

# Mechanical Performance and Microstructure Evolution of Nano-TiO<sub>2</sub> Enhanced Cement – A Comprehensive Experimental Analysis

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## ABSTRACT

This study focuses on improving the mechanical properties and microstructure of cement-based materials, which are crucial for the durability and safety of construction projects. Conventional cement, although commonly used, has certain limitations in terms of its mechanical strength and durability. Therefore, there is a requirement for innovative methods to enhance these properties. This study investigates the potential of nano-TiO<sub>2</sub> (titanium dioxide) as an additive to overcome these limitations. The objective of this research is to perform a thorough experimental analysis to examine how different concentrations of nano-TiO<sub>2</sub> impact the mechanical performance and microstructural changes in cement paste and mortar. The study examines the influence of nano-TiO<sub>2</sub> on the compressive and flexural strengths of cementitious materials. It also explores how nano-TiO<sub>2</sub> modifies the microstructure to enhance compactness and resilience. The results suggest that incorporating nano-TiO<sub>2</sub> into cement leads to a substantial improvement in both compressive and flexural strengths. This enhancement is particularly notable when the nano-TiO<sub>2</sub> concentration is at an optimal level of 1.0% by weight. The SEM and XRD analyses demonstrate that this concentration enhances the microstructure by decreasing voids and facilitating the development of C-S-H crystals. However, excessive concentration may have negative consequences, such as the creation of extra empty spaces. The results indicate that nano-TiO<sub>2</sub> has considerable promise in enhancing cement-based materials, thereby aiding the advancement of construction materials that are more long-lasting and effective. This study contributes to the knowledge of how nano-TiO<sub>2</sub> improves cement and emphasizes its potential uses in the construction sector.

**Keywords:** nano-particles, cement paste, cement mortar, microstructure, mechanical properties, Nano-TiO<sub>2</sub>.

## INTRODUCTION

The evolution of cementitious materials in recent decades has been significantly influenced by advancements in nanotechnology. Integrating nanoparticles into cement composites has emerged as a promising method to improve their mechanical and microstructural characteristics. This study specifically examines the effects of nano-TiO<sub>2</sub> (titanium dioxide) on the performance of cement paste and mortar. Nano-TiO<sub>2</sub>, known for its photocatalytic attributes and elevated reactivity, has demonstrated promise in enhancing the robustness, longevity, and other fundamental

characteristics of cementitious substances [1–3]. Cement plays a vital role in construction, and its quality and performance have a direct impact on the longevity and safety of buildings [4–6]. Conventional cement, although commonly utilized, has certain limitations, especially in terms of its mechanical robustness and vulnerability to environmental deterioration [7–9]. Recent research has investigated the application of nanoparticles to tackle these problems, with nano-TiO<sub>2</sub> being particularly well-studied because of its distinctive characteristics [10–12].

Multiple studies have examined the advantages of integrating nano-TiO<sub>2</sub> into cement

composites. Chen et al. (2009) conducted a study on the photocatalytic activity of  $\text{TiO}_2$ -modified concrete materials. They discovered notable enhancements in the self-cleaning and air-purifying characteristics of the concrete when recycled glass cullets were utilized as aggregates [13]. This highlights the prospective ecological advantages of incorporating nano- $\text{TiO}_2$  in construction materials.

Chen, et al. (2012) conducted additional research to investigate the hydration process and characteristics of cement composites blended with nano- $\text{TiO}_2$ . According to their report, the inclusion of nano- $\text{TiO}_2$  improved both the mechanical properties and the hydration process, resulting in the creation of stronger and longer-lasting cement structures [14]. This emphasizes the dual function of nano- $\text{TiO}_2$  in enhancing both the physical and chemical properties of cement-based materials.

In a notable study conducted by Kawakami et al. (2007), the researchers examined the impact of incorporating  $\text{TiO}_2$  powder into cement mortar on the removal of  $\text{NO}_x$  and the physical characteristics of the material. The study discovered that the addition of  $\text{TiO}_2$  not only enhanced the environmental quality by decreasing  $\text{NO}_x$  levels but also improved the physical characteristics of the cement mortar, rendering it more appropriate for a wide range of construction applications [15].

Nanoparticles can greatly enhance the mechanical properties of cementitious composites. Nazari et al. (2010) showed that incorporating  $\text{Fe}_2\text{O}_3$  nanoparticles in the concrete mixing matrix enhanced the mechanical characteristics of the resulting concrete. This study offers a comparative framework for comprehending the distinct benefits of various types of nanoparticles, such as nano- $\text{TiO}_2$ , in augmenting cementitious materials [16].

In addition to  $\text{Fe}_2\text{O}_3$ , there has been significant research conducted on  $\text{TiO}_2$  nanoparticles due to their capacity to enhance mechanical characteristics. In their study, Nazari et al. (2010) discovered that incorporating  $\text{TiO}_2$  nanoparticles into cementitious composites led to a substantial enhancement in both compressive and flexural strengths. The statement highlights the capacity of  $\text{TiO}_2$  nanoparticles to improve the strength and stability of cement-based materials [17].

Raki et al. (2010) conducted a study that offers a thorough examination of how nanoscience and nanotechnology are used in cement and concrete. Their results emphasize the capacity of nanoparticles to alter the microstructure of cement composites, resulting in enhancements in durability,

strength, and other crucial characteristics. This review highlights the significance of continuous research in this domain to fully maximize the advantages of nanotechnology in construction materials [18].

Senff et al. (2012) investigated how the addition of nano- $\text{SiO}_2$  and nano- $\text{TiO}_2$  affects the rheological behavior and hardened properties of cement mortars. Their research suggests that the inclusion of these nanoparticles can substantially modify the rheological characteristics of the mortar, resulting in improved workability and enhanced mechanical properties. This study offers valuable insights into the practical utilization of nanoparticles in cement-based materials [19].

