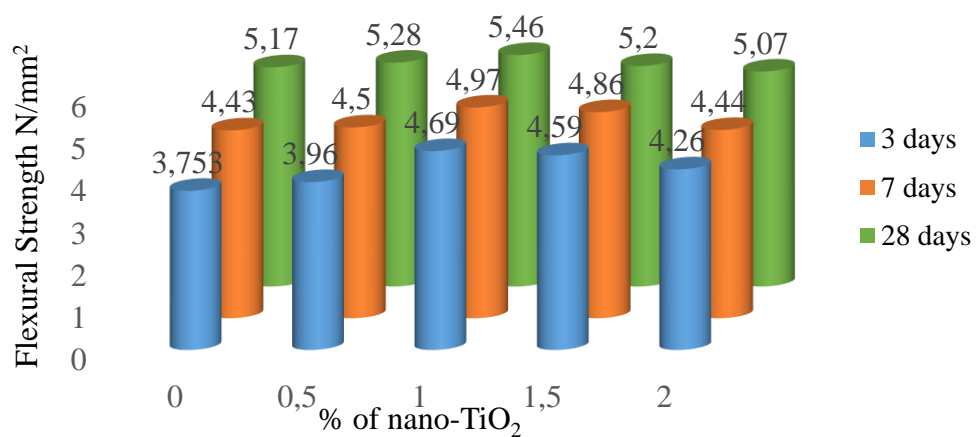


Fig. 6. Flexural strength for concrete M60 grade

From the Figure 5, it can be identified that increase in period of curing 3 days to 28 days there is increase in split tensile strength similarly as in the case of conventional concrete. It can be noticed that 1% of nano-TiO₂ as partial substitution for cement has shown higher split tensile strength than all other percentages of replacements for all curing periods. As the partial replacement increasing from 0.5 to 1.0% the split tensile strength is increased and thereafter decreased. The maximum split tensile strengths obtained are 3.225N/mm² at 3days, 3.288N/mm² at 7 days and 3.816N/mm² at 1% of nano-TiO₂.

From the Figure 6, it can be observed that period of curing 3 days to 28 days shows increase in flexural tensile strength similarly as in the case of conventional concrete. As the partial replacement increasing from 0.5 to 1.0% the flexural strength is increased and thereafter declined. Also, it can be noticed that nano-TiO₂ as 1% partial substitution for cement has exhibited higher split tensile strength than all other percentages of replacements for all curing periods. The maximum flexural strengths obtained are 4.69 N/mm² at 3 days, 4.97N/mm² at 7 days and 5.46N/mm² at 1% of nano-TiO₂.

