

Original Article

Influence of Oxide Nanoparticles on Mechanical Performance and Freeze - Thaw Resistance of Rigid Pavement

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Abstract - Globally, rigid concrete pavements suffer premature deterioration when repeated traffic is combined with moisture and temperature fluctuations, as capillary water ingress and freeze-thaw cycles cause internal microcracking, loss of stiffness and a reduction in the modulus of elasticity. In Peru, this problem is critical in high Andean areas, where sub-zero temperatures and high humidity accelerate internal damage and increase road maintenance costs. The research comparatively evaluated the effect of nano-SiO₂, nano-TiO₂ and nano-ZnO as concrete admixtures for rigid pavement with a design strength of $f'_c = 280$ kg/cm², analysing workability, mechanical strength and durability. Dosages of 1–5% for nano-SiO₂, 0.5–2.5% for nano-TiO₂ and 0.3–0.7% for nano-ZnO were studied, relative to the cement mass. In the fresh state, slump and temperature were assessed; in the hardened state, compressive and flexural strength were assessed. Durability was assessed by freeze-thaw testing in accordance with ASTM C666, using the dynamic modulus of elasticity as an indicator of damage, and by sorptivity in accordance with ASTM C1585. The results showed significant improvements compared to the standard mix, with the response depending on the type and dosage. Nano-SiO₂ exhibited the best overall performance, with an optimal dosage of 3%, increases of 27.65% in compression and 36.89% in flexural strength, an RDME of 92.58% and a reduction in both initial and secondary sorptivity. Nano-TiO₂ was optimal at 2%, and nano-ZnO at 0.6%, with more moderate improvements. The study provides evidence for the design of durable, rigid pavements.

Keywords - Concrete pavements, Flexural strength, Freeze-Thaw, Nanoparticles, Sorptivity.

1. Introduction

Globally, the performance of rigid hydraulic concrete pavements is governed by the interaction between traffic loads and environmental factors [1]. Temperature variations, combined with moisture content, induce temperature gradients in the slab that generate curling and warping stresses; when combined with repeated traffic, these stresses increase fatigue loads, accelerate structural deterioration and affect the long-term performance of the pavement [2, 3]. In Peru, the road network crosses high-altitude Andean regions characterised by low seasonal temperatures and high humidity, creating conditions conducive to water ingress and the onset of freeze-thaw damage in concrete [4]. Within this context, the premature deterioration of rigid pavements manifests itself in the form of defects that compromise their functionality before they reach their design life [5, 6].

The problem becomes particularly relevant when moisture ingress and recurrent sub-zero temperatures act simultaneously, as these conditions promote the formation and

propagation of microcracks within the concrete, even before severe surface damage becomes evident. This process progressively deteriorates the mechanical response of the material and can be identified through reductions in the relative dynamic modulus of elasticity, a sensitive parameter for assessing accumulated internal damage associated with capillary water ingress and repeated thermal exposure [7, 8]. Studies in cold climates indicate that conventional concrete exposed to approximately 200 freeze-thaw cycles can exhibit reductions in the relative dynamic modulus of elasticity of more than 50%, even without showing immediate structural failure; this confirms that damage accumulates predominantly internally and validates this parameter as a sensitive indicator for assessing the progressive deterioration of the material [9, 10].

This trend is reinforced by the recurrence of sub-zero temperatures across the country, as evidenced by the following cases. During the winter of 2024, severe frosts were reported in the high Andean regions of Puno, Arequipa and



Tacna, with night-time temperatures of around -15°C [11]. Similarly, in 2025, Peru's National Meteorological and Hydrological Service issued frost warnings for regions such as Junín, Puno, Arequipa and Tacna, with temperatures falling below 0°C and lows ranging from -14°C to -17°C [12, 13]. Furthermore, in 2025, direct physical signs of freezing were recorded in the Puno region, such as the formation of ice layers on surface water bodies, associated with temperatures of around -16°C [14]. Finally, during the same year, spells of severe cold were reported in the Pasco region, where temperatures in the high-altitude areas of the Daniel Alcides Carrión province dropped to around -10°C [15].

In response to these conditions, the specialist literature has proposed strategies to improve the performance of concrete under freeze-thaw cycles [16, 17]. Among the most commonly used methods is the controlled introduction of air, aimed at reducing the internal pressures generated by the expansion of frozen water [16], and fibre reinforcement, used to increase the material's toughness and limit the propagation of cracks caused by thermal stresses and cyclic damage [18]. Recent research has investigated the use of nanoscale materials as a means of modifying the behaviour of concrete at the microstructural level, by promoting a denser matrix and reduced porosity; these factors are linked to resistance to capillary water ingress and the mitigation of cyclic damage [19, 20]. However, in high-altitude environments characterised by available moisture, sustained capillary action and recurring sub-zero temperatures, there is still a need for comparative and systematic evidence to establish the actual effectiveness of these solutions in rigid pavements, particularly in terms of internal deterioration and progressive loss of stiffness [6, 16]. More specifically, the available evidence does not provide a direct comparison of nano-SiO₂, nano-TiO₂ and nano-ZnO under the same concrete composition and experimental conditions for rigid pavement applications, making it difficult to determine their relative effectiveness and optimum incorporation ranges.

In response to this need, this study conducts a comparative analysis of the physical and mechanical performance and durability of concrete for rigid pavements modified with oxide nanoparticles, through the incorporation of nano-SiO₂, nano-TiO₂ and nano-ZnO as concrete additives.

The study focuses on quantifying their influence on flexural strength, which is the key parameter for pavement behaviour under repeated traffic loads, and on controlling cracking, capillary water ingress and progressive loss of stiffness, assessed through indicators sensitive to accumulated internal damage. Taken together, the results enable an assessment of the real potential of concrete nanomodification compared to conventional solutions and provide technical criteria for the design of rigid pavements with greater durability and improved structural performance in high-altitude Andean contexts.

2. Literature Review

In line with the preliminary observations outlined above, the specialist literature has conducted in-depth analyses of the behaviour of concrete pavements under freeze-thaw cycles through the development and application of both traditional and advanced strategies, specifically designed to mitigate the internal damage associated with the expansion of frozen water and the progressive degradation of the material's microstructure [5]. Among the most widely used are air-entraining additives (Figure 1(a)), which generate microbubbles and reduce internal stresses caused by the expansion of frozen water, particularly when an optimal air content (2–6%) is maintained [21, 22].

Furthermore, fibre reinforcement (Figure 1(b)) increases toughness and crack control, limiting the propagation of cracks associated with freeze damage [23], whereas PCMs incorporated into lightweight aggregates (Figure 1(c)) help to delay the formation of ice and improve thermal performance under real-world conditions [24].

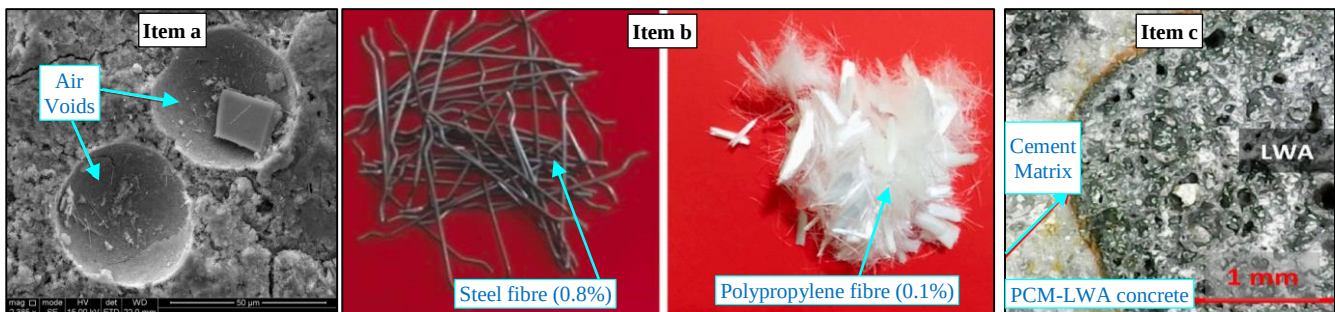


Fig. 1 Improvement strategies against freezing: (a) Air entrainment, (b) Fibres, and (c) PCM.