Xiong et al. (2006) investigated the characteristics of cement-based composites that were enhanced with nano- $\text{TiO}_2$ . Their study showed that incorporating nano- $\text{TiO}_2$  enhanced the compressive strength and durability of the composites, providing further evidence for the beneficial use of nano- $\text{TiO}_2$  as an additive in cementitious materials [20].

Utilizing standardized test methods and procedures is essential for guaranteeing consistent and dependable outcomes when assessing the properties of cement and mortar [7, 21]. The ASTM C1437 (1999) offers a standardized procedure for measuring the flow of hydraulic cement mortar [22]. This test is crucial for evaluating the workability and uniformity of mortar mixes. Similarly, EN 1015-11 (1999) and EN 1015-3 (2007) offer techniques for measuring the bending and crushing strength of solidified mortar, as well as the viscosity of newly mixed mortar, respectively [23, 24]. These standards are essential for guaranteeing the caliber and efficacy of cementitious materials in construction.

The assessment of cement strength is governed by the EN 196-1 (2005), which provides guidelines for evaluating the compressive and flexural strengths of cement [25]. These established protocols are crucial for assessing the mechanical characteristics of cement composites and verifying their appropriateness for diverse construction uses [7, 15].

Furthermore, it is crucial to conduct research on the potential radiological hazards linked to the utilization of natural materials in cement manufacturing, alongside technical studies. In their study, Abdallah et al. (2022) examined the performance, measurements, and possible radiological hazards associated with the natural radioactivity found in cements utilized in Jordan. These

findings emphasize the importance of thoroughly evaluating and controlling the potential dangers associated with the use of natural materials in construction [26].

Another study investigates the incorporation of natural sand-supported nano-TiO<sub>2</sub> hydrosol (TiO<sub>2</sub>@Sand) into cement mortar, focusing on its impact on mechanical and photocatalytic performance, as well as the underlying mechanisms. The results are promising, showing a significant improvement in both mechanical strength and photocatalytic activity of the cement mortar [27].

Recent research has also investigated the utilization of substitute materials and residual products in the manufacturing of cement. In their study, Gougazeh et al. (2022) investigated the behavior of oil shale from Jordan during the process of combustion and its potential application in cement manufacturing. According to their research, oil shale ash can be utilized as a substitute green ingredient in cement, providing both environmental and economic advantages [28]. Alsafasfeh et al. (2022) conducted a study on the utilization of oil shale ash as an environmentally friendly ingredient in cement manufacturing. Their research emphasizes the possibility of implementing sustainable methods in the construction sector [29].

Prior research has thoroughly examined the influence of nano-TiO<sub>2</sub> on different aspects of cement composites, such as improved photocatalytic activity, hydration processes, and mechanical properties. However, there is still a lack of understanding regarding the overall effects of nano-TiO<sub>2</sub> on both the microstructural changes and mechanical behavior of cement paste and mortar at varying concentrations. Furthermore, there is a scarcity of research that thoroughly investigates the ideal concentration of nano-TiO<sub>2</sub> that achieves a harmonious combination of enhanced mechanical strength and practical factors such as workability and long-term durability.

Despite numerous studies highlighting the potential benefits of nano-TiO<sub>2</sub> in enhancing cementitious materials, there remains a notable gap in the literature regarding the specific percentages of improvement achieved in concrete properties. While research by Chen et al. (2012) and Xiong et al. (2006) demonstrated enhancements in compressive and flexural strengths, as well as durability, they did not provide detailed quantitative analyses of these improvements [14, 20]. Similarly, Kawakami et al. (2007) and Nazari et al. (2010) discussed the overall positive impact of

TiO<sub>2</sub> nanoparticles on mechanical properties but did not quantify the extent of these enhancements [15, 30]. This lack of specific percentage data underscores the need for further research that systematically quantifies the improvements in concrete properties due to the addition of nano-TiO<sub>2</sub>, thereby providing more concrete evidence of its efficacy in construction applications.

This study aims to fill these knowledge gaps by conducting a comprehensive experimental analysis on the impact of nano-TiO<sub>2</sub> on the microstructure and mechanical characteristics of cementitious materials. This study is unique because it systematically examines various concentrations of nano-TiO<sub>2</sub> to identify the ideal amount that improves compressive and flexural strengths while minimizing negative effects like increased void formation. This study not only enhances the fundamental comprehension of the impact of nano-TiO<sub>2</sub> on the characteristics of cement-based materials but also provides practical knowledge for the advancement of construction materials that are more long-lasting and resistant. The results have substantial ramifications for enhancing the utilization of nanotechnology in the construction sector, specifically in enhancing the efficiency and durability of cement-based buildings.