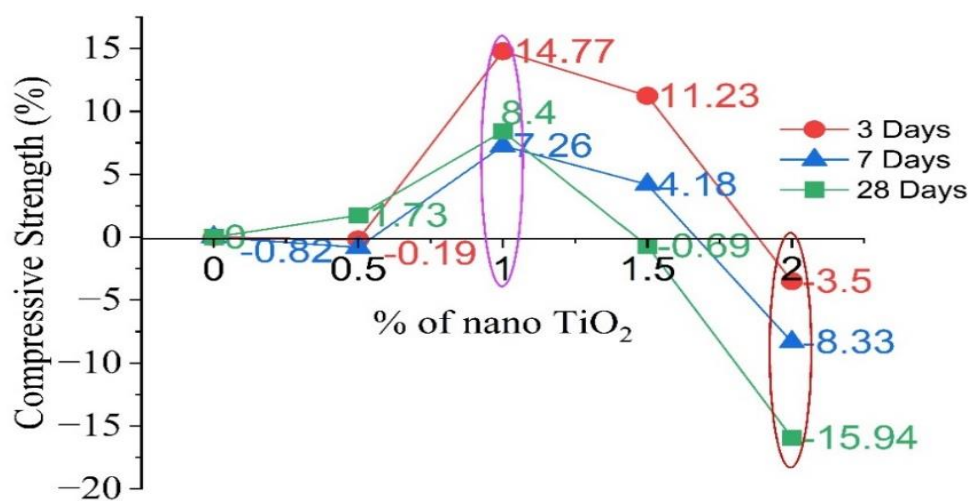


Figure 7: Compressive strength variation with % Nano-TiO₂ for M40 grade concrete

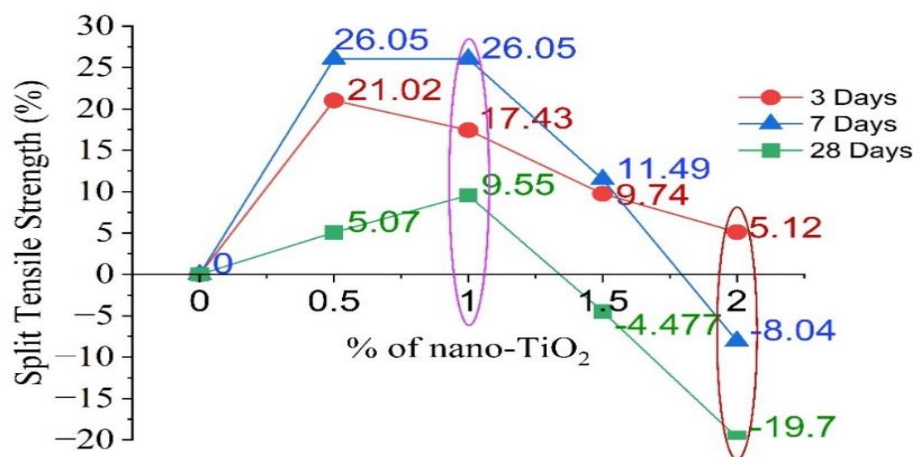


Fig. 8. Split tensile strength variation with % Nano TiO₂ for M40 grade concrete

From the Figure 7 with reference to compressive strength of conventional concrete, the variations with the increase in nano-TiO₂ content can be observed percentage wise. At 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. The strength at 1% are 7.26% 8.4% and 14.77% higher than

concrete of 0% nano-TiO₂. The decline of compressive strength at 2% can be clearly assessed from this chart.

From the Figure 8 with reference to conventional concrete split tensile strength, the variations with the increase in nano-TiO₂ content can be observed. At 0.5% and 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. From this chart the decrease in split tensile strength at 2% can be clearly observed.

From the Figure 9 with reference to conventional concrete flexural strength, the variations with the increase in nano-TiO₂ content can be observed. At 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. From this chart the decrease in flexural strength at 2% can be clearly observed.

From the Figure 10 with reference to compressive strength of conventional M60 grade concrete, the variations with the increase in nano-TiO₂ content can be observed percentage wise. At 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. The strength at 1% are 6.41% 22.77% and 26.02% higher than concrete with 0% nano-TiO₂. The decline of compressive strength at 2% can be clearly assessed from this chart.