Conventional strategies, such as air entrainment, fibre reinforcement and phase-change materials, improve concrete's resistance to freeze-thaw cycles primarily by mitigating the effects of damage [16]. In contrast, the incorporation of oxide nanoparticles addresses the

microstructural cause of deterioration by modifying the cementitious matrix and the interfacial transition zone through nano-filling and nucleation effects [25]. This approach reduces porosity and transport properties, thereby limiting water ingress, the degree of saturation and the build-up of

internal pressures responsible for micro-cracking [26]. In rigid pavements, structural performance is primarily determined by the response to bending and fatigue, whilst durability against freeze-thaw cycles is governed by water transport and the microstructural stability of the matrix [27]. Under this approach, the results reported for concrete and mortar provide evidence that is directly applicable to paving, as the mechanisms of hydration, the formation of cementitious products, the refinement of the porous structure and the improvement of the ITZ are common to the material used in the paving slabs.

2.1. Nano-SiO₂ (Silicon Dioxide)

Nano-Silica (Nano-SiO₂) has been extensively studied as a high-performance additive in cementitious concrete and rigid pavements, due to its fine particle size and high chemical reactivity [28]. In this context, we present a review of recent studies that have examined their effect on the mechanical properties, durability and microstructure of concrete, as well as the optimal dosage ranges.

In Saudi Arabia, researchers at Najran University conducted a systematic review of the current state of the art regarding the use of nanosilica in cementitious concrete, with the aim of analysing how different types and proportions influence hydration, strength, durability and microstructure, as well as identifying the challenges to its commercial application. The study took the form of a review article, in which over 150 scientific publications were analysed, organising the evidence according to fresh-state behaviour, hydration processes, mechanical performance, durability and microstructural observations.

The results indicated that dosages in the range of 2–4% of nanosilica are optimal for improving the physical, mechanical and durability properties of concrete, densifying its microstructure through the pozzolanic reaction with Ca(OH)₂ and the formation of C-S-H gel, although technical and operational challenges were noted that limit its implementation on an industrial scale [29].

Researchers in Jordan have conducted a comprehensive review of the use of nanosilica in concrete and cementitious materials, with the aim of summarising its effects on mechanical properties and durability, including its influence on microstructure and the reduction of micropores. The study was designed as a review based on a detailed analysis of nearly one hundred scientific articles, organising the evidence around mechanical behaviour, performance against deteriorating agents and microstructural characterisation. The results indicated consistent increases in mechanical strength and durability, associated with the activation of cement hydration, the densification of the microstructure and the interfacial transition zone; however, practical limitations to its application were also identified, such as agglomeration problems, costs and dispersion difficulties, with sonication

being highlighted as an alternative to improve this aspect, and it was established that the recommended dosage of nanosilica generally lies between 2% and 3% of the cementitious content, depending on the type of cement [30].

Researchers at the Motilal Nehru National Institute of Technology, Allahabad (India) assessed the feasibility of using nanomaterials, specifically nano-silica, in concrete pavements, with the aim of identifying an effective method and an optimal dosage. The study was conducted using an indirect approach, based on a systematic review of scientific literature (2006–2024) obtained from databases such as Scopus, Web of Science, Google Scholar and Scimago.

A total of 231 publications were selected according to defined inclusion criteria and analysed using statistical and bibliometric techniques with VOSviewer. The results revealed a substantial body of international scientific literature and indicated that moderate increases in nano-silica improve the mechanical properties and durability of concrete by around 3%, whilst dosages exceeding 5% may have adverse effects; furthermore, it was observed that colloidal nano-silica exhibits a more homogeneous dispersion than powdered nano-silica [31].

Researchers from the School of Civil Engineering at the Kalinga Institute of Industrial Technology in India systematically reviewed the impact of various nanomaterials on the mechanical and durability properties of Pavement-Quality Concrete (PQC). Methodologically, the study employed qualitative and quantitative content analysis, drawing on 298 documents. The results showed that nano-SiO₂, at an optimal dosage of 2%, led to increases of up to 48.88% in compressive strength, 60.7% in flexural strength and 78.6% in indirect tensile strength; Graphene Oxide (GO), at an approximate dosage of 0.03%, showed comparable improvements in the mechanical properties of PQC; whilst nano-TiO₂, at a dosage of 1%, was associated with a 36.30% increase in compressive strength, improvements of over 100% in abrasion resistance and an increase of approximately 475% in fatigue performance [32].

2.2. Nano-TiO₂ (Titanium Dioxide)

Nanoscale Titanium Dioxide (Nano-TiO₂) has been studied as a functional nano-additive in cementitious concrete due to its ability to simultaneously improve mechanical strength and durability [33]. Based on this, representative studies evaluating the effect of nano-TiO₂ on the strength, durability and pore structure of concrete are presented below.

In China, researchers at the Yancheng Institute of Technology developed a photocatalytic concrete paving stone reinforced with TiO₂ nanotubes to evaluate its mechanical performance and its ability to remove NO. TiO₂ nanotubes were synthesised using a hydrothermal method and applied to the surface of C40 concrete, after which mechanical and

environmental tests were carried out. The results showed that the nanotube-reinforced paving stone achieved a compressive strength of 45.98 MPa, higher than the control concrete at 44.50 MPa and the concrete treated with TiO₂ P25 at 44.51 MPa. It also exhibited greater abrasion resistance, with a value of 3.85, compared to 3.57 for the control and 2.94 for the P25 system. In terms of environmental performance, the material achieved 50% efficiency in NO removal at a concentration of 1 ppm after 110 minutes of UV irradiation, exceeding the 35% achieved by the concrete with TiO₂ P25, and maintained stable behaviour with minimal variations in NO conversion before and after surface polishing [34].

In India, researchers at Jaypee University of Engineering and Technology evaluated the partial replacement of cement with nano-TiO₂ in C20/25 concrete, analysing its fresh, mechanical, durability and microstructural properties. Mixes were prepared with contents ranging from 0% to 3.0%, using STPP as a dispersant and curing the specimens in water at 27 °C. The tests showed that workability decreased significantly with increasing nano-TiO₂ content, reaching a reduction in slump of around 54% at 3.0%. Mechanical strengths showed maximum values of around 1.5%, with improvements compared to the reference concrete and decreases for higher replacement levels. In terms of durability, progressive reductions in water absorption, porosity, sorptivity and chloride penetration were observed, whilst UPV values exceeding 5000 m/s indicated good internal homogeneity. Microstructural analysis confirmed a denser matrix with fewer pores and microcracks [35].

In India, researchers at SRM University Delhi-NCR assessed the effect of nano-TiO₂ on M25 mortars with a 1:1:2 mix ratio, in which 20% of the cement was replaced by fly ash. Nano-TiO₂ was incorporated at levels of 1%, 2%, 3%, 4% and 5% by weight of the cement, using a polycarboxylate superplasticiser and cured at a temperature of approximately 27 °C. Compressive strength was tested at 7, 28 and 56 days in accordance with ASTM C109 and IS 4031, supplemented by FTIR and XRD analyses to evaluate the hydration products. The results showed an increase in strength up to an optimum content of between 2% and 3%, reaching a maximum value of 23.95 MPa at 56 days, whilst higher dosages reduced performance due to the agglomeration of nanoparticles. FTIR and XRD analyses confirmed this behaviour, showing greater formation of C-S-H gel and a lower presence of calcium hydroxide in the optimal mixtures [36].