## EXPERIMENTAL WORK

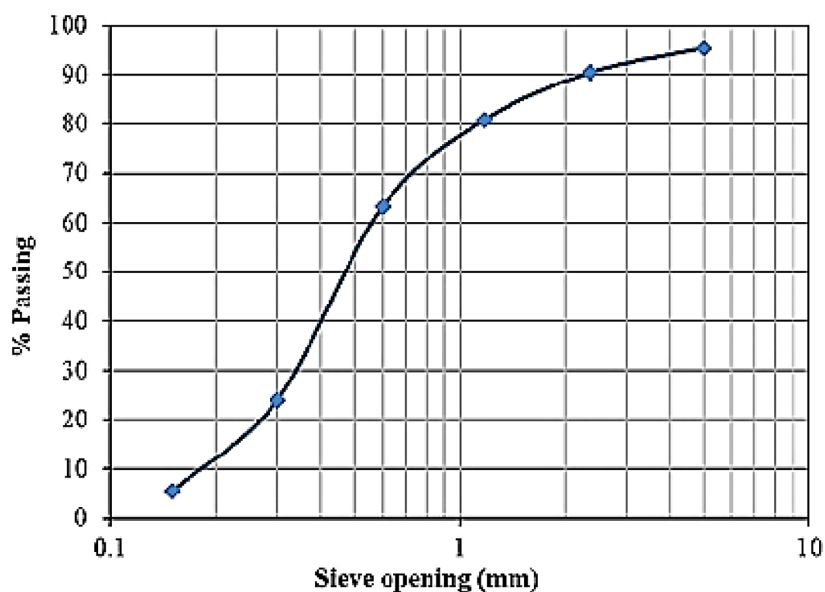
### Materials

The experimental study utilized Ordinary Portland Cement (OPC type I 42.5N) obtained from Lafarge Cement Company (LCCO), which adhered to the specifications outlined in the Jordanian standard (JSS. 30/1979) [26]. The cement's chemical composition was determined through X-Ray Fluorescence (XRF) analysis, as presented in Table 1.

The mortars were made using natural sand sourced from a quarry in Jerash, Jordan. The sand had particles smaller than 2 mm as presented in Figure 1, a fineness modulus of 2.25, and a specific gravity of 2.60 g/cm<sup>3</sup>. The sand met the specifications outlined in the Jordanian standard (JSS. 96/1987) [26]. For mixing and curing, clean and potable water from authorized sources in Jordan was used, in accordance with the Jordanian standard (JSS. 1376/2003) [26]. The specific properties of the nano-TiO<sub>2</sub> (DOP ORGANIC KIMYA SAN, Turkey) used in the study can be found in Table 2.

**Table 1.** Chemical composition of cement by XRF analysis

Composition	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	MgO
Percentage (%)	64.28	16.77	9.25	6.85	2.42	0.38	0.03	0.02



**Figure 1.** Particle size distribution of sand

### Scanning electron microscopy (SEM)

The microstructural analysis of the cement paste and mortar samples was conducted using a JEOL-5500 LV scanning electron microscope (SEM). This technique was employed to examine the morphology, chemical composition, and crystalline structure of the samples. The SEM analysis provided detailed images with magnifications ranging from 20X to 30,000X, enabling the observation of C-S-H formations and void structures. The SEM was particularly valuable for assessing the distribution of nano-TiO<sub>2</sub> particles and the overall compactness of the microstructure. This information was crucial in correlating the microstructural characteristics with the observed mechanical properties.

### X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was performed to identify and quantify the crystalline phases present in the cement paste samples. The technique was

utilized to detect the formation of hydration products such as calcium silicate hydrate (C-S-H) and calcium hydroxide (Ca(OH)<sub>2</sub>), and to assess the impact of nano-TiO<sub>2</sub> on the hydration process. The XRD data provided insights into the size of the nano-crystals and the overall crystallinity of the material, which were used to infer the efficiency of the hydration process at different nano-TiO<sub>2</sub> concentrations.

### Flow table test

The workability of the mortar was assessed using the flow table test, as described in ASTM C1437-99. This test method is used to measure the flow of hydraulic cement mortars, providing a standardized approach to evaluating the workability and consistency of the mixes.

### Compressive strength

The compressive strength was tested on both cubic (7.07 × 7.07 × 7.07 cm) and prism (4 × 4 × 16 cm) samples in accordance with EN 196-1:2005 to

**Table 2.** Properties of nano-TiO<sub>2</sub>

Mean particle size (nm)	Specific surface area (m <sup>2</sup> /g)	Bulk density (g/cm <sup>3</sup> )	True density (g/cm <sup>3</sup> )	Purity (%)	Structure
15	240	0.04–0.06	3.9	99	Anatase

evaluate the influence of sample geometry on the mechanical properties. This approach allows for a more comprehensive assessment of the material's strength characteristics under different stress distributions. The cubic samples provide a standard measure of compressive strength, while the prism samples, which were also used for flexural strength tests, offer insights into how the material behaves under both compressive and flexural loads. Testing both types of samples ensures that the results are robust, and representative of real-world applications where different geometries may be encountered. Head displacement was adopted as press settings in the compressive strength test.

### Flexural strength

The flexural strength of mortar prisms was tested using a three-point bending method and head displacement was adopted as press settings, as specified in EN 1015-11:1999. This standard provides the procedure for determining the flexural and compressive strength of hardened mortar, which was followed to evaluate the structural performance of the mortar with varying nano-TiO<sub>2</sub> content.

### Preparation of paste and mortar

Pastes were created by combining cement (C) and nano-TiO<sub>2</sub> to form cementitious materials

(cm), with a water-to-cementitious material (W/cm) ratio of 0.29. Nano-TiO<sub>2</sub> was incorporated into the cement mixture at weight ratios of 1.0%, 1.5%, 2.0%, 2.5%, and 3.0%. Table 3 provides a detailed overview of the paste preparations. The amounts of paste ingredients presented in Table 3 were used to fill Vicat's mold, (40 mm in deep and 80 mm in diameter), to determine the amount of water required for the standard consistency. Each mix proportion was used for one test. The mortars were prepared in accordance with EN 196-1:2005, which specifies the method for testing the compressive and flexural strengths of hydraulic cement mortars. The proportions and mixing procedure adhered to this standard to ensure consistency across all samples.

Fresh mortars were prepared using a cm/sand ratio (cm/S) of 0.33 and a W/cm ratio of 0.50. Nano-TiO<sub>2</sub> was incorporated into the cement mixture as a substitute for a portion of the cement, using the same proportions as in previous paste samples. Table 4 displays the mortar preparations. The amounts of mortar ingredients were used to fill 6 cubes of 50 mm cube specimens.