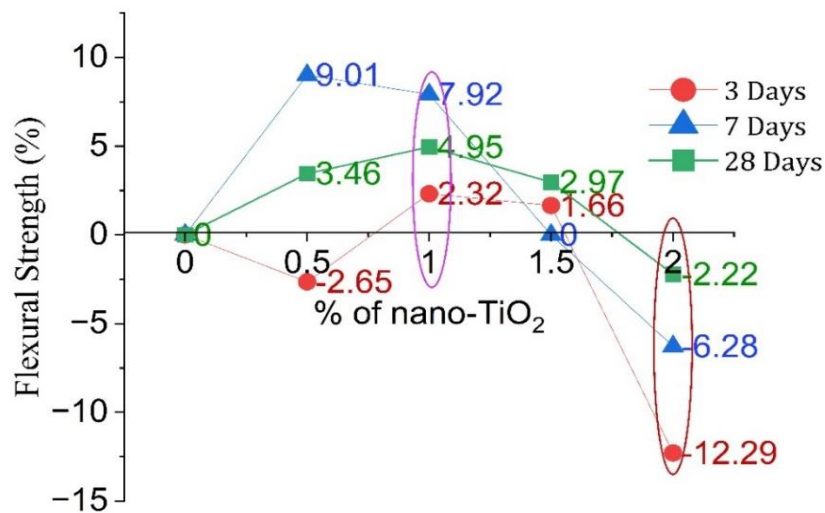


Fig. 9. Flexural strength variation with % Nano-TiO₂ for M40 grade concrete

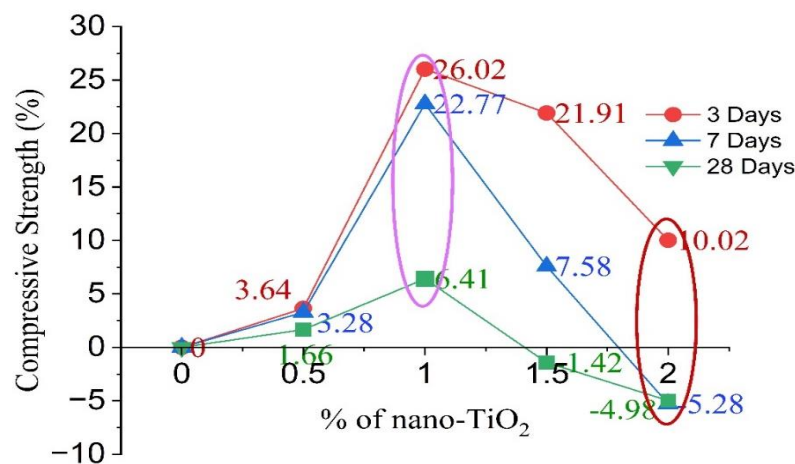


Fig. 10. Compressive strength variation with % Nano-TiO₂ for M60 grade concrete

From the Figure 11 with reference to M60 grade conventional concrete the split tensile strength, variations with the increase in nano-TiO₂ content can be observed. At 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. From this chart the decrease in split tensile strength at 2% can be clearly observed.

From the Figure 12 with reference to M60 grade conventional concrete flexural strength, the variations with the increase in nano-TiO₂ content can be observed. At 1% of nano-TiO₂ in all the curing periods strength obtained are higher comparing with other percentages of replacement, vice-versa at 2% of nano-TiO₂. From this chart the decrease in flexural strength at 2% can be clearly observed.

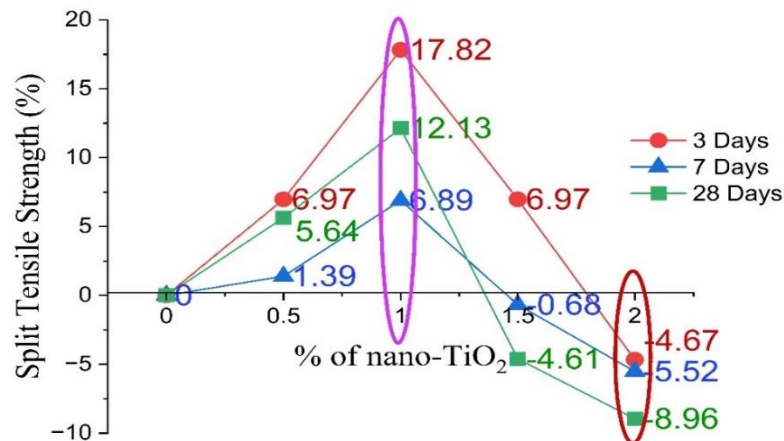


Figure 11: Split tensile strength variation with % Nano-TiO₂ for M60 grade concrete

