

Improvement of Concrete Mechanical Properties by Adding Nanomaterials

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Abstract: This study examined the impact of replacing the cement weight in concrete with nanomaterials (mix (C) 1% Carbon Nanotube CNT, mix (T) 2.5% Nano Titanium and mix (S) 3% Nano Silica) on the concrete mechanical properties through experimental investigations and comparison with normal concrete to show the extent of the effect of adding nanomaterials on the properties of concrete. The differences and increasing in the compressive strength for the mix (T) by comparison with the mix (N) and the mix (S) in the age of 7 days the increasing was 17% and 80% for the mix (S) and (T) respectively, In the age of 28 days the increasing was 44% and 60% with the mix (S) and (T) respectively and in the age of 56 days the increasing was 32% and 36% with the mix (S) and (T) respectively. The compressive strength for mix (C) was decreased 39%, 22%, 13% compared with the compressive strength for mix (N) at 7, 28 and 56days, respectively. The best improvements in compressive strength and splitting tensile strength for the mix that was added Nano Titanium (T) replacement by (2.5%) from weight of cement. For the mixes with nano material, a highest unit weight was observed at the age of 28 days. Referring to the results of SEM (Scanning electron microscope), the effect of nano Silica on the durability and mechanical properties of concrete can be explained by microstructure and from measuring X-ray diffraction (XRD) of concrete samples with the added Nano materials, these materials added to the concrete have clearly affected on the crystalline and chemical structure of the cement mortar components.

Keywords: CNT, XRD, Nano Silica, Nano Titanium, SEM

1. Introduction

Nanotechnology refers to the utilization of minuscule particles of matter, either in their isolated form or through their modification, with the aim of generating novel materials on a macroscopic scale. Nanotechnology pertains to the manipulation and study of particles in the nanoscale, specifically at dimensions of 10^{-9} meters. At the nanoscale, there exists a distinct contrast from the microscale, wherein the influence of gravity diminishes, giving way to the dominance of electrostatic forces and the emergence of quantum phenomena. As particles decrease in size to the nanoscale, the ratio of atoms located on the surface compared to those within the particle increases. This phenomenon, known as "nano-effects," plays a crucial role in determining the qualities shown by these particles at the macroscopic level. It is through the understanding and manipulation of these nano-effects that the potential of nanotechnology is realized. [1] [2].

Understanding concrete at this new level is opening up new possibilities for enhancing its strength, durability, and monitoring. Concrete is a micro-material heavily influenced by its Nano-properties. Atomic force microscopy (AFM), X-ray diffraction analysis (XRD), scanning electron microscopy (SEM), and focused ion beam (FIB) are just few of the tools used to analyses concrete at the Nano-level to better understand its structure. Common concrete contains silica (SiO_2) as a normal component. Nanoscale

research has led to improvements in concrete's mechanical qualities thanks, in part, to the discovery that Nano-silica can be used to increase particle packing in concrete. [3] [4].

Hanaa S. Hamadi et. al. [5] investigated the effects of used nano materials (Silica) on the properties of hard concrete by the experimental studies. Significant progress has been made in replacing cement with nano-silica the mechanical properties of concrete provide a large amount of CSH while reducing the amount of crystalline CH due to its high pozzolanic activity. The optimal content of NS is 2%, and the compressive strength is increased by 1, 1.5, 2% compared to samples without NS. It can be concluded that the compressive strength increase amount by increasing the percentage of NS.

Chenglong Zhuang and Yu Chen [6] This study investigated the impact of nano-SiO₂ on various parameters of cement, including setting time, droop, shrinkage, strength, and mechanical characteristics. Furthermore, this review incorporates scanning electron microscopy (SEM) examinations of the microstructure and X-ray diffraction (XRD) findings pertaining to the many hydration products. The improved setting time, slump, and shrinkage of nano-SiO₂ concrete can be attributed to its high activity and nucleation. The use of nano-SiO₂ in concrete has demonstrated a noteworthy influence, particularly with regards to augmenting its durability. It is important to acknowledge that the incorporation of nano-SiO₂ in concrete leads to only slight enhancements in its mechanical properties. The discussion concludes by presenting an explanation of the macro performance of concrete enhanced with nano-silica, based on its microstructure.

Avuthu Narender Reddy et. al. [7] Studied the on incorporating colloidal nano silica particles into the reference concrete mix. In the concrete mixture, fly ash was used to replace the cement at 10, 15, 20, and 25%. In addition, between one and two percent of nano silica was added to concrete to improve its freshness and hardness. The improved concrete's flexural strength, split tensile strength, and compressive strength were determined through a variety of tests. The results demonstrated that the addition of nano silica to concrete improves its mechanical properties, and the mix containing 25% fly ash and 1% nano silica had the highest strength characteristics when compared to all other mixes.