A rotary mixer equipped with an inclined beater was employed for the purpose of mixing. The initial step involved the introduction of Nano-TiO<sub>2</sub> into water, followed by vigorous stirring at a speed of 3000 revolutions per minute for a duration of 5 minutes. Subsequently, the mixture

**Table 3.** Paste preparations

Paste designation	Mix proportion (g)		Water content	
	C	Nano-TiO <sub>2</sub>	W/C <sub>m</sub>	W <sub>w</sub> (g)
PT <sub>0.0</sub>	400	0	0.29	116
PT <sub>1.0</sub>	396	4	0.29	116
PT <sub>1.5</sub>	394	6	0.29	116
PT <sub>2.0</sub>	392	8	0.29	116
PT <sub>2.5</sub>	390	10	0.29	116
PT <sub>3.0</sub>	388	12	0.29	116

**Table 4.** Mortar preparations

Mortar designation	Mix proportion (g)			Water content	
	C	Nano-TiO <sub>2</sub>	S	W/C <sub>m</sub>	W <sub>w</sub> (g)
MT <sub>0.0</sub>	555	0	1665	0.50	277.5
MT <sub>1.0</sub>	549.45	5.55	1665	0.50	277.5
MT <sub>1.5</sub>	546.67	8.33	1665	0.50	277.5
MT <sub>2.0</sub>	543.90	11.10	1665	0.50	277.5
MT <sub>2.5</sub>	541.12	13.88	1665	0.50	277.5
MT <sub>3.0</sub>	538.35	16.65	1665	0.50	277.5

was blended with cement and agitated for an additional duration of 2 minutes. Vicat's mold was used to cast fresh pastes in order to determine the setting time.

After a period of 28 days, samples were chosen for the purpose of determining their dry density (unit weight) and examining their microstructure using SEM and XRD. Mortar specimens were fabricated by blending cement and sand in a dehydrated state for a duration of 2 minutes. Subsequently, nano-TiO<sub>2</sub> was introduced with water and mixed for an additional 3 minutes. Two flow table measurements were conducted immediately after the mixing process. Subsequently, mortars were inserted into cubic containers measuring 7.07 × 7.07 × 7.07 cm for the purpose of conducting compressive tests. Additionally, prisms measuring 4 × 4 × 16 cm were used for both flexural and compressive tests. Compaction was achieved using a table vibrator.

After 24 hours, the specimens were removed from the mold and placed in fresh water at a temperature of 23 ± 2 °C for curing. A total of twelve cubic specimens with dimensions of 7.07 × 7.07 × 7.07 cm were created from each mixture and subjected to testing at 2, 7, 28, and 56 days to examine the impact of nano-TiO<sub>2</sub> on the strength of mortar as it evolves over time. Additionally, twelve prism specimens with dimensions of 4 × 4 × 16 cm were created for each mixture to test their flexural strength.

## RESULTS AND DISCUSSION

### Effect of Nano-TiO<sub>2</sub> content on the behavior of cement paste

#### Setting time

Observations revealed that the inclusion of nano-TiO<sub>2</sub> at a weight concentration of 1.0% resulted in a notable reduction in the setting time of the cement paste (Figure 2). The observed phenomenon can be ascribed to the substantial surface area and reactivity of nano-TiO<sub>2</sub> particles, which facilitate the swift utilization of available water and expedite the creation of initial hydration products, such as Calcium Silicate Hydrate (C-S-H) and Calcium Hydroxide (Ca(OH)<sub>2</sub>). The presence of nano-TiO<sub>2</sub> particles likely increases the number of nucleation sites, which in turn accelerates the process of cement hydration and results in shorter setting times. Chen et al. (2012) also found similar results, showing that the inclusion of nano-TiO<sub>2</sub> improved the hydration process, leading to faster setting and increased strength at an early stage [8].

Conversely, when the nano-TiO<sub>2</sub> content was increased to 1.5%, there was a significant prolongation of the setting time. The excessive abundance of nano-TiO<sub>2</sub> particles can be attributed to the agglomeration and hinderance of effective dispersion within the cement matrix. Nanoparticle agglomeration leads to the formation of localized regions with a high concentration of particles,