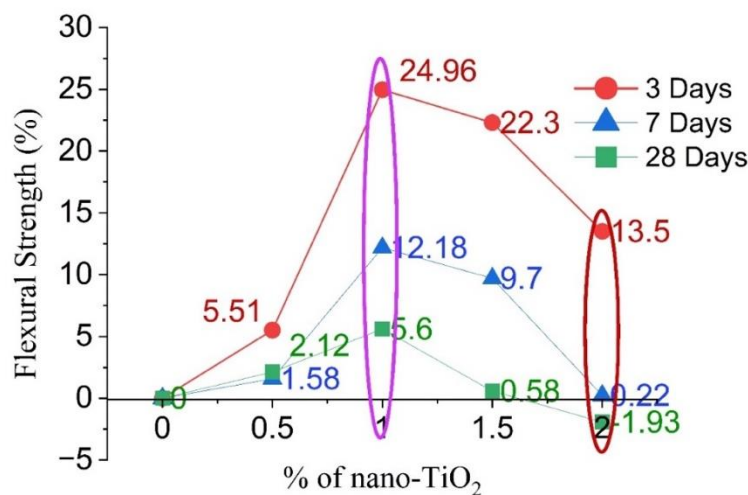


Fig.12. Flexural strength variation with % Nano-TiO₂ for M60 grade concrete

4.3 Micro-Structural Analysis Through SEM and XRD

In general, nanoparticles can provide better void filling and other favorable filler effects due to their extremely small particle size when compared to conventional concrete OPC cementing substances; the filler impacts produce an OPC concrete microstructure with enhanced density and reduced pore size [20]. An SEM test was performed on M40 and M60 grade concrete specimens with different nano-TiO₂ as 0%, 1%, 1.5%, and 2% by dry weight of cement to examine the effect of nano-TiO₂ on the microstructure of specimens.