In India, researchers at SVR Engineering College assessed the effect of nano-TiO₂ on a GGBS-based geopolymer concrete cured at room temperature. The ratio of alkaline activators was optimised, with an optimum value of 2.5 identified, and nano-TiO₂ was incorporated at levels between 1% and 5% relative to the GGBS, maintaining a water-to-binder ratio of 0.55. Mechanical strengths and durability were assessed using RCPT tests at 60 V for 6 hours,

water absorption, AVPV, sorptivity and chemical attacks with 5% MgSO₄ and NaCl solutions for 28 days, in addition to microstructural analysis. The results showed that the optimum nano-TiO₂ content was 3%, achieving a compressive strength of 81.03 MPa compared to 66.66 MPa for the reference geopolymer, with increases of 19.5% in flexural strength and 14.76% in indirect tensile strength. In terms of durability, lower absorption values were recorded, a reduction in AVPV from 2.794% to 2.26% and lower sorptivity, whilst for higher contents, between 4% and 5%, performance decreased due to nanoparticle agglomeration and microcracking [37].

2.3. Nano-ZnO (Zinc Oxide)

Nano-ZnO has been studied in cementitious materials due to its ability to modify hydration and microstructure, thereby influencing mechanical performance and durability; however, its effect depends on the dosage and method of incorporation [38]. Experimental evidence was therefore analysed regarding its impact on strength, setting and microstructural properties.

In Spain, researchers at the University of Oviedo assessed the effect of zinc oxide nanoparticles on lightweight concrete, comparing their incorporation as an additive and as a partial substitute for cement. Concentrations of 0%, 0.5%, 1.0%, 1.5% and 2.0% by weight were studied, resulting in nine mixtures comprising a reference concrete and eight mixtures containing ZnO. To this end, 54 cubes measuring 50 mm per side were produced, six per mixture, using silica sand and LECA-type lightweight aggregate, cured in water at temperatures between 20 and 22 °C for 35 days. It was observed that the presence of ZnO delayed the development of mechanical properties, requiring an additional 7 days to achieve structural performance. Compressive strength was determined in accordance with standard UNE-EN 12390-3, and the microstructure was analysed using SEM and EDX. The results showed that the reference lightweight concrete achieved a strength of around 30 MPa; when ZnO partially replaced the cement, the strength decreased significantly, with values below 20 MPa obtained for contents greater than 1.0%, whilst when incorporated as an additive, strengths above 20 MPa were maintained up to contents of 1.5% [39].

In Jordan, researchers at Ajloun National University assessed the effect of nano-ZnO on the hydration, microstructure, water response and setting of cementitious materials. Paste mixtures were formulated with nano-ZnO contents ranging from 0.0% to 3.0%, using a water-to-cementitious material ratio of approximately 0.29, and hydration was monitored from the start until 1440 minutes; Mortars were also prepared with a cement-to-sand ratio of approximately 0.33 and a water-to-cement ratio close to 0.50, partially replacing the cement with the same percentages. Vicat, slump, mechanical strength and characterisation tests were carried out using scanning electron microscopy and X-ray diffraction, supplemented by statistical analysis. The results indicated that nano-ZnO prolonged setting times and

modified early hydration; a dosage of around 2.0% produced a denser microstructure with an approximate 15% reduction in harmful porosity, whilst around 3.0% yielded the best mechanical performance, with an increase of nearly 20% in compressive strength, attributed to the nano-filling effect [40].

In Saudi Arabia, researchers at Najran University assessed the influence of Zinc Oxide Nanoparticles (N-ZnO), synthesised via the oxalate route, on the performance of lightweight foamed concrete. After obtaining crystalline nanoparticles with an average size of around 80 nm, six mixtures were prepared with a target density of 800 kg/m³, a water-cement ratio of around 0.5, a cement-sand ratio of between 1 and 1.5, and N-ZnO contents ranging from 0.0% to 1.0%. Fresh, mechanical, transport, thermal and porosity properties were analysed. The results showed that increasing the N-ZnO content reduced slump flow and setting times, with a slight increase in density, identifying an optimal dosage of around 0.6%. At 28 days, this dosage achieved increases of 70% in compressive strength, 82% in flexural strength and 84% in indirect tensile strength compared to the control; furthermore, sorptivity was reduced by 19%, water absorption by 23% and intrinsic permeability by 24%, whilst permeable porosity decreased from 57% to 51%. Microstructural analysis using SEM and MIP revealed a denser matrix and a higher proportion of micropores up to the optimal mix, confirming the better overall performance of the LWFC around that N-ZnO content [41].

In India, researchers at the Institute of Engineering and Technology assessed the effect of nanosilica and nano-zinc oxide on the mechanical and chemical performance of M25 concrete, as well as their interaction during C₃S hydration. ZnO and SiO₂ nanoparticles were synthesised using the sol-gel method, and seven mixtures were designed with a water-cement ratio of approximately 0.45, including a reference concrete and partial cement substitutions of 0.5%, 0.75% and 1.0%. Compression and indirect tensile tests were carried out at 7 and 28 days, and durability was assessed using acid attack and ultrasonic pulse velocity. The results showed that the optimal dosage for nanosilica was around 0.75%, with increases of approximately 16.5% in compressive strength and 19% in indirect tensile strength at 28 days; in the case of nano-ZnO, the best performance was obtained at around 0.5%, with reductions in strength observed at higher dosages. Furthermore, the mixtures containing nanoparticles exhibited better resistance to acid attack and, according to UPV, the concrete with nano-silica showed excellent quality, whilst the concrete with nano-ZnO tended towards moderate quality [42].

In recent years, the use of oxide nanoparticles in cementitious materials has attracted interest as a strategy for improving the strength and durability of concrete, particularly against water ingress and deterioration caused by freeze-thaw cycles. The studies mentioned above have demonstrated that

nanoparticles such as nano-SiO₂, nano-TiO₂ and nano-ZnO improve the microstructure of concrete through nano-filling effects and strengthening of the interfacial transition zone, leading to increases in strength and reductions in transport properties. However, these studies have been conducted using different methodologies and dosages, which makes it difficult to directly compare their performance.

In this context, it became clear that there was a need to carry out a comparative assessment of the use of oxide nanoparticles within a single experimental design specifically tailored to rigid pavements. This research analysed the incorporation of nano-SiO₂, nano-TiO₂ and nano-ZnO as additives to concrete for rigid pavements, dosed in relation to the cement mass, with a design strength of $f'_c = 280 \text{ kg/cm}^2$. Fresh-state behaviour was assessed in accordance with ASTM C143, and temperature was measured in accordance with ASTM C1064. Mechanical performance was determined through compressive strength in accordance with ASTM C39 and flexural strength in accordance with ASTM C78, the latter serving as a representative parameter of the pavement's structural behaviour under traffic loads. Prior to this, the suitability of the coarse aggregate was verified by means of the Los Angeles abrasion test in accordance with ASTM C131. Durability was assessed using freeze-thaw cycles in accordance with ASTM C666 (Method A), using the relative dynamic modulus of elasticity as an indicator of internal damage, whilst water transport was analysed using the sorptivity test in accordance with ASTM C1585. This experimental approach enabled the generation of evidence directly applicable to the design of rigid pavements, the identification of optimal dosage ranges, and the clarification of the functional role of each nano-oxide under wet service conditions and cyclic loading.

3. Materials and Methods

This section describes the experimental programme developed to evaluate the performance of a rigid concrete pavement modified with oxide nanoparticles. It presents the materials, the reference mix, and the incorporation of nano-SiO₂, nano-TiO₂ and nano-ZnO as concrete additives. The behaviour of the concrete was assessed in the fresh state through slump and temperature tests, and in the hardened state through compressive strength, flexural strength, freeze-thaw and sorptivity tests.

3.1. Standard Rigid Concrete Pavement

A plain concrete slab is a rigid structural system consisting of hydraulic concrete slabs without continuous reinforcement, supported on base, sub-base and subgrade layers as shown in Figure 2, whose structural performance is controlled by the arrangement of transverse and longitudinal joints that regulate cracking caused by shrinkage and thermal variations [43]. This type of pavement ensures functional continuity between adjacent slabs through load-transfer mechanisms, primarily expansion joints, which distribute

vehicle loads, reduce differential deflections and limit stress concentrations [44]. Its structural behaviour is governed by its response to traffic loads and thermal gradients, and is assessed in terms of bending stresses, deflection and fatigue damage, with the aim of ensuring the durability, geometric stability and service life of the pavement system [45].