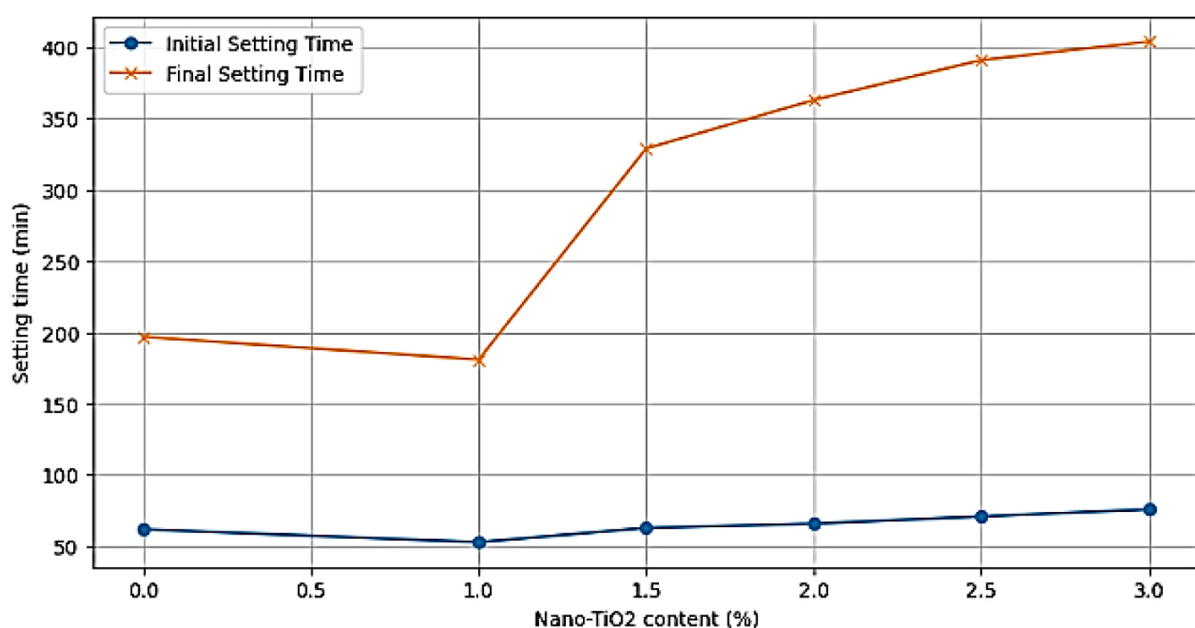


Figure 2. Variations of setting time with nano-TiO<sub>2</sub> content

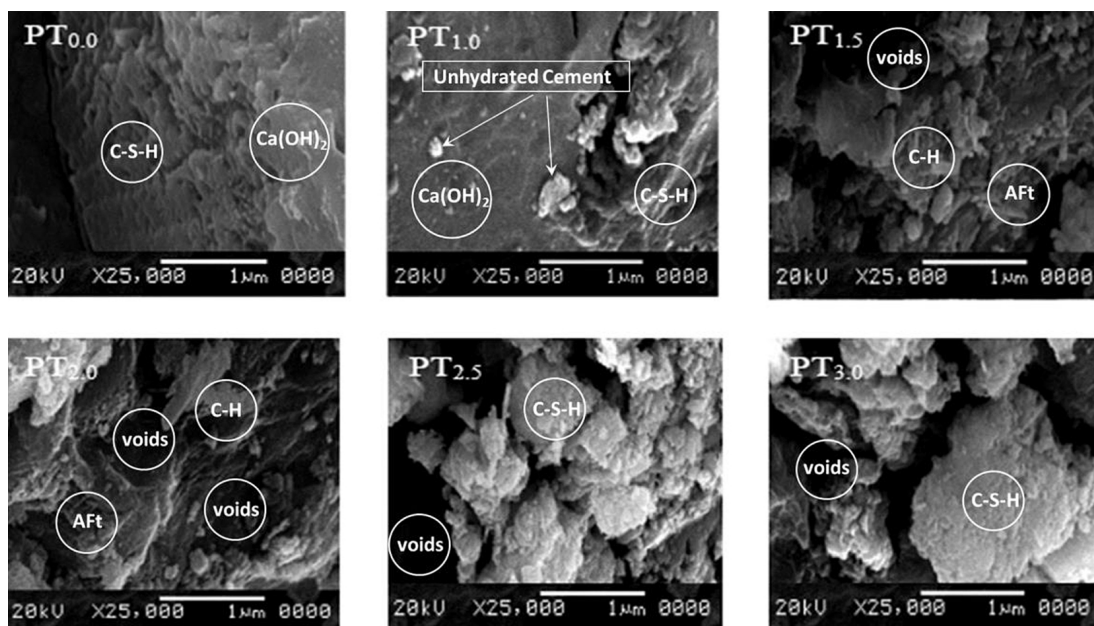
which in turn decreases the surface area available for hydration and consequently slows down the overall hydration process. Moreover, the heightened need for water to moisten and distribute the greater amount of nano-TiO<sub>2</sub> particles may result in a relative scarcity of available water required for hydration, thereby causing additional delays in the setting time. Senff et al. (2012) found that higher concentrations of nanoparticles can have a negative impact on the workability and setting time of cement-based materials. This is because of the increased viscosity and particle-particle interactions caused by the nanoparticles.