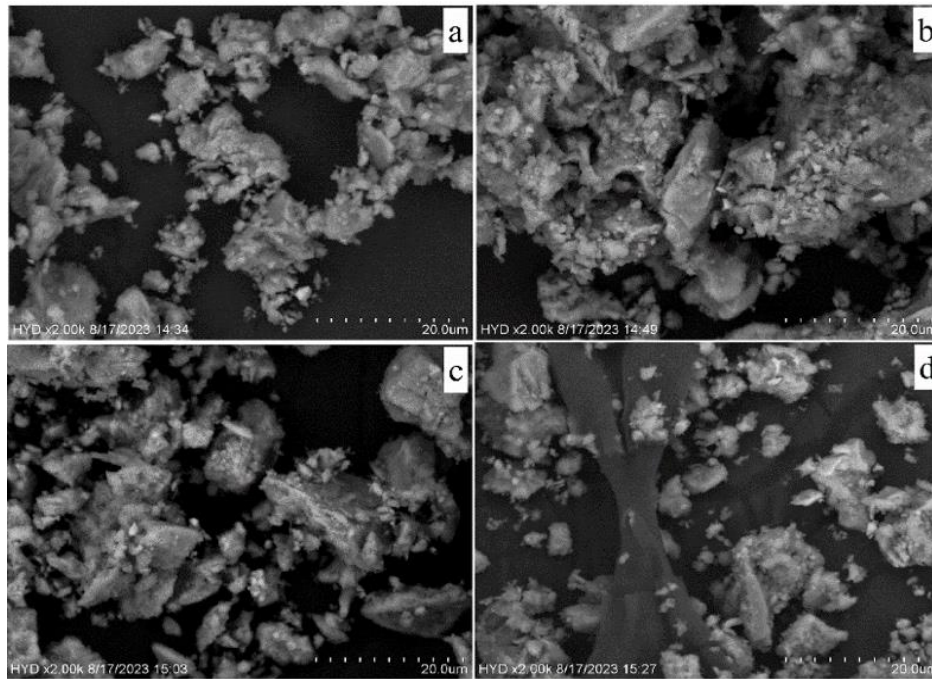


Fig. 13. SEM images of M40 concrete blended with nano-TiO₂

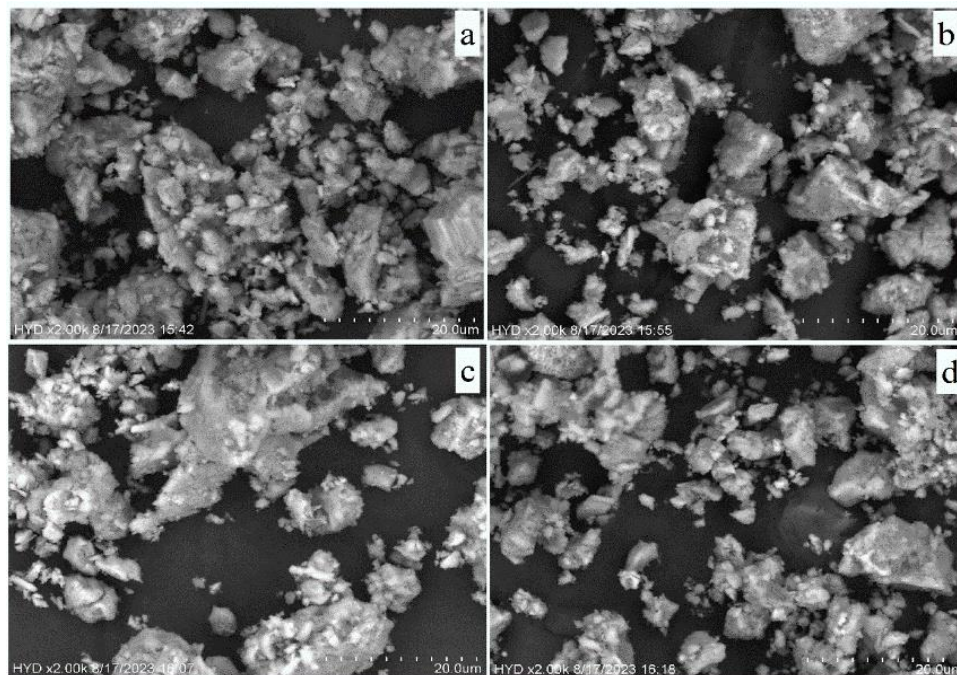


Fig.14. SEM images of M60 concrete blended with nano-TiO₂

Figures 13 and 14 depict M40 and M60 grade concrete results, respectively. Figure 14 depicts a nano-TiO₂ microstructure with more reacting particles and a denser structure. As a result, employing nano-TiO₂ reduced the size and quantity of unreacted particles. Some researchers found a similar good effect of nanoparticles on lowering voids ratio [20, 25]. Nano-TiO₂ indeed improves the microstructure by enhancing hydration and filling pores, leading to longer longevity strong concrete. The needle-shaped calcium silicate hydrate (C-S-H) that forms contribute to a denser matrix, which not only increases strength but also reduces drying shrinkage. This hydrophilic behavior aids in moisture retention and enhances overall performance. Overall, the incorporation of nano-TiO₂ is a promising way to optimize concrete properties.

Electron beam-induced contamination can significantly hinder nanoparticle measurements, making them either erroneous or completely impossible. The challenges posed by contamination can significantly affect the analysis of particle size and morphology. In scanning electron microscopy (SEM), the secondary electron (SE) images often suffer from contamination obstructing fine details, making particle identification difficult. This is especially problematic if the contamination is unevenly distributed or if it significantly alters the surface characteristics of the particles [26].