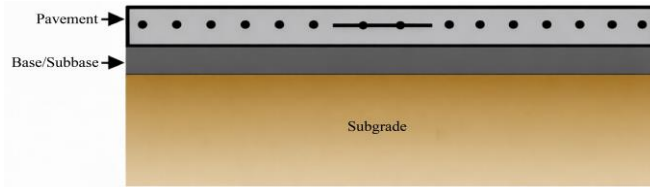


Fig. 2 Pavement design diagram [45]

3.2. Materials for the Rigid Pavement System

3.2.1. Concrete Mix Design

Conventional materials for hydraulic concrete were used to construct the plain concrete rigid pavement, including Portland cement, water, and fine and coarse aggregates [46], selected in accordance with the requirements set out in the current technical standards for concrete pavements, which are presented in Table 1. The concrete strength was determined by considering the pavement's design traffic level and the recommended values for rigid pavements, adopting a compressive strength of 280 kg/cm², corresponding to a heavy traffic class [47]. In this context, the fine aggregate consisted of natural coarse sand from the Mito Orcotuna quarry, whilst the coarse aggregate consisted of crushed stone with a maximum nominal size of ¾ in, extracted from the Ataura Jauja quarry.

Table 1. Mix design for concrete with $f'_c = 280 \text{ kg/cm}^2$

Material	Unit	Value
Water–cement ratio	-	0.38
Portland cement	kg	566.95
Coarse sand	kg	693
Crushed stone	kg	732
Water	L	189

The mix proportions shown in Table 1 were adopted as the control mix; experimental mixes were formulated on this

basis by adding Nano-SiO₂, Nano-TiO₂ and Nano-ZnO, expressed as a percentage by weight of the cement, whilst keeping the water-to-cement ratio and the aggregate content constant.

3.2.2. Quality of Coarse Aggregate

In order to assess the quality and suitability of the coarse aggregate used in the construction of plain concrete rigid pavement, the Los Angeles abrasion test was carried out in accordance with ASTM C131-06, which determines the aggregate's resistance to abrasion, impact and fragmentation under mechanical stress [48]. This test is particularly relevant for pavements subjected to heavy traffic, as coarse aggregate is the component of concrete that governs its behaviour in terms of wear and mechanical degradation [49]. In this study, the test was applied exclusively to coarse crushed stone aggregate with a maximum nominal size of ¾ in.

Los Angeles Abrasion Test Procedure

The Los Angeles abrasion test was carried out in accordance with the procedure set out in ASTM C131, as shown in Figure 3. Firstly (Item a), a representative sample of coarse crushed stone aggregate with a maximum nominal size of ¾ in was obtained; this was washed and oven-dried at 110 °C until a constant weight was reached. Subsequently (Item b), the material was fractionated by sieving and recombined in accordance with Gradation B as established by the standard, preparing a total sample of 5000 g, comprising 2500 g of material passing the 19 mm (¾ in) sieve and retained on the 12.5 mm (½ in) sieve, and 2500 g of material passing the 12.5 mm (½ in) sieve and retained on the 9.5 mm (¾ in) sieve. The prepared sample was placed in the Los Angeles machine (Item c), together with a load consisting of 11 steel balls, each with an individual mass of between 390 g and 445 g, giving a total load mass of 4584 g, as specified for that gradation. The machine drum was rotated at a constant speed of 33 rpm, completing a total of 500 revolutions. Once the rotation cycle was complete (Item d), the material was discharged and sieved on sieve No. 12, separating the fine fraction generated during the test. The retained material was washed, dried again in an oven to constant weight and weighed, yielding a final mass of 3851 g.



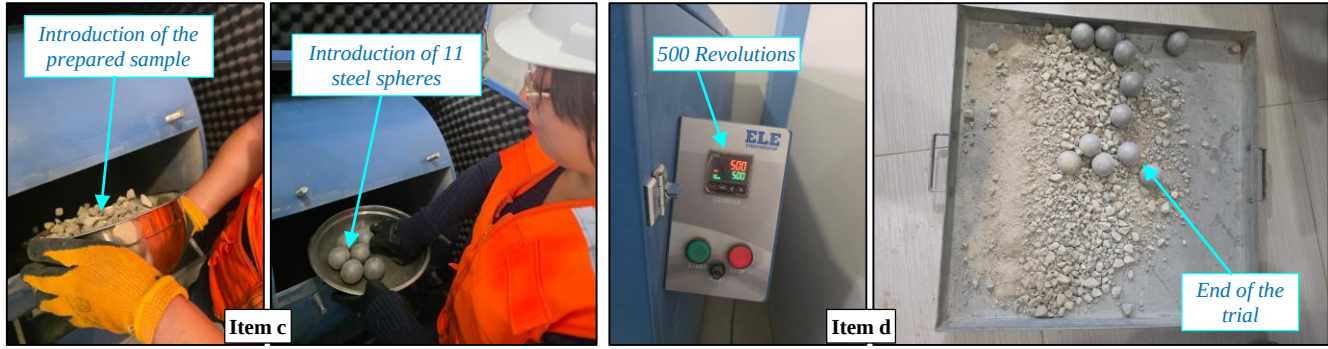


Fig. 3 Los Angeles wear test procedure

Based on these values and using Equation 1, as detailed in ASTM C131-06 [48], a loss due to attrition of 23% was determined for Los Angeles, which is below the maximum limit of 40% established for coarse aggregates intended for hydraulic concrete pavements, in accordance with the requirements of MTC Table 438-06 [50], thus confirming the suitability of the stone material for use in this study.

$$LA \text{ Loss (\%)} = \frac{M_{\text{initial}} - M_{\text{final}}}{M_{\text{initial}}} * 100 \quad (1)$$

3.3. Nanoparticles Incorporated into Concrete

Nanoparticles are solid materials whose characteristic dimensions are on the order of nanometres and which, due to their high specific surface area and reactivity, can modify the hydration processes of cement, the microstructure of the matrix and the properties of hardened concrete [51]. In this study, three nanoparticles were selected: Silicon Dioxide (Nano-SiO₂), Nanoscale Titanium Dioxide (Nano-TiO₂) and Zinc Oxide (Nano-ZnO).

3.3.1. Nano-SiO₂ (Silicon Dioxide)

Nano-SiO₂ is an inorganic nanomaterial consisting of particles up to 50 nm in size [52]. Microstructural characterisation using Scanning Electron Microscopy (SEM), as shown in Figure 4, reveals that the incorporation of nano-SiO₂ results in a denser and more homogeneous matrix, with fewer voids and better continuity of the cementitious products, consistent with the improvements observed in mechanical performance and durability. This microstructural densification results in lower water absorption and permeability of the material [53].

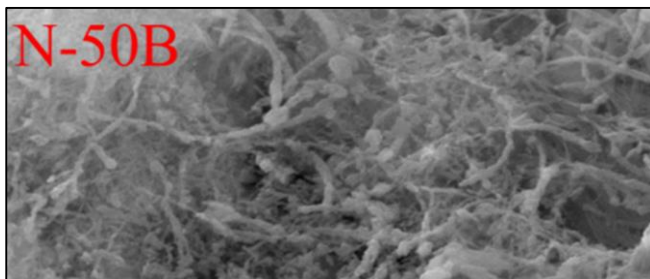


Fig. 4 SEM characterisation of Nano-SiO₂ [52]

According to the literature review, the optimal dosage of nano-SiO₂ is consistently found to be around 3% of the cementitious content, thereby maximising improvements in the mechanical performance, microstructural densification and durability of concrete, particularly in rigid pavement applications [29, 30, 52]. In this context, with the aim of experimentally validating the information reported in the literature, this study evaluated nano-SiO₂ contents of 1%, 2%, 3%, 4% and 5% as an additive to concrete, dosed in relation to the mass of cement.