*Microstructure study*

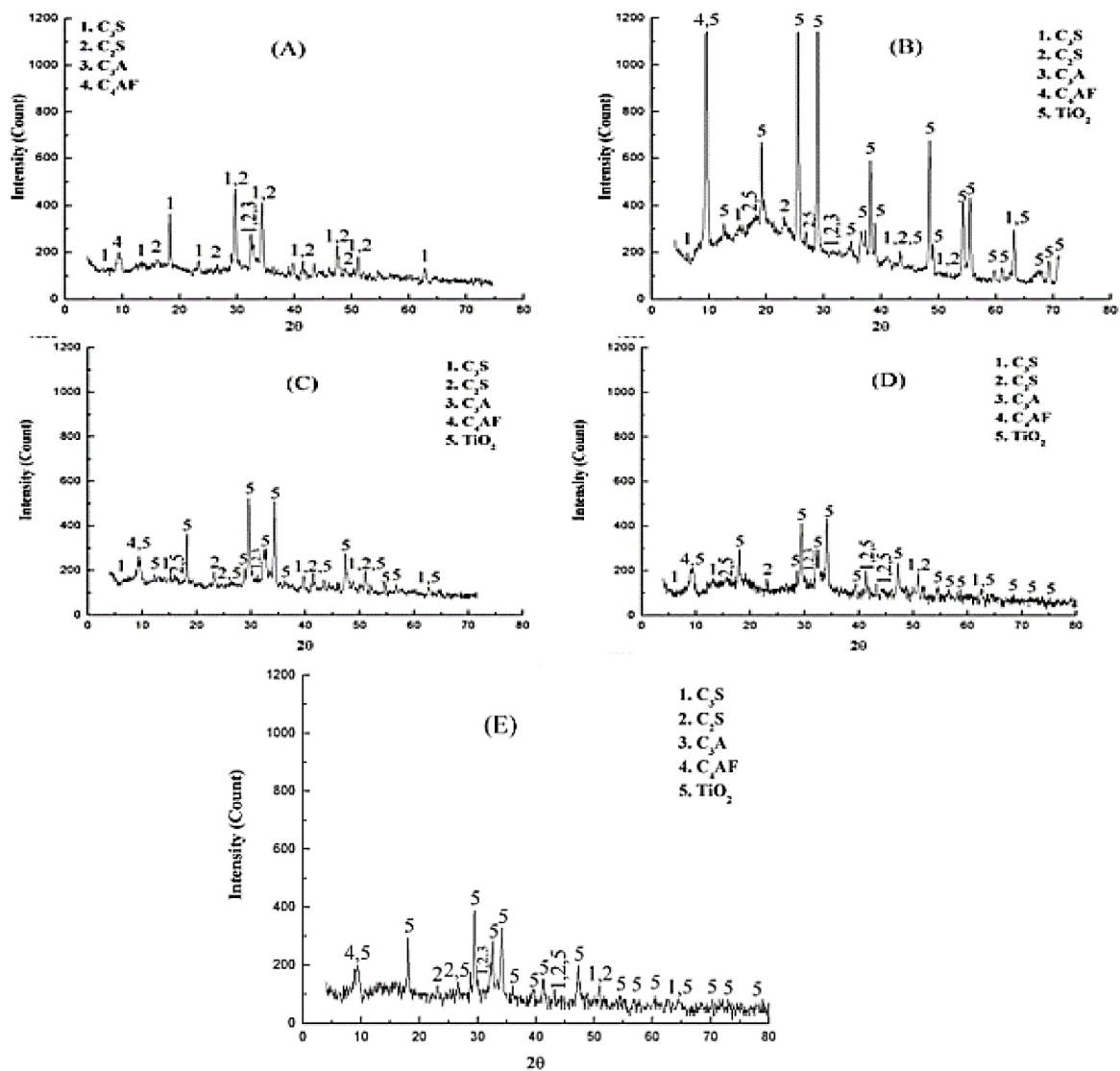
The scanning electron microscope (SEM) images, as presented in Figure 3, of cement paste containing 1.0% nano-TiO<sub>2</sub> show a microstructure that is more tightly packed and well-arranged. This is evident from the presence of dense formations of calcium-silicate-hydrate (C-S-H) and a decrease in empty spaces. The observed enhancements in mechanical properties can be attributed to the more uniform and compact microstructure, which promotes improved load distribution and increased strength. The positive impacts of incorporating 1.0% nano-TiO<sub>2</sub> on the microstructure can be ascribed to the ideal distribution of nanoparticles, which promotes the creation of a seamless and strong C-S-H network. Nevertheless, when the concentration of nano-TiO<sub>2</sub> is raised to 1.5%, scanning electron

microscopy (SEM) images reveal a microstructure that is more varied, with larger empty spaces and reduced density of calcium-silicate-hydrate (C-S-H) formations. This observation is consistent with the previously mentioned longer setting time and decreased workability. The accumulation of nano-TiO<sub>2</sub> particles at higher concentrations is likely to disturb the consistency of the cement matrix, resulting in an elevation of porosity and a less dense microstructure. According to the literature, the impact of nanoparticles on improving microstructure is greatly influenced by how well they are spread out within the matrix. If the dispersion is not good, it can cancel out the potential advantages and even harm the properties of the material [12].

Figure 4 displays the X-ray diffraction (XRD) patterns of cement paste, comparing samples with and without nano-TiO<sub>2</sub>. Upon comparing Figure 3A with Figure 3E, it is clear that the addition of 1.0% nano-TiO<sub>2</sub> improves the hydration process. The sample (A) without nano-TiO<sub>2</sub> shows characteristic peaks corresponding to the main hydration products, including calcium silicate hydrate (C-S-H), calcium hydroxide (Ca(OH)<sub>2</sub>), and unreacted clinker phases such as tricalcium silicate (C<sub>3</sub>S) and dicalcium silicate (C<sub>2</sub>S). These peaks indicate the standard hydration process of ordinary Portland cement. In sample (B), with 1.0% nano-TiO<sub>2</sub>, there is a noticeable increase in the intensity of peaks associated with C-S-H and Ca(OH)<sub>2</sub>. This suggests that the addition of 1.0% nano-TiO<sub>2</sub> enhances the hydration process,



**Figure 3.** Microstructure of the samples with different contents of nano-TiO<sub>2</sub>



**Figure 4.** XRD patterns of cement paste without nano  $\text{TiO}_2$  (A), with 1.0% by weight of nano- $\text{TiO}_2$  (B), with 1.5% by weight of nano- $\text{TiO}_2$  (C), with 2.0% by weight of nano- $\text{TiO}_2$  (D), with 3.0% by weight of nano- $\text{TiO}_2$  (E)

leading to a denser and more crystalline structure. The higher intensity of these peaks indicates more extensive formation of hydration products, which can contribute to improved mechanical properties and durability of the cement paste.

The XRD analysis supports the previous discoveries regarding the impact of nano- $\text{TiO}_2$  on the hydration process and microstructural formation of cement paste. An ideal concentration of nano- $\text{TiO}_2$  for enhancing the hydration process and improving the microstructure seems to be approximately 1.0% by weight. Past this point, the advantages lessen and the effectiveness of hydration decreases, most likely because the nanoparticles clump together and physically impede the hydration process. The comprehensive examination is consistent with the observed mechanical

properties and workability results, thereby strengthening the conclusion that a 1.0% concentration of nano- $\text{TiO}_2$  is the most effective content for enhancing the performance of cementitious materials. The results emphasize the importance of achieving a delicate equilibrium between improving hydration and ensuring optimal particle distribution to achieve the most favorable results in cement paste and mortar formulations.