Mainak Ghosal and Arun Kr Chakraborty [8] have added with a small amount of nanoparticles, such as Nano-silica (nS) to common Portland cement (OPC) have upgraded both the present moment and medium-term strength of concrete mortar when included streamlined amounts concerning the heaviness of concrete and water added according to the ordinary standard consistency recipe yet their drawn-out strength stays. Akash Kumar and Gurpreet Singh. [9] seen as increasing the strength of concrete mortar by up to 3.5% Nano silica for a 0.35 water to percentage ratio. Nano silica particles with a size of 0.2 to 0.3 microns have been used to replace 1%, 2%, 3%, 4%, 5%, and 6% of the concrete's composition by weight. The setting time increment with growing Nano silica expansion was demonstrated by the results. Up to 5% of concrete's fresh and solidified qualities are improved by nano silica; after that, the strength starts to decline. Early use of nano silica increases strength more than later use.

Biman Mukherjee et. al. [10] looked into the Nano silica (NS) and nano vanadium (NV) were substituted for regular Portland cement (OPC) in various proportions, and the resulting mixtures were then hydrated for 3, 7, and 28 days. Test findings were confirmed by SEM methods, and compressive and split tensile strengths were calculated. In comparison to regular concrete, the acidity and alkalinity tests show less weight loss and compressive strength degradation when 1% NS and 0.1% NV are used to replace OPC by weight.

2. Experimental Work

The present study was investigated the development of concrete mechanical properties by adding Nano silica (S), Nano titanium (T) and carbon nanotube CNT (C) as a partial replacement of cement weight and study of Nano concrete through conventional tests as well as Scanning Electron Microscopy (SEM) and X-Ray Diffraction Analysis (XRD) tests, compared with normal concrete samples.

2.1. Methodology Of The Experimental Work

Four mixes were produced to study the variables presented in Table 1 that were expected to have considerable effects on the mechanical concrete properties.

The total binder dosage in all mixtures and the water to binder ratio were remained constant at 350 kg/m³ and 0.35, respectively. The partial replacement of Nano Silica was 3% by the total content of cement 10.5

kg/m³. The partial replacement of Nano Titanium was 2.5% by the total content of binder 8.75kg/m³. The quantity of Carbon Nanotube (CNT) added to concrete was 1% by the total content of binder 3.5kg/m³ Table 2 [11] [12] [13].

In order to achieve the desired compressive strength of 25 MPa at 28 days (for reference mix), the concrete mix was created based on the testing outcomes of employed concrete ingredients. The ACI Standard Practice ACI 211.1-22 was used to determine the blended amounts of concrete [14]. Before casting concrete specimens, a job mix was mixed, slump tested and cast. With a water-to-cement ratio of 0.35, the resulting mixing ratio is (cement: sand: gravel) (1:2.2:2.7) by weight. ASTM C143/C143M-12 was used to measure the slump, which was found to be roughly 140 mm [15]. Using a concrete mixer after mixing dry materials, gradually the quantity of water was added while the mixer is in motion. The concrete was mixed for three minutes, allowed to rest for two minutes, and then mixed again for three minutes and during the mixing process, a plastic cover was placed over the entrance of the concrete mixer to prevent volatilization of nanomaterials.

Table 1 Details of the Nano materials codes mixtures

Coding for Samples	% Nano materials replacement from cement weight in the concrete mix
T	2.5% Nano Titanium
S	3% Nano Silica
C	1% Carbon Nanotube (CNT)
N	Normal concrete

Table 2 Mix Proportion for a Cubic Meter Mixture

No.	MIX ID	Cement Kg	Nano Silica Kg	Nano Tio2 Kg	CNT Kg	Water L	SP L	Fine aggregate	Coarse aggregate
1	N	350	-	-	-	123	-	700	1400
2	S	339.5	10.5	-	-	123	3	700	1400
3	T	341.25	-	8.75	-	123	3	700	1400
4	C	346.5	-	-	3.5	123	4	700	1400

2.2. Materials

2.2.1 Cement

The used cement is ordinary cement, which conforms to ASTM Standard Specification of Ordinary Portland Cement– TYPE I (ASTM C150/C150M 2017) [16]. Tables 3-4 show the Physical properties and chemical analysis for the cement.

2.2.2 Fine Aggregate

The used river sand was from Al-Kanahash area in Nineveh Governorate-Iraq, natural sand with grading limits within the requirements of the Iraqi specification No.45/1984 [17] was used throughout in this work. Tables 5 show the fine aggregate sieve analysis.

2.2.3 Coarse Aggregate

Crushed gravel of nominal maximum size (5-14) mm from Al-Kanahash area in Nineveh Governorate-Iraq was used. Tables 6 show the grading of this aggregate, which conforms to the Iraqi specifications of IQS No.45-1984 [17].

2.2.4 Water

Potable water according to IQS 1703/2018 was used in the mixing and curing process of concrete.