Preparation of Silicon Dioxide

Silicon dioxide nanoparticles were prepared using a sol-gel process based on sodium silicate, following the methodological sequence outlined in Figure 5. Firstly (Item a), the precursor solutions were prepared using 0.5 M sodium silicate and hydrochloric acid as the acidifying agent. For the titrant solution, a hydrochloric acid dilution was prepared using 8.3 mL of HCl in 100 mL of distilled water, in order to ensure gradual control during neutralisation. Secondly (Item b), the sodium silicate solution was placed under constant stirring, and the initial pH of the system (11.8) was reduced by slowly adding the HCl solution until a final pH close to 7.1 was reached, with the pH monitored continuously using a digital potentiometer; this condition is associated with the formation of the gel characteristic of the sol-gel process.

Thirdly (Item c), once the target pH had been reached, the system underwent a maturation stage to promote gel formation and stabilisation. This stage was carried out by leaving the system to stand at room temperature for 12 hours. The temperature of the system was monitored using a thermocouple immersed in the sample, maintaining the programmed operating value. Finally (item d), the gel obtained was filtered through filter paper, followed by successive washes with distilled water until the pH of the filtrate stabilised in the range of 7 to 8. Subsequently, the retained material was dried in an oven, using a maximum temperature of 120 °C, and adjusting the drying time according to the quantity and thickness of the sample, with the aim of achieving uniform drying. Once dry, the final solid corresponding to silicon dioxide was obtained.

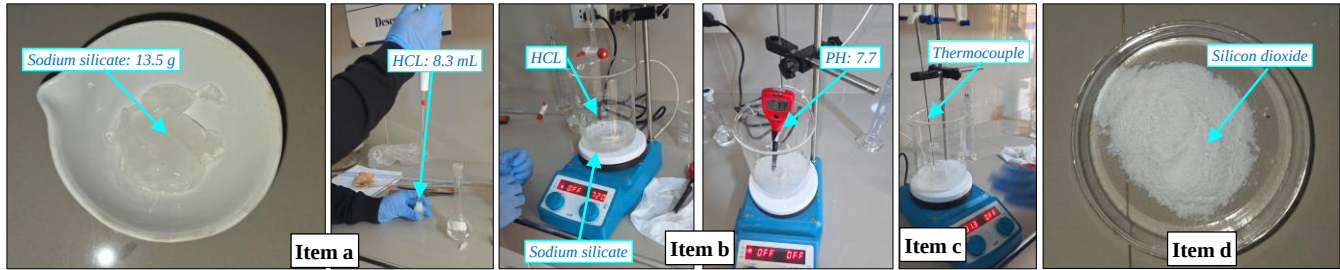


Fig. 5 Production of Silicon Dioxide

3.3.2. Nano-TiO₂ (Titanium Dioxide)

Nano-TiO₂ is an inorganic nanomaterial, usually in powder form, with particle sizes of around 40 nm [54]. Microstructural characterisation using Scanning Electron Microscopy (SEM/FESEM) and complementary analyses such as XRD and FTIR has shown that the incorporation of nano-TiO₂ promotes the formation of a denser and more compact matrix, with refinement of the pore structure and improvement of the Interfacial Transition Zone (ITZ), as shown in Figure 6, in line with the observed increases in mechanical properties and the reduction in concrete permeability [55, 56].

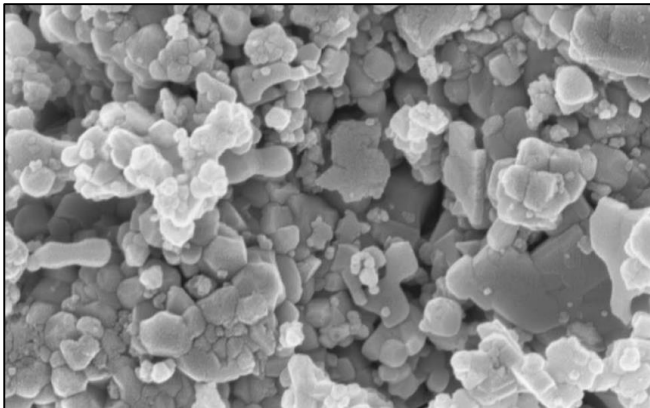


Fig. 6 SEM characterisation of Nano-TiO₂ [54]

An analysis of the specialist literature indicates that the most appropriate dosage of nano-TiO₂ is 2% of the cementitious content, a range within which the greatest benefits are achieved in terms of mechanical strength,

microstructural compaction and the durability of the concrete [35-37]. Based on this criterion, and with the aim of experimentally verifying the reported trends, this study considered nano-TiO₂ dosages of 0.5%, 1%, 1.5%, 2% and 2.5% as an additive to concrete, calculated relative to the mass of cement.

Preparation of Titanium Dioxide

Titanium dioxide nanoparticles were prepared by controlled precipitation from titanyl sulphate, following the methodological sequence outlined in Figure 7. Firstly (item a), the precursor solutions were prepared using 0.3 M titanyl sulphate and 1 M sodium hydroxide. The mass of titanyl sulphate was determined using an analytical balance, recording a weight of 4.8 g for the preparation of the corresponding solution. Secondly (item b), the titanyl sulphate solution was placed under constant stirring, with an initial pH of approximately 4 recorded. Subsequently, the sodium hydroxide solution was added gradually, titrating until a pH of 9 was reached, with the value monitored in real time using a digital potentiometer, a condition under which the formation of the precipitate is favoured. Thirdly (item c), once the target pH had been reached, the suspension was kept under agitation to ensure homogenisation of the system and stabilisation of the solid formed, resulting in a whitish dispersion characteristic of the precipitate. Finally (item d), the solid obtained was separated by filtration and subjected to successive washes with distilled water until the pH of the filtrate stabilised at 8. Subsequently, the retained material was oven-dried at controlled temperatures not exceeding 120 °C until the dry solid corresponding to the TiO₂ nanoparticles was obtained.

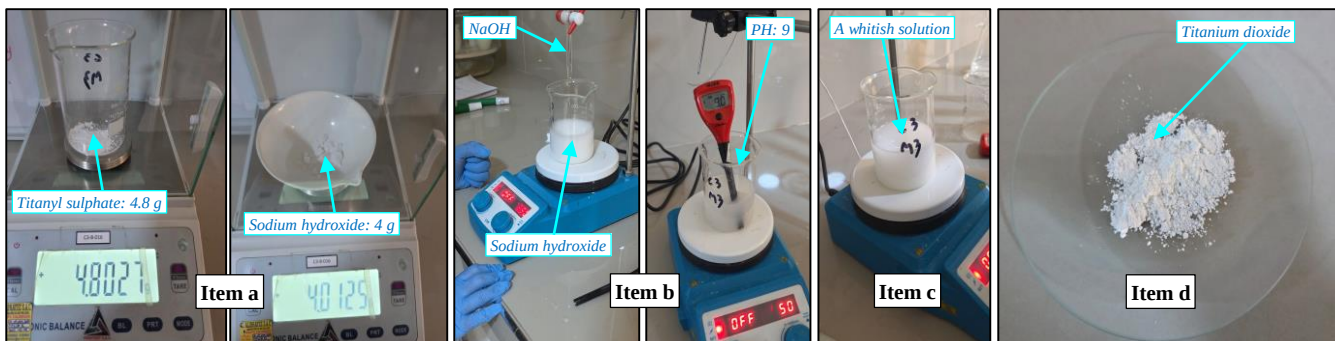


Fig. 7 Production of Titanium Dioxide

3.3.3. Nano-ZnO (Zinc Oxide)

Zinc oxide is a functional nanomaterial characterised by its high specific surface area and a particle size typically less than 50 nm, which promotes intense interaction with the cementitious matrix from the earliest stages of hydration [57]. Unlike other nano-oxides, nano-ZnO exhibits dual physicochemical activity, acting both as a nanoscale filler and as a modifier of stress transfer mechanisms within the cementitious material [58]. Microstructural analysis of cementitious matrices containing nano-ZnO reveals a more homogeneous distribution of the solid phase, with lower capillary porosity and a strengthened cement-aggregate interface, which improves paste-aggregate adhesion and leads to a more stable mechanical response under complex loads [40], as shown in Figure 8.