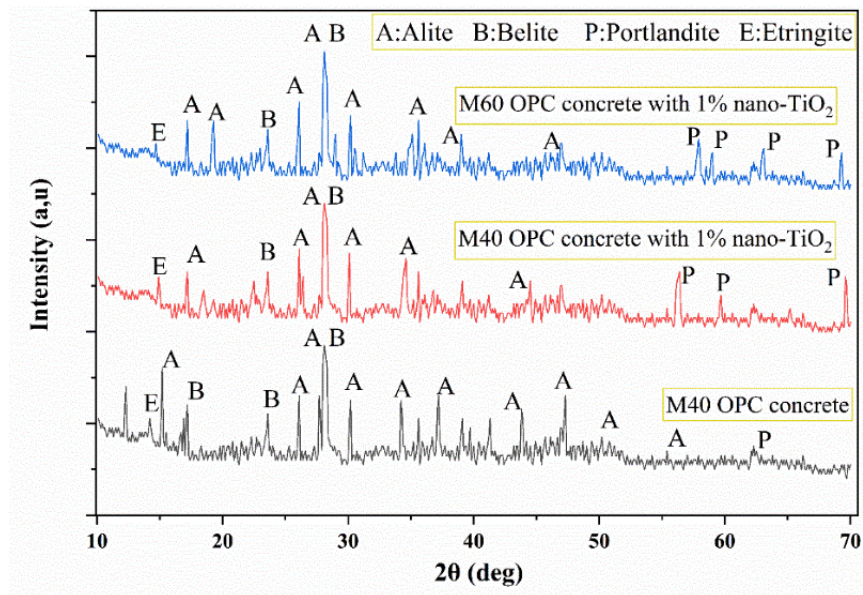


Fig.15. XRD traces of different concrete samples (a) M40 OPC concrete (b) M40 OPC concrete with 1.0% nano-TiO₂ (c) M60 OPC concrete with 1.0% nano-TiO₂

Figure 15 depicts an XRD examination of concrete with nano-TiO₂ after 28 days of curing to evaluate structural evolution. Major specimen phases have been identified as Ettringite, Portlandite, Alite, and Belite. Peak height changes and the creation of additional peaks were observed after 28 days. The intensity of the Alite and Belite phases diminished, and a new peak of portlandite was discovered. With the inclusion of nano-TiO₂, no additional new crystalline form was identified. The results showed that Ca(OH)₂ crystals (portlandite), which are required for the production of C-S-H gel, emerge in concrete containing nanoparticles but not in samples without nanoparticles, suggesting the synergistic effect of nanoparticles on the creation of subsequent C-S-H gel.

5. Conclusions

Workability declination in *both* concretes M40 and M60 with the increase in nano TiO₂. Slump and compaction factors values decreased with increase in nano-TiO₂ content *because* of its high specific surface area which increase water demand. Maximum compressive strengths for both M40 and M60 concretes are obtained at 1% nano TiO₂ at rate of 8.40% and 6.41% compared to conventional concrete. Using very fine particles reduces space required for CSH gel development thereby resulted in decrease in compressive strength after 1% nano-TiO₂ content. Similar trend is observed in split tensile and flexural strengths, maximum values are yielded at 1% nano-TiO₂ content with an increase of 9.55% and 12.41% than that of regular concrete for grades of concrete M40 and M60.

In general concrete is treated as brittle material, strong in compression and weak in tension, upon substitution of nano-TiO₂ the test results revealed, that spilt tensile strength of concrete much higher than concrete without nano-TiO₂. SEM images *show* the dispersion as the nano-TiO₂ content increase which resulted decrease in strength beyond 1% nano-TiO₂ content. XRD analysis provides evidence on formation of portlandite and Ca(OH)₂ at 1% of Nano-TiO₂ for achieving maximum strengths for both M40 and M60 concrete grades. Based on this experimental work results nano-TiO₂ of 1% can be concluded as preferable content at which maximum strength values are found.

Using concrete with nano-TiO₂ not only improves strength and longevity but also incorporates self-cleaning properties due to its photocatalytic nature. This means that structures can remain cleaner for longer periods, significantly reducing maintenance costs and effort.

In terms of sustainability, these factors contribute to a lower overall environmental impact. A longer-longevity structure needs lesser repairs and replacements, which in turn conserves resources and reduces waste. Additionally, the self-cleaning aspect can help maintain aesthetic qualities without the need for harsh chemicals, further supporting eco-friendly practices. Overall, integrating materials like nano-TiO₂ into concrete is a promising step toward more sustainable construction.

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