Table 3 Physical properties for the cement

Physical test		Limits-Type I	Badosh cement results
Setting Time	Initial Time (Min)	60	125
	Final Time (Min)	600	300
	Blaine (m ² /kg)	160	300
	Expansion (%)	0.80 Max	0.04
Comp. Strength (MPa)	03 Days	9	11
	07 Days	14	18
	28 Days	—	29

Table 4 Chemical analysis for the cement

Chemical compositions	Limits	Badosh cement test results
Loss on Ignition (LOI) %	3.00 Max	2.35
Insoluble Residue (I.R) %	0.75 Max	0.72
Silicon Oxide (SiO ₂) %	20.00 Min	21
Aluminum Oxide (Al ₂ O ₃) %	6.00 Max	4.13
Iron Oxide (Fe ₂ O ₃) %	6.00 Max	3.85
Calcium Oxide (CaO) %	-----	61.87
Magnesium Oxide (MgO) %	6.00 Max	3.52
Sulphur Trioxide (SO ₃) %	3.00 Max	1.79
Alkali Eq.	0.60	0.53
C3S	-----	47.3
C2S	-----	27.0
C3A	8.00 Max	7.1
C4AF	-----	9.04
Chloride (Cl)	-----	0.01

Table 5 Grading of fine aggregate

Sieve size (mm)	Cumulative passing %	IQS No.45/ 1984
9.5	100	100
4.75	100	90 -100
2.36	83.5	75 -100
1.18	75.5	55 – 90
0.6	52.2	35 -59
0.3	20.8	8 -30
0.15	4.8	0 -10
Fineness modulus	2.63	

Table 6 Grading of coarse aggregate

Sieve size (mm)	Cumulative passing %	IQS No.45/1984 (5-14) mm
20	100	100
14	100	90-100

10	70	50-85
5	10	0-10
2.36	0	---

2.2.5 Nano Silica (SiO₂)

Powdered SiO₂ nanoparticles from LUOYANG ADVANCED MATERIAL CO., LTD Company imported from China was used throughout this study Tables 7 - 8 show its Physical and chemical properties. According to ASTM C1240-05 [18].

2.2.6 Nano Titanium (TiO₂)

Powdered TiO₂ nanoparticles from LUOYANG ADVANCED MATERIAL CO., LTD Company imported from China was depended in this research Tables 9 - 10 illustrate the Physical and chemical properties.

2.2.7 Carbon Nanotube (CNT)

A black color thin fiber with a low reflectance and matt surface appearance from LUOYANG ADVANCED MATERIAL CO., LTD Company imported from China was used throughout this study Tables 11 – 12 exhibit the Physical and chemical properties.

2.2.8 Superplasticizer Chemical admixture

A superplasticizer used as high range water reducing admixture. It is known commercially, Sika ViscoCrete-5930 L one of Sika products. The technical description of Sika Visco Crete is presented in Table 13. According to ASTM C494-15 [19], this superplasticizer is classified as type F.

Table 7 Physical Properties of Nano SiO₂*

Property	Results	ASTM C1240-05
Physical form	Powder	
Color	White	
Diameter size (nm)	30 nm	
Moisture 2 hours at 105 °C	≥ 1.7%	3.0% (max)
Specific surface area, m ² /g	150-200 m ² /g	

*According to the manufacturer data

Table 8 Chemical Analysis of Nano SiO₂ *

Property	Result	ASTM C1240-05
SiO ₂ content based on ignited material wt. %	≥ 99.81%	85.0% (min)
Relative density	2.4	
Melting point/freezing point	1610 °C	
Initial boiling point and boiling range	2230 °C	

*According to the manufacturer data

Table 9 Physical Properties of Nano TiO₂ *

Property	Results
Physical form	Powder
Color	White
Diameter size (nm)	20~50 nm

Loss on dry, 105°C, 2H	0.30%
Specific surface area, m ² /g	60-100 m ² /g

*According to the manufacturer data

Table 10 Nano TiO₂ Chemical Analysis*

Property	Result
Purity	≥ 99.8%
Molecular Weight	79.87
Crystal structure	Anatase
Sulphated assay	3.50%

*According to the manufacturer data

Table 11 Physical Properties of Carbon Nanotube CNT *

Property	Results
Physical form	Thin Fiber
Color	Black
Outside Diameter (nm)	5-15 nm
Length	10-30 microns
Specific surface area, m ² /g	220-300 m ² /g

*According to the manufacturer data

Table 12 Carbon Nanotube CNT Chemical Analysis *

Property	Results
Ash	< 3%
Tap Density	0.094 g/cm ³
Molecular weight	12.01 g/mol
Relative density	0.011 - 0.27 g/cm ³ at 25 °C
Specific surface area, m ² /g	220-300 m ² /g

*According to the manufacturer data

Table 13 The Sika ViscoCrete-5930 L Technical description*

Properties	Technical description
Color	Brown
Appearance	Liquid
Specific of gravity	1.085 ± (0.01)
PH-value	4 - 6
Recommended dosage	(0.8-1.8) % by weight of binder
Compatibility with cement	All types of cement
Chloride ion content	nil
Shelf life	Up to 12 months

*According to the manufacturer data



Fig. 1 (A) Carbon nanotube (B) Nano Titanium (C) Nano silica. (D) Nanomaterials in containers

3.RESULTS AND DESCUSSION

3.1. Compressive strength

Compression strength measurements were performed on the reference cylinders to ASTM 39 [20]. Table 12 displays the results of the compressive strength at 7, 28, and 56 days.