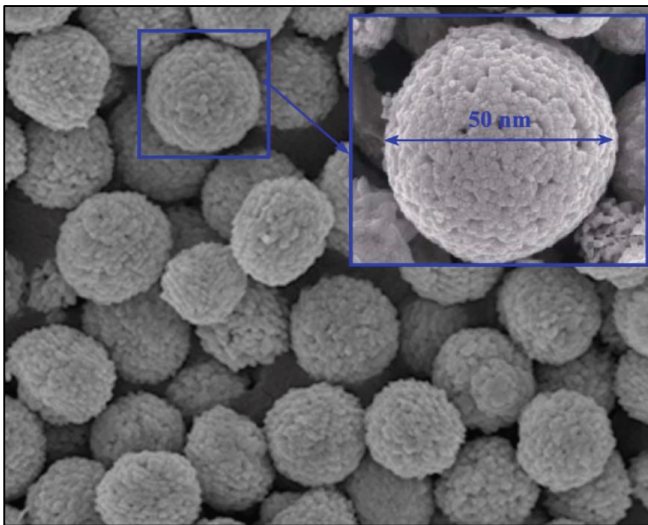


Fig. 8 SEM characterisation of nano-ZnO [57]

Based on a review of the literature, it has been found that the optimal performance of nano-ZnO in cementitious materials is generally achieved at dosages of 0.5% and 0.6%

by weight of cement; within this range, simultaneous improvements in mechanical strength, microstructural densification and reduced transport properties have been reported, without any significant adverse effects on workability or strength development [41, 42]. In line with this evidence and with the aim of experimentally assessing its influence, this study employed nano-ZnO dosages of 0.3%, 0.4%, 0.5%, 0.6% and 0.7%, incorporated as an additive to the concrete.

Preparation of Zinc Oxide

The zinc oxide nanoparticles were obtained using a controlled precipitation method from precursor solutions, following the methodological sequence outlined in Figure 9. Firstly (Item a), the precursor solutions were prepared at a concentration of 1 M, using zinc sulphate and sodium hydroxide as reagents. The quantities of solid reagents were determined using an analytical balance, weighing approximately 28.75 g of zinc sulphate and 4 g of sodium hydroxide, which were dissolved separately in distilled water until the required volume was reached.

Secondly (item b), the zinc sulphate solution was placed under constant stirring, and the sodium hydroxide solution was then added gradually drop by drop. During this process, the pH of the suspension was continuously monitored using a digital potentiometer until a value close to 8.8 was reached, a condition necessary for the formation of the precipitate. Thirdly (item c), once the target pH had been reached, the suspension was kept under constant stirring for a 1-hour ageing period, in order to promote controlled particle aggregation and facilitate subsequent solid-liquid separation. Finally (item d), the precipitate obtained was separated by filtration and subjected to successive washes with distilled water until the pH of the filtrate stabilised in the range of 8. Subsequently, the solid was dried in an oven at controlled temperatures, ultimately yielding the powder corresponding to the zinc oxide nanoparticles.

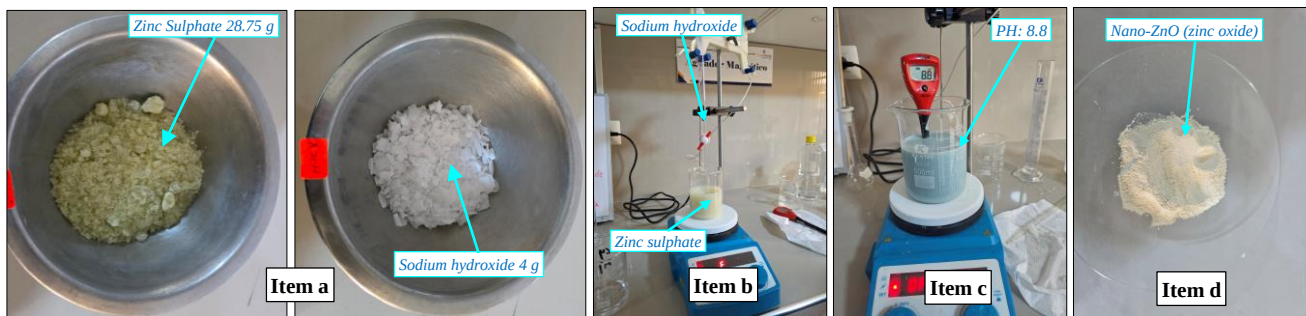


Fig. 9 Production of Zinc Oxide

3.4. Dosages of Nanoparticles for Concrete

Table 2 shows the dosage ranges for nano-SiO₂, nano-TiO₂ and nano-ZnO used in the study. These levels were determined in proportion to the cement content of the base

mixture, taking into account the different reactivity of each nanoparticle, in order to evaluate, under controlled conditions, the influence of the type and dosage on the performance of the concrete.

Table 2. Nanoparticle dosages

Material	Dosage (%)	ID	Quantity (kg)
Nano-SiO ₂	1%	S ₁	5.670
Nano-SiO ₂	2%	S ₂	11.339
Nano-SiO ₂	3%	S ₃	17.009
Nano-SiO ₂	4%	S ₄	22.678
Nano-SiO ₂	5%	S ₅	28.348
Nano-TiO ₂	0.5%	T ₁	2.835
Nano-TiO ₂	1%	T ₂	5.670
Nano-TiO ₂	1.5%	T ₃	8.504
Nano-TiO ₂	2%	T ₄	11.339
Nano-TiO ₂	2.5%	T ₅	14.174
Nano-ZnO	0.3%	Z ₁	1.701
Nano-ZnO	0.4%	Z ₂	2.268
Nano-ZnO	0.5%	Z ₃	2.835
Nano-ZnO	0.6%	Z ₄	3.402
Nano-ZnO	0.7%	Z ₅	3.969

3.4.1. Incorporation into Concrete

Once the nanoparticle dosages indicated in Table 2 had been determined, the concrete mixtures were prepared following the methodological sequence shown in Figure 10.

Item (a) describes the preparation and individual dosing of the nanoparticles used, which were previously weighed in the dry state and kept in separate containers to ensure accuracy in their addition relative to the cement weight and to prevent cross-contamination between materials.

Subsequently, Item (b) shows the direct incorporation of the nanoparticles into the fresh concrete during the mixing stage, with their addition carried out gradually and distributed over the cementitious matrix, followed by controlled manual mixing, with the aim of promoting homogeneous dispersion and minimising the formation of agglomerates.



Fig. 10 Procedure for incorporating nanoparticles

3.5. Testing Programme

3.5.1. Fresh Concrete

Fresh concrete was characterised using standardised workability and temperature tests, as shown in Figure 11. Workability was assessed using the slump test in accordance with ASTM C143 [59], which allows the consistency, cohesion and workability of concrete to be estimated before setting begins [60], whilst also being sensitive to changes in the water-to-cement ratio, the characteristics of the aggregates and the incorporation of modifying agents; furthermore, the standard cone serves as a geometric reference used in the assessment of the flowability of highly workable mixes [61]. The procedure involved placing the metal cone on a rigid, horizontal, non-absorbent base that had been previously moistened; it was then filled with fresh concrete in three

layers, each compacted with 25 blows using a 5/8" steel rod. After levelling the upper surface, the mould was removed vertically and without rotation in a continuous motion lasting approximately 5s, and the vertical difference between the original height of the mould and the highest point of the settled concrete was immediately measured and recorded in millimetres (Item a). The temperature of the fresh concrete was determined in accordance with ASTM C1064 [62], given that this variable directly influences hydration, workability, setting time and strength development [63, 64]. To this end, the internal temperature was measured in a representative sample of the freshly mixed concrete using a digital thermometer, inserted to a sufficient depth to ensure thermal contact; the instrument was kept immersed until the reading stabilised and the corresponding value was recorded (Item b).