### Effect of nano- $\text{TiO}_2$ content on the behavior of cement mortars

#### Workability

Table 5 summarizes the average flow diameters of mortar samples with different nano- $\text{TiO}_2$



**Table 5.** Mortar average flow diameters with nano-TiO<sub>2</sub> content

Average diameter (mm)	Specimen					
	MT <sub>0.0</sub>	MT <sub>1.0</sub>	MT <sub>1.5</sub>	MT <sub>2.0</sub>	MT <sub>2.5</sub>	MT <sub>3.0</sub>
D	182	157	139	132	121	114

contents. The results demonstrate that all mortars containing nano-TiO<sub>2</sub> exhibit lower flow values compared to the control specimen. The flow spread values decrease as the nano-TiO<sub>2</sub> content increases.

The decrease in workability can be ascribed to the augmented surface area of the binder resulting from the partial substitution of cement with nano-TiO<sub>2</sub>. The inclusion of small particles modifies the flow characteristics of the mixture, consequently impacting the ease of handling of the building materials.

*Compressive strength of mortar cubes*

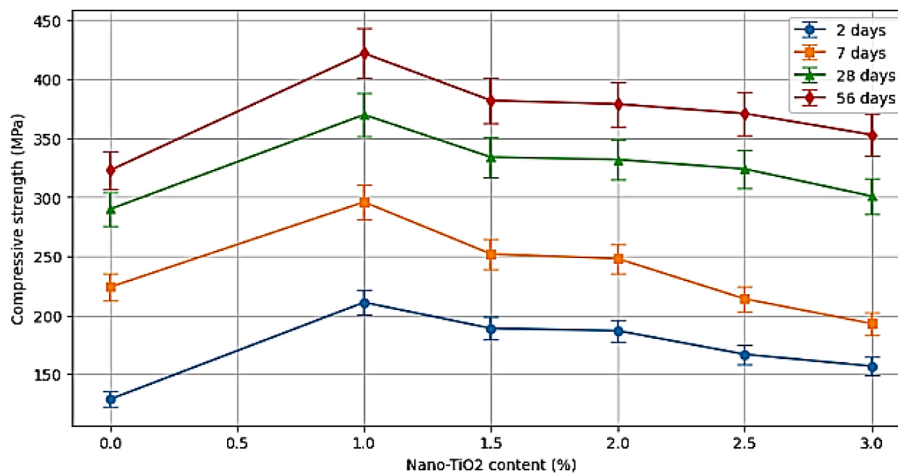
Figure 5 illustrates the impact of the amount of nano-TiO<sub>2</sub> on the compressive strength of mortar cubes. The addition of nano-TiO<sub>2</sub> greatly enhances the compressive strength at different time intervals (2, 7, 28, and 56 days). When the nano-TiO<sub>2</sub> content is 1.0%, the compressive strength is improved by 64%, 32%, 28%, and 31% after 2, 7, 28, and 56 days, respectively. Increasing concentrations of nano-TiO<sub>2</sub> (1.5%, 2.0%, 2.5%, and 3.0%) exhibit diminishing returns, accompanied by adverse effects at higher ages. These findings indicate that nano-TiO<sub>2</sub> has a beneficial effect on the initial hydration of C3S, but its influence on the later-stage hydration of C<sub>2</sub>S is minimal [14].

The primary cause for this behavior may be attributed to the nanoparticles' capacity to alter the physical and chemical properties of the mortar

materials. Firstly, the introduction of nanoparticles into mortar samples can alter their microstructure by occupying the nanopores, resulting in a decrease in porosity. This, in turn, leads to an overall increase in compressive strength. Additionally, nanoparticles have the potential to react chemically by increasing the production of C-S-H gel, thereby enhancing the mechanical strength[31]. The presence of this extra C-S-H will lead to the formation of a thicker and more robust interfacial transition zone (ITZ) between the cement and aggregates, serving as nucleation sites. Furthermore, the alignment of the crystal structure of Ca(OH)<sub>2</sub> in the aggregate and cement mortar can be improved as a result of the interaction between nanoparticles and Ca(OH)<sub>2</sub>. This interaction leads to the formation of C-S-H gel and consequently reduces the quantity of Ca(OH)<sub>2</sub> [31].

*Modulus of rupture*

Figure 6 illustrates the impact of nano-TiO<sub>2</sub> on the modulus of rupture for mortar prisms at various stages of curing. An inclusion of 1.0% nano-TiO<sub>2</sub> leads to an augmentation in flexural strength of 8% and 11% after 28 and 56 days, respectively. Furthermore, the flexural strength diminishes beyond this point, exhibiting decreases at all ages that were examined. The inclusion of 1.0% nano-TiO<sub>2</sub> content enhances the speed of cement hydrate formation and enhances the quality of the pore structure.



**Figure 5.** Compressive strength of mortar cubes with nano-TiO<sub>2</sub> content at different ages

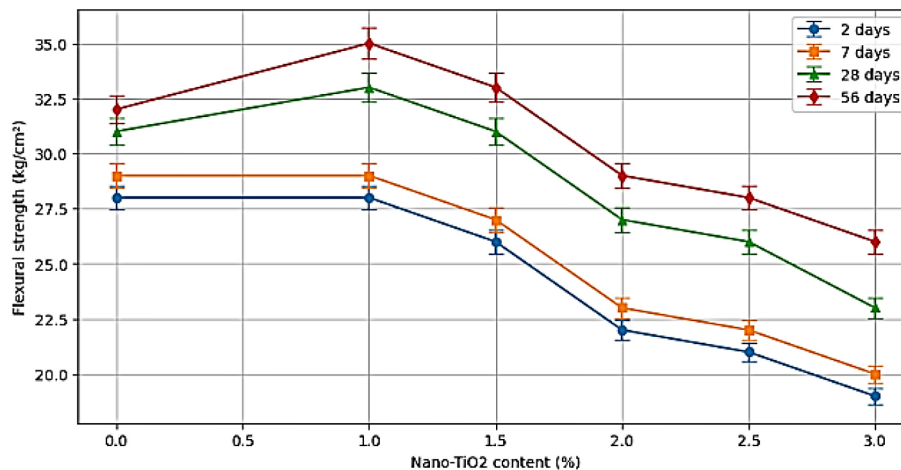


Figure 6. Modulus of rupture for mortar prisms with nano-TiO<sub>2</sub> content at different ages

Nevertheless, a higher concentration of nano-TiO<sub>2</sub> necessitates a greater amount of water, resulting in a reduction in the coefficient of water absorption and diminishing the enhancement of pore structure [19].