For normal mixture or control concrete (N) the experimental results of compressive strengths at 7, 28 and 56 days was 11.49, 21.75 and 27.3 MPa, respectively. As the compressive strength increased with time, which is normal state for the concrete. While for mixture with added nano silica (S) replacement by (3%) from weight of cement, the experimental results of compressive strengths at 7, 28 and 56 days was 18.34, 23.90 and 28.10 MPa, respectively.

This indicate that when adding the Nano silica to the concrete the compressive was increasing by about 50% at 7 days, 10% at 28 days and at 56 days just increased by 2%.

The mixture with Nano Titanium (T) replacement by (2.5%) from weight of cement the compressive strengths at 7, 28 and 56 days was 21.60, 34.65 and 37.30 MPa, respectively.

When the Carbon nanotube (C) replacement by (1%) from weight of cement as shown in Figures 2-3 and table 12 the compressive strengths at 7, 28 and 56 days was 8.55, 17.8 and 24 MPa, respectively.

It was found that the compressive strength for mix (C) was decreased 39%, 22%, 13% compared with the compressive strength for mix (N) at 7, 28 and 56 days, respectively.

By studying the results of the compressive strength, the best improvements in compressive strength was for the mix with added Nano Titanium (T) replacement by (2.5%) from weight of cement, this can be explained by the effect of nano titanium on the microstructure of concrete, which led to an increase in compressive strength. As shown in Figs 3-4.

A similar observation has been noted by Sumit Sharma et al. [21] the compressive strength is improved when cement is replaced with (1.5%) nano TiO_2 .

Jay Sorathiya et al. [22] found that the Maximum increase in compressive strength of concrete with 1% used TiO_2 , both at 7 and 28 days.



Fig. 2 Cylinders test under compression machine and after crush mode failure for some samples