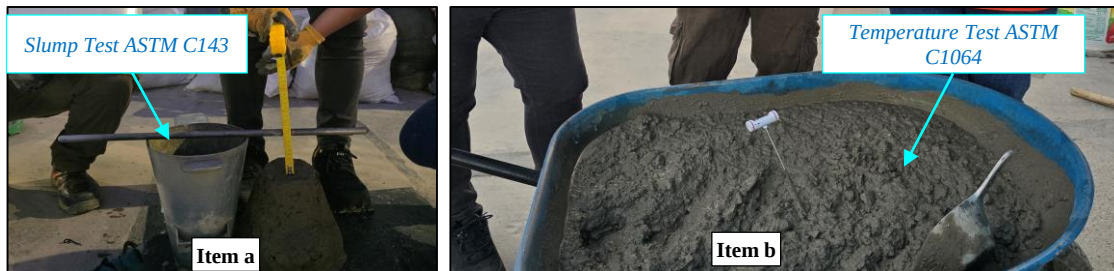


Fig. 11 Slump and temperature test procedures

3.5.2. Hardened Concrete

Compressive Strength ASTM C39

The concrete compressive strength test was carried out in accordance with ASTM C39 [65], with the aim of determining the load-bearing capacity of hardened concrete under axial compressive loads, a fundamental mechanical parameter for assessing its structural performance [66].

This test allows for the analysis of the influence of the mix design, curing conditions and the incorporation of modifying materials, and is also a widely used criterion for concrete quality control [67].

In this study, cylindrical specimens measuring 100 mm × 200 mm were tested, cured and evaluated at 7, 14 and 28 days, by applying a continuous and controlled axial load until failure. The compressive strength was determined from the maximum recorded load and the cross-sectional area of the specimen, as illustrated in Figure 12.

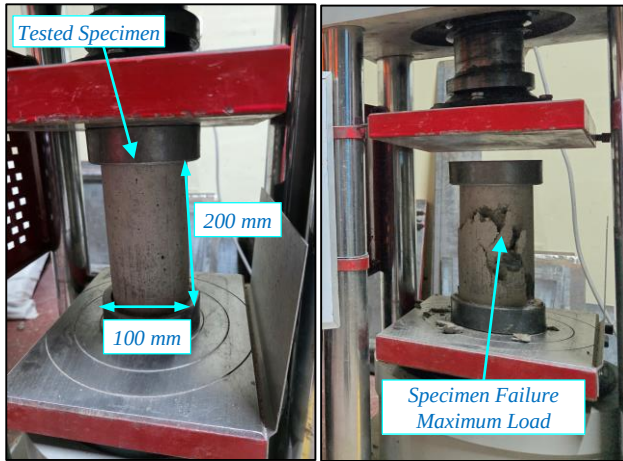


Fig. 12 Compressive strength test procedure

Flexural Strength ASTM C78

The concrete flexural strength test was carried out in accordance with ASTM C78 [68], with the aim of determining the Modulus of Rupture (MOR) of hardened concrete by applying a load at a third of the span, a parameter representative of its resistance to tensile stresses caused by bending [69].

This test is particularly relevant for elements subjected to bending stresses, as the MOR allows the behaviour of concrete during initial cracking to be assessed and enables the effect of design and material variables on the flexural response to be compared [70].

In this study, prismatic specimens measuring 150 × 150 × 550 mm, with an effective span of 450 mm, were tested and evaluated at 7, 14 and 28 days, with the load applied continuously and in a controlled manner until failure. The procedure used is shown in Figure 13.

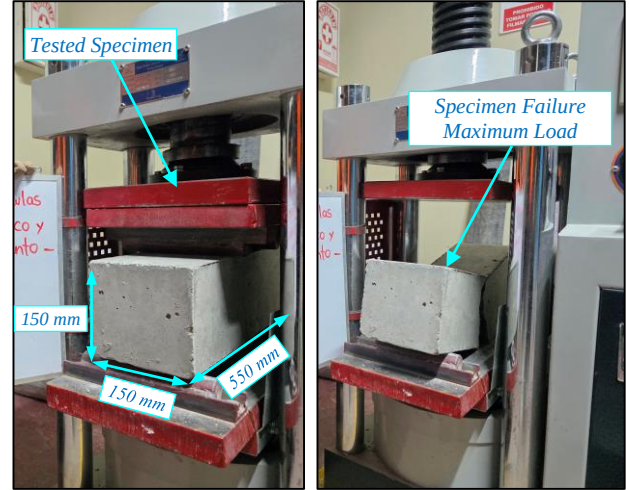


Fig. 13 Flexural strength test procedure

Freeze-Thaw Testing of Concrete – ASTM C666

The concrete freeze-thaw test, in accordance with ASTM C666 [71], is an experimental method designed to assess the durability of concrete against repeated freeze-thaw cycles, simulating severe cold weather conditions that can cause progressive damage due to water expansion within the porous network, microcracking and loss of material integrity [72]. This procedure allows deterioration to be quantified primarily through the reduction in the relative dynamic modulus, determined from resonant frequency measurements, and may be supplemented, depending on the experimental approach adopted, with the assessment of mass variations, dimensional changes or other non-destructive methods, providing an objective basis for comparing mixes and manufacturing conditions [73].

Furthermore, the standard sets out exposure methods, with Method A (freezing and thawing in water) being one of the most widely used, and constitutes a widely used benchmark for verifying the performance and compatibility of materials when concrete is subjected to sub-zero thermal cycling environments, particularly in applications such as slabs and pavements, where surface durability and resistance to freeze-thaw deterioration are critical [74]. In order to objectively quantify the progressive deterioration of concrete subjected to repeated freeze-thaw cycles, the performance of the mixtures was assessed using the Relative Dynamic Modulus of Elasticity (RDME), a non-destructive parameter recommended by ASTM C666 for monitoring internal damage to the material [71], which is determined in accordance with the mathematical expression shown in Equation (2).

$$RDME (\%) = \left(\frac{f_n}{f_0} \right)^2 * 100 \quad (2)$$

Where f_0 is the initial resonance frequency of the specimen (cycle 0), and f_n is the resonance frequency after n freeze-thaw cycles.

ASTM C666 Test Procedure

In this study, the concrete freeze-thaw test procedure was carried out in accordance with ASTM C666 (Method A), as shown in Figure 14, where: (a) the $75 \times 100 \times 400$ mm prismatic concrete specimens were cured in lime-saturated water at a temperature of 23°C for 14 days, and subsequently kept fully submerged in water to ensure complete saturation prior to the start of the test; (b) once saturated, the specimens were placed in a programmable freeze-thaw chamber, where they were subjected to repeated thermal cycles, consisting of a freezing phase at -18°C and a thawing phase in water at 4°C , in accordance with the ranges established in the

standard; (c) each cycle was carried out continuously and in a controlled manner, lasting 3 hours per cycle, with temperature and saturation conditions maintained throughout the process; (d) at predetermined intervals of 30 cycles [71], the specimens were temporarily removed from the chamber for the non-destructive determination of the dynamic modulus, by measuring the resonant frequency, and subsequently returned to the chamber to continue the test; and (e) the procedure was repeated until 300 freeze-thaw cycles had been completed, enabling the assessment of the concrete's resistance to progressive damage induced by severe thermal cycles.