This phenomenon can be explained by the fact that as the content of nano-TiO<sub>2</sub> increases, the distance between individual nanoparticles decreases. The limited space available hampers the growth of Ca(OH)<sub>2</sub> crystals, leading to a reduced number of crystals and a lower ratio of crystals to strengthening agents. As a result, the cement matrix experiences shrinkage and creep, causing the pore structure to become relatively more porous. In general, the inclusion of nanoparticles enhances the pore structure of mortar. Conversely, nano-TiO<sub>2</sub> particles function as a substance that fills in gaps, increasing the thickness of the paste and greatly decreasing the amount of empty spaces in mortar. Additionally, they function as a catalyst, speeding up the process of cement hydration as a result of their reactivity, and serve as a nucleus

within the cement paste, decreasing the dimensions and enhancing the random arrangement of Ca(OH)<sub>2</sub> crystals [15].

The addition of 1% nano TiO<sub>2</sub> to the cement mixture enhances the compressive strength compared to the control sample. The increased strength of 1% nano TiO<sub>2</sub> may be attributed to its smaller particle size, which allows for greater pore filling and consequently reduces porosity. Adding 1% nano-TiO<sub>2</sub> increased the strength by 64%, 32%, 28%, and 31% at 2, 7, 28, and 56 days, respectively. The variation in strength may be attributed to a decrease in the quantity of nanoparticles, resulting in fewer nucleation sites and consequently lower mechanical strengths. Nevertheless, the reduction in potency observed when the dosage is increased from 1.5% to 3% may be attributed to the agglomeration phenomenon resulting from elevated surface energy. Excessive water absorption could result in self-desiccation and cracking of specimens [32].

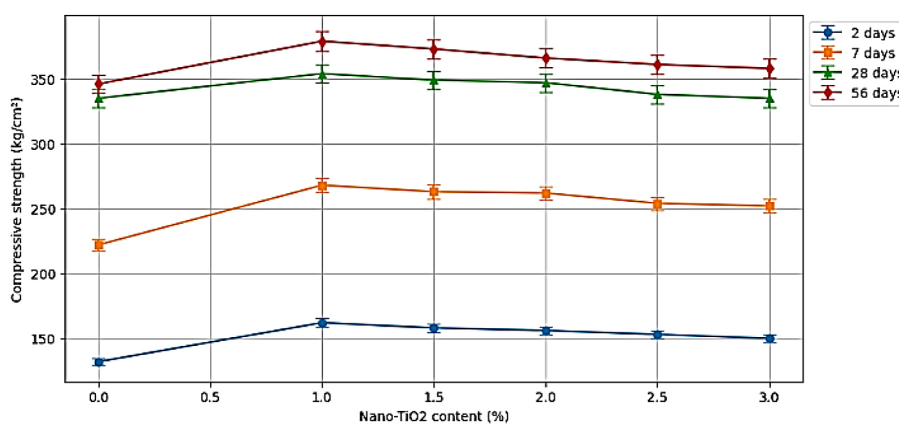


Figure 7. Compressive strength for mortar prisms with nano-TiO<sub>2</sub> content at different ages

### Compressive strength of mortar prisms

Figure 7 depicts the changes in the compressive strength of mortar prisms containing varying amounts of nano-TiO<sub>2</sub> over different time periods of 2, 7, 28, and 56 days. Specimens containing nano-TiO<sub>2</sub> consistently demonstrate superior compressive strength compared to plain cement mortar across all tested time periods. The highest level of strength enhancement is achieved when using a 1.0% replacement of nano-TiO<sub>2</sub>, suggesting that this is the most suitable amount for maximizing the compressive strength of mortar. The addition of nano-TiO<sub>2</sub> enhances the microstructure and mechanical properties of cement paste and mortar. The study confirms that the optimal content of nano-TiO<sub>2</sub> for improving the performance of cementitious materials is 1.0% by weight, providing a balance between increased strength and manageable workability.

### CONCLUSIONS

This study has shown that the inclusion of nano-TiO<sub>2</sub> in cement paste and mortar has a substantial impact on their mechanical properties and microstructure. The most effective amount of nano-TiO<sub>2</sub> was determined to be 1.0% by weight, resulting in a significant enhancement in both compressive and flexural strengths. More precisely, the mortar's ability to withstand compression increased by as much as 64% during the initial stages (2 days), and its ability to resist bending improved by 11% after 56 days of curing. Nevertheless, when the nano-TiO<sub>2</sub> content exceeded 1.0%, there was a decline in workability, making it harder to compact the material. This likely led to reduced strength gains and increased variability in the outcomes. When the concentration of nano-TiO<sub>2</sub> reached 1.5% or higher, the setting time of the material increased. Additionally, the microstructure showed an increase in voids and a decrease in uniformity, resulting in reduced mechanical performance. The results indicate that nano-TiO<sub>2</sub> has the potential to greatly improve cement-based materials. However, it is important to carefully adjust the concentration of nano-TiO<sub>2</sub> in order to maximize its advantages without causing any negative impact on other properties.

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