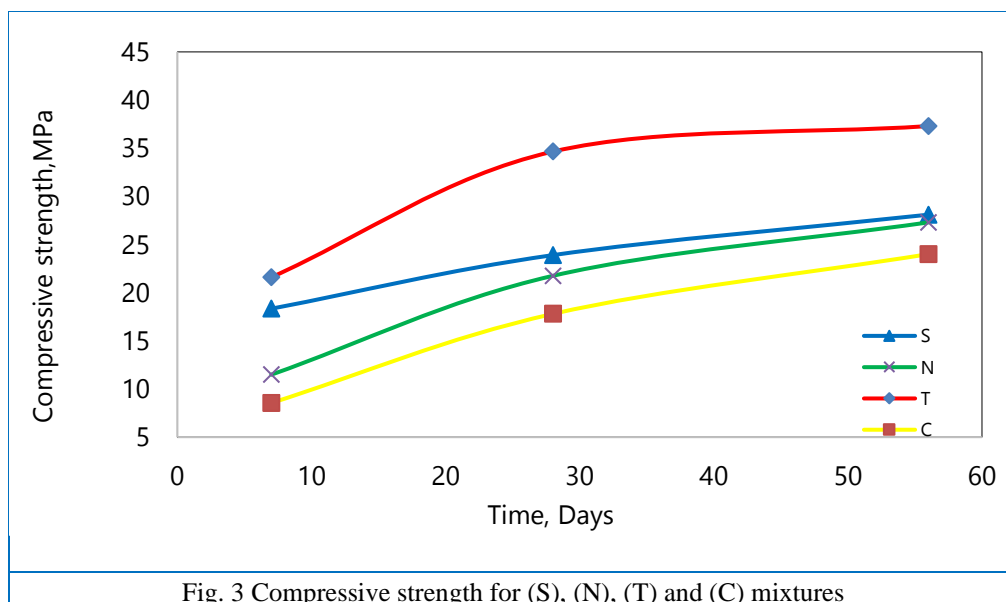


Fig. 3 Compressive strength for (S), (N), (T) and (C) mixtures

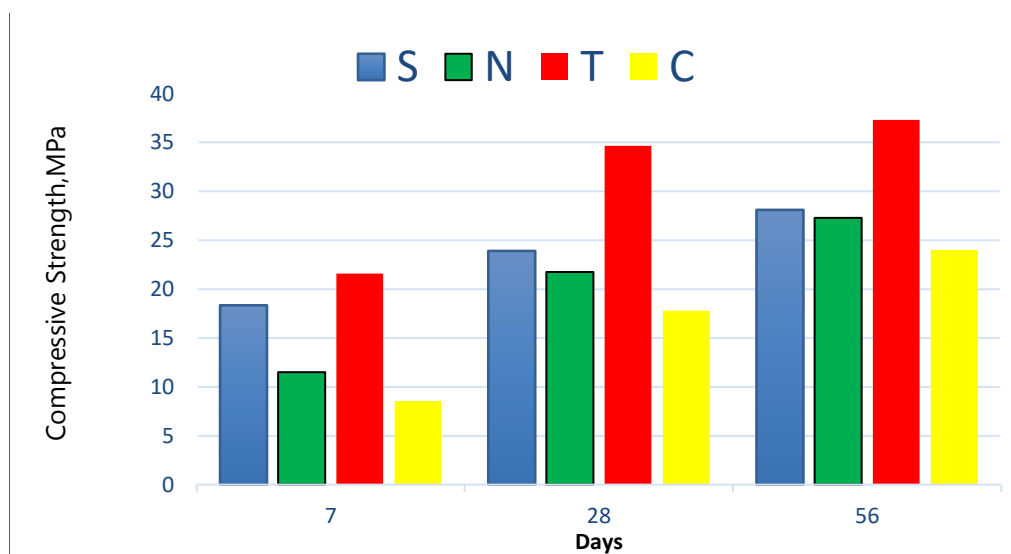


Fig. 4 Compressive strength for (S), (N), (T) and (C) mixtures

No.	MIX ID	f'_c (MPa) at 7 days	f'_c (MPa) at 28 days	f'_c (MPa) at 56 days
1	N (Normal concrete)	11.9	21.7	27.3
2	S (3% Nano Silica)	18.3	23.9	28.1
3	T (2.5% Nano Titanium)	21.6	34.6	37.3
4	C (1% Carbon Nanotube)	8.5	17.8	24

3.2. Splitting strength

The standard cylinders were subjected to splitting strength experiments in accordance with ASTM C496 [23]. Table 13 and Fig. 5 display the results of the splitting strength at 28 days of age. It can be observed that, the best improvements in splitting tensile strength for the mixture that was added Nano Titanium (T) replacement by (2.5%) from weight of cement.

No.	MIX ID	Splitting Strength (MPa) at 28days
1	N (Normal concrete)	1.83
2	S (3% Nano Silica)	2.34
3	T (2.5% Nano Titanium)	2.61
4	C (1% Carbon Nanotube)	2.23

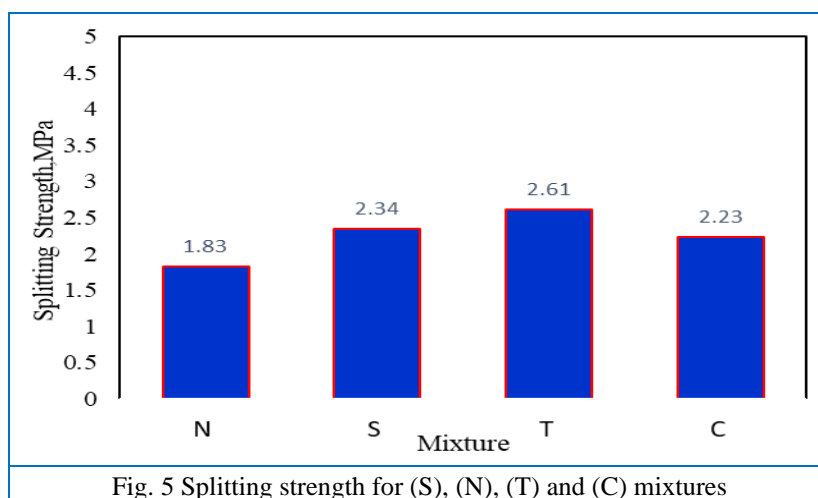


Fig. 5 Splitting strength for (S), (N), (T) and (C) mixtures

3.3. Concrete density mixtures

An important factor in determining where to use concrete in structures is density, Therefore the differences in the weights of concrete were studied depending on the nanomaterials added to it and ages per days.

The weight of specimens for all mixtures was measured experimentally (Density = Mass / Volume (Kg/m^3)) at 7, 28 and 56 days, From the Fig. 6 and Table 14 a reduction in the unit weight for the concrete mixes with nano materials compared with normal mix concrete. For the mixes with nano material, a highest unit weight was observed at the age of 28 days.

The highest unit weight for the mixture with Nano Titanium (T) replacement by (2.5%) from weight of cement. This (T) mixture shows a highest compressive strength indicating that compressive strength depends on the unit weight and density of concrete.