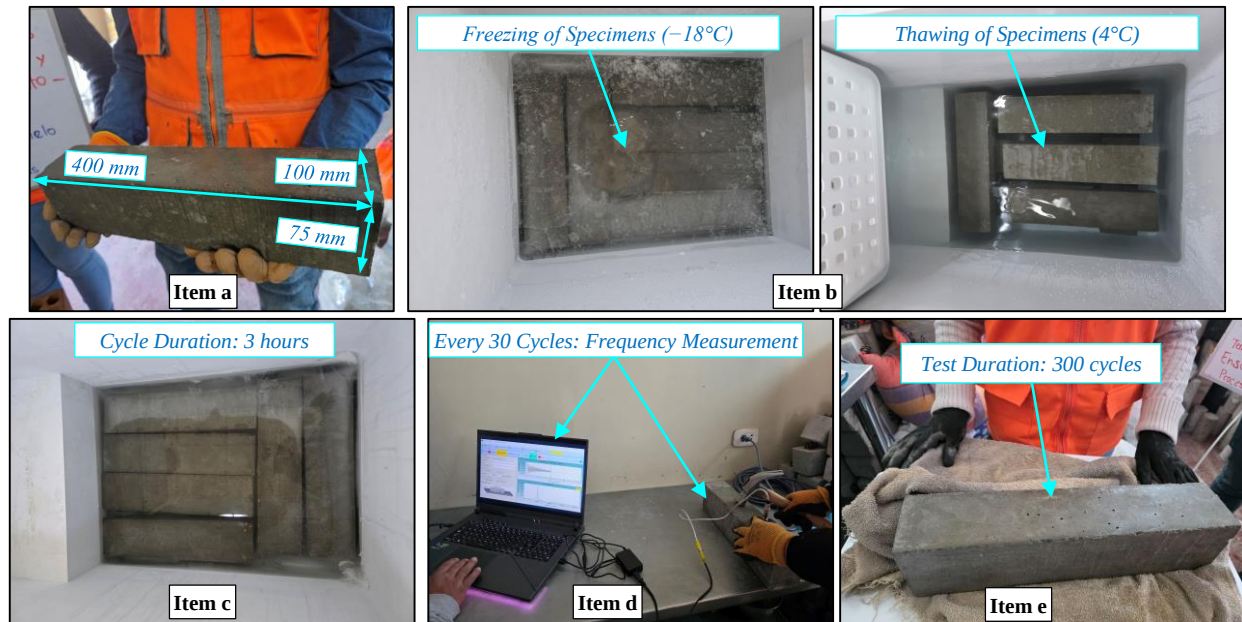


Fig. 14 Freeze-Thaw test procedure of concrete

ASTM C1585 Sorptivity Test

The concrete sorptivity test, in accordance with ASTM C1585 [75], is an experimental method designed to evaluate the capacity of hardened concrete to absorb water by capillary action, providing an indirect measure of the connectivity and fineness of the material's surface pore network [76]. This procedure allows the initial and secondary capillary absorption rates to be quantified based on the change in mass of the specimen over time, serving as a representative indicator of moisture transport and the potential ingress of aggressive agents, such as chloride ions, which affect the durability of concrete [77].

ASTM C1585 Test Procedure

In this study, the concrete sorptivity test procedure was carried out in accordance with ASTM C1585, as shown in Figure 15, where: (a) the test specimens consisted of cylindrical concrete discs 100 mm in diameter and 100 mm thick, obtained from hardened concrete cylinders, which were preconditioned by oven drying at a temperature of 50°C , maintained until a constant mass was reached, in order to

remove the free moisture present in the porous network; subsequently, the specimens were cooled to ambient temperature inside a desiccator to prevent moisture reabsorption; (b) once conditioned, the specimens were sealed on all their lateral surfaces and on the top surface using a waterproof material (SikaTop-107 Seal PER) [78], leaving only one circular face exposed, with the aim of ensuring that water ingress occurred exclusively via unidirectional capillary absorption; (c) the prepared specimens were placed on supports within suitable containers, ensuring that the exposed face was in contact with a 3 mm-deep water film, a condition that was maintained throughout the test; (d) during the exposure period, the change in mass of the specimens was recorded at pre-established time intervals, covering the initial and secondary stages of absorption, and the cumulative water absorption per unit of exposed area was calculated as a function of the square root of time; and, based on the slope of the resulting curve, the initial and secondary sorptivity were determined, enabling the characterisation of the concrete's capillary water transport behaviour and a comparison of the performance of the different mix designs evaluated.



Fig. 15 Sorptivity test procedure

4. Results

4.1. Slump and Temperature

Table 3 presents the results for the slump and temperature of the fresh concrete for the control mix and the nanoparticle-modified mixes. The control mix, without the addition of nanoparticles, achieved a slump of 12.07 cm and a recorded temperature of 23.1 °C, consistent with normal mixing conditions and without additional thermal effects. In the mixtures containing nano-SiO₂, incorporated at dosages of 1%, 2%, 3%, 4% and 5%, the slump decreased progressively to 11.52 cm, 10.80 cm, 10.16 cm, 9.84 cm and 9.52 cm, respectively. This reduction was attributed to the high specific surface area of nano-silica, which increased the demand for free water and promoted early pozzolanic reactions with calcium hydroxide, generating a denser matrix and reducing the workability of the fresh concrete. In terms of temperature, the highest value within this group was 24.2 °C for the mixture containing 4% nano-SiO₂, which was associated with increased hydration activation due to the nucleating effect. In the mixtures containing nano-TiO₂, incorporated at dosages of 0.5%, 1%, 1.5%, 2% and 2.5%, the slump values were 11.75 cm, 11.11 cm, 10.48 cm, 9.94 cm and 9.47 cm, indicating a progressive loss of workability associated with the nano-filler effect and the compaction of the cementitious matrix. The maximum temperature in this group was 23.9 °C for the mixture containing 1.5% nano-TiO₂, attributable to higher surface energy and activation of the hydration processes, whereas lower values were recorded at higher dosages. In the case of nano-ZnO, incorporated at dosages of 0.3%, 0.4%, 0.5%, 0.6% and 0.7%, an initial increase in slump was

observed, reaching 12.70 cm at 0.3% and 12.17 cm at 0.4%. This behaviour was associated with the initial retarding effect of ZnO on cement hydration, which favoured the fluidity of the system. However, as the dosage increased, the slump decreased to 11.43 cm, 10.52 cm and 10.04 cm, reflecting the predominance of the filling effect and the increase in internal friction. The maximum temperature was recorded in the mixture with 0.6% nano-ZnO (24.1 °C), consistent with controlled activation of hydration and greater densification of the paste.

Table 3. Slump and temperature results

ID	Slump (cm)	Temperature (°C)
Control	12.07	23.1
S ₁	11.52	22.5
S ₂	10.80	23.7
S ₃	10.16	22.8
S ₄	9.84	24.2
S ₅	9.52	22.6
T ₁	11.75	23.4
T ₂	11.11	22.9
T ₃	10.48	23.9
T ₄	9.94	22.4
T ₅	9.47	23.6
Z ₁	12.70	23.3
Z ₂	12.17	22.7
Z ₃	11.43	23.8
Z ₄	10.52	24.1
Z ₅	10.04	22.3

Figure 16 compares the slump and fresh concrete temperature values of the mixtures containing nanoparticles with those of the standard mixture (12.07 cm and 23.1 °C). For nano-SiO₂, the highest slump within its group was recorded at 1% (11.52 cm), corresponding to a 4.6% reduction compared to the standard, whilst the poorest performance was observed at 5% (9.52 cm), with a reduction of 21.1%, demonstrating that the increase in ultrafine particles intensified the water demand and stiffened the mixture; in terms of temperature, the maximum occurred at 4% (24.2 °C) with an increase of 4.8%, and the minimum at 1% (22.5 °C) with a reduction of 2.6%, reflecting moderate thermal variations associated with the activation/adjustment of hydration. For nano-TiO₂, the greatest slump occurred at 0.5% (11.75 cm) with a reduction of 2.7%, whilst the minimum slump and also the most critical

case overall was reached at 2.5% (9.47 cm) with a reduction of 21.5%, confirming the loss of workability at high contents; in terms of temperature, the maximum was obtained at 1.5% (23.9 °C) with an increase of 3.5%, and the minimum at 2% (22.4 °C) with a reduction of 3.0%. For nano-ZnO, the highest slump occurred at 0.3% (12.70 cm) with an increase of 5.2% compared to the standard (the highest favourable variation in the set), whilst the minimum was recorded at 0.7% (10.04 cm) with a reduction of 16.8%, showing that the initial benefit was reversed as the dosage increased; in terms of temperature, the maximum was observed at 0.6% (24.1 °C) with an increase of 4.3%, and the minimum at 0.7% (22.3 °C) with a reduction of 3.5%, which demonstrated that the thermal variations remained limited and were influenced by the interaction of ZnO with the hydration kinetics.