Table 14 Concrete unit weight for (S), (N), (T) and (C) mixtures

No.	MIX ID	Density, Kg/m ³		
		7 Days	28 Days	56 Days
1	N (Normal concrete)	2468.5	2436.42	2453.4
2	S (3% Nano Silica)	2336.02	2391.50	2346.59
3	T (2.5% Nano Titanium)	2383.01	2446.23	2381.50
4	C (1% Carbon Nanotube)	2342.25	2357.16	2342.06

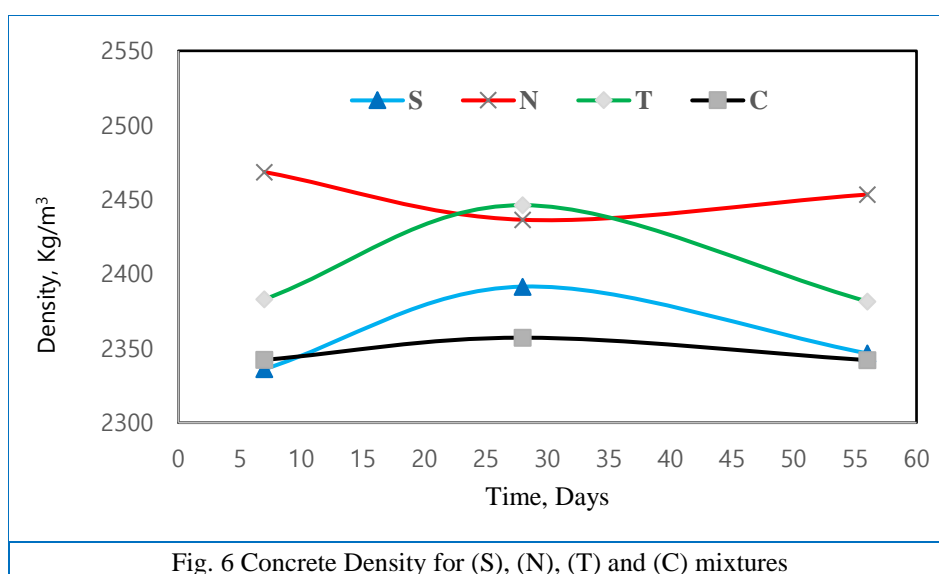
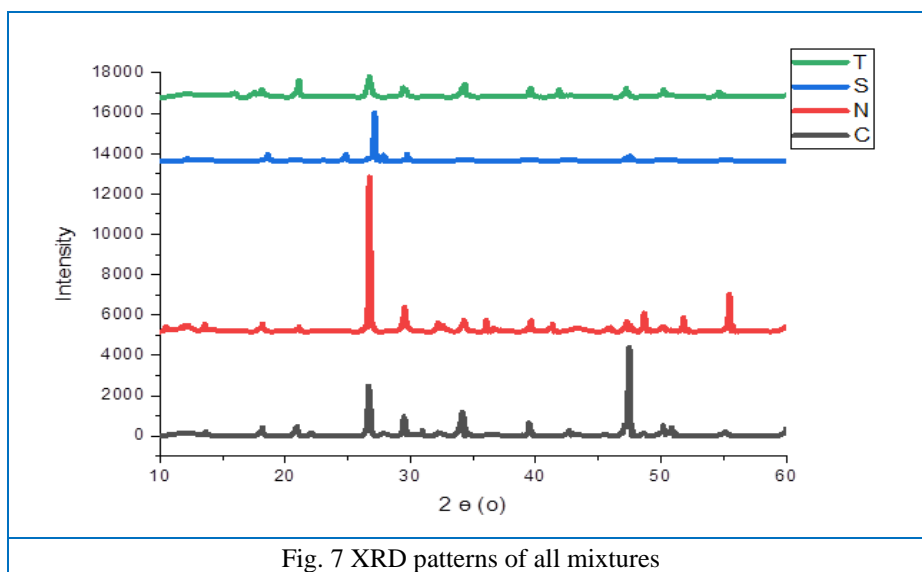


Fig. 6 Concrete Density for (S), (N), (T) and (C) mixtures

3.4. X-ray diffraction (XRD) for mixtures

It is evident from measuring X-ray diffraction (XRD) of concrete samples with the added materials, which are Carbon Nanotubes (CNT) (C), Nano Silica (S) and Nano Titanium (T), these materials added to the concrete have clearly affected on the crystalline and chemical structure of the cement component. In the case of (CNT) we noticed that it led to formation of new and main crystalline structures 2θ (47.470), as well as an increase in the intensity of the diffraction pattern at 2θ (34.130), which is attributed to calcium silicates of both types C₂S (Belite) and C₃S (Alite), noting an increase in the degree of crystallization of the system, where Nano Silica can fill the voids between particles of C-S-H gel, demonstrating interstitial filling. Furthermore, as the pozzolanic reaction with Ca(OH)₂, the amount of C-S-H gel increases and leads to increased compacting formation.

When adding nano silica, a less change was observed than the previous case, with an increase in the intensity of the diffraction pattern at 2θ (18.590), which is attributed to portlandite, and a disappearance of the diffraction pattern at 2θ (55.46), which indicates the clear interaction between the added silica and calcium hydroxide. The addition of Nano Titanium had a significant impact on the crystal structure of the cement components through the appearance of many strong diffraction patterns from the beginning of the diffraction at 2θ (10) to its end 2θ (76). This indicates the ability of this material to interfere and interact with most of the cement components. Shows Fig. 7.



3.5. Scanning Electron Microscopy (SEM) for mixes

From the results of Scanning electron microscope, the effect of nano Silica on the durability and mechanical properties of concrete can be explained by microstructure.

Figure 7 are the SEM of Nano Silica modified concrete. By comparing the microstructures of Nano Silica modified concrete and control group concrete, it was found that Nano Silica not only filled the gaps between the particles in the concrete matrix, but also promoted the chemical reaction and also generates C-S-H gel to fill the pores of the concrete slurry. That is why the Nano Silica improved the durability and mechanical properties of concrete.

It can be seen from the figures that the concrete without Nano Silica was more likely to form larger crystals and pores, and the whole structure was more in-compact. When nano Silica was added to concrete, the size of pore and crystal becomes smaller and the coupling between the crystals becomes tighter, which makes the microstructure more compact.

The impact of Nano Titanium and Nono Silica on the durability and mechanical characteristics of concrete can be elucidated through an examination of the microstructure. It was observed that Nano Titanium and Nono Silica facilitated chemical reactions and encouraged the formation of C-S-H gel, which filled the voids in the concrete mixture. As a result, Nano Titanium and Nono Silica enhanced both the durability and mechanical properties of the concrete. The introduction of Nano Titanium and Nono Silica into the concrete led to a reduction in pore size and crystal size, as well as an improvement in the interconnection between crystals, resulting in a more compact microstructure. Show Figures 8-11.

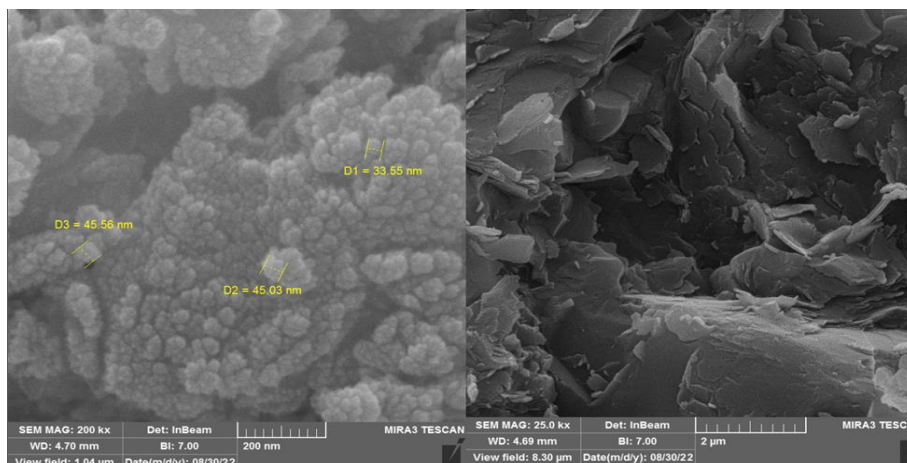


Fig. 8 SEM test for (S) mix

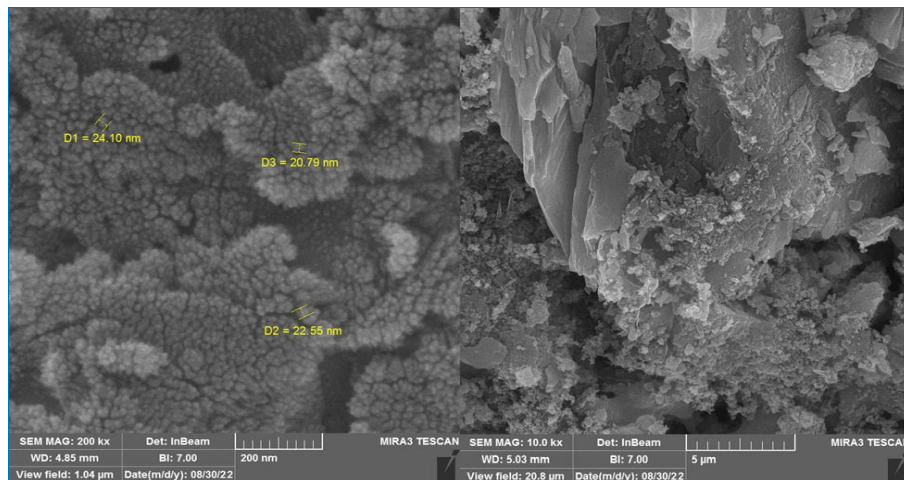


Fig. 9 SEM test for (T) mixt

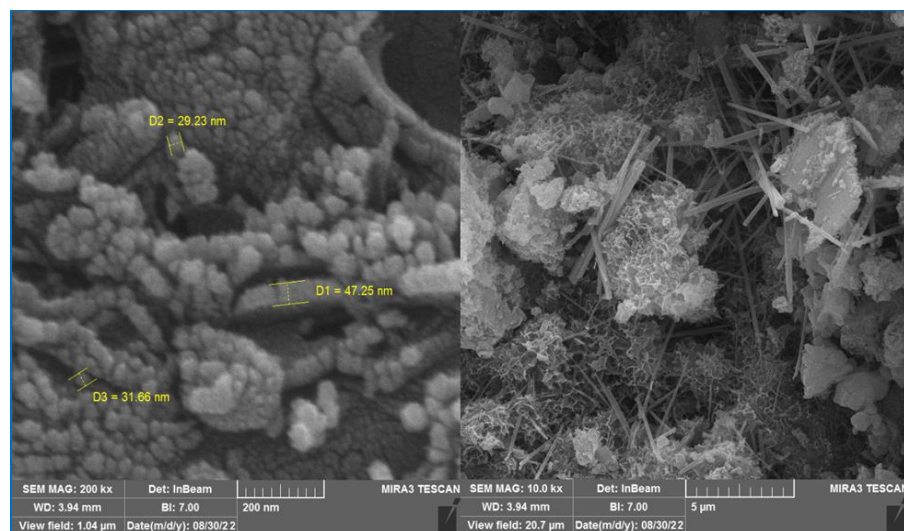


Fig. 10 SEM test for (C) mixt

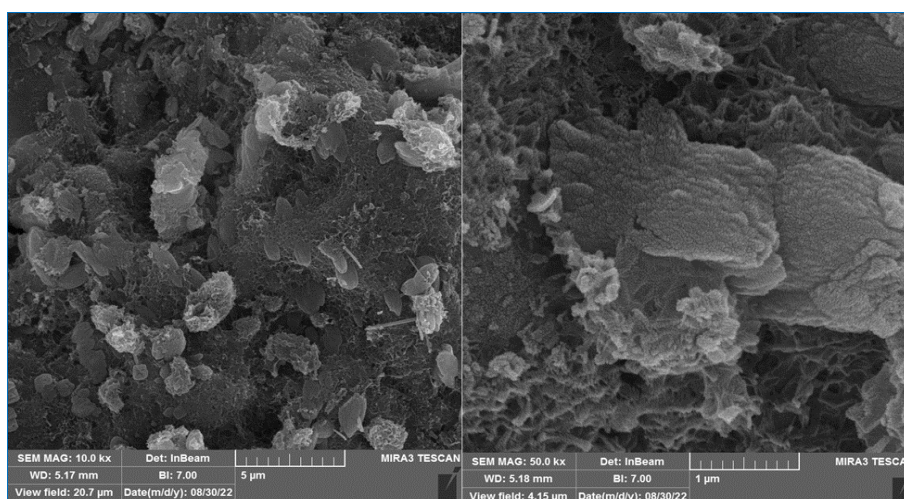


Fig. 11 SEM test for (N) mix

4. CONCLUSIONS

The main conclusions of the present study could be summarized as follows:

- When adding nanomaterials to the concrete mixtures showed an improvement in the compressive strength of concrete.
- The percentage of improvement in compressive strength was about 62% at 28 days and 80% at 7 days, compared to the reference mixture from adding nano-Titanium (TiO_2) in an amount of 2.5% replacing from weight of cement, while adding Nano Silica (SiO_2) in an amount of 3% replacing from weight of cement the percentage of improvement in compressive strength was about 2% at 56 days, 10% at 28 days and 50% at 7 days.
- The compressive strength decreased by 14% at 56 days, 22% at 28 days and 40% at 7 days, compared to the reference mixture, after the addition of carbon nanotube (CNT) in an amount of 1% replacing from weight of cement.
- In comparison to the reference mixture, adding 2.5% Nano Titanium (TiO_2), 3% Nano Silica (SiO_2), and 1% Carbon Nanotube (CNT) to the total cement weight improved the concrete splitting strength by 43%, 28%, and 22%, respectively, at 28 days.
- The density for the mixture with added Nano Titanium (T) replacement by (2.5%) from weight of cement gave highest density compared to the density for other mixtures.
- It was cleared from measuring X-ray diffraction (XRD) of concrete samples with the added Nanomaterials, which are Carbon Nanotubes CNT, Nano Silica and Nano Titanium, these materials added to the concrete have clearly affected the crystalline and chemical structure of the mixture components.
- The addition of Nano Titanium Oxide had a significant impact on the crystal structure of the cement components through the appearance of many strong diffraction patterns from the beginning of the diffraction at 2θ (10) to its end 2θ (76). This indicates the ability of this material to interfere and interact with most of the cement components.
- From the SEM results it was found that nano- SiO_2 not only filled the gaps between the particles in the concrete matrix, but also promoted the chemical reaction and also generates C-S-H gel to fill the pores of the concrete matrix. The structure of concrete was more optimized with the addition of nano- SiO_2 and the volume of capillary pores in the concrete matrix decreased.
- Incorporation of Nano Titanium TiO_2 in mixture (T) exhibits significant improvement in properties of mixture composites, enhances the density of the microstructure, furthermore, the compressive strength of the concrete. This is due to the rapid formation of C-S-H gel which accelerates the hydration of the cement and the content of $\text{Ca}(\text{OH})_2$ crystals is reduced and the large pores are filled.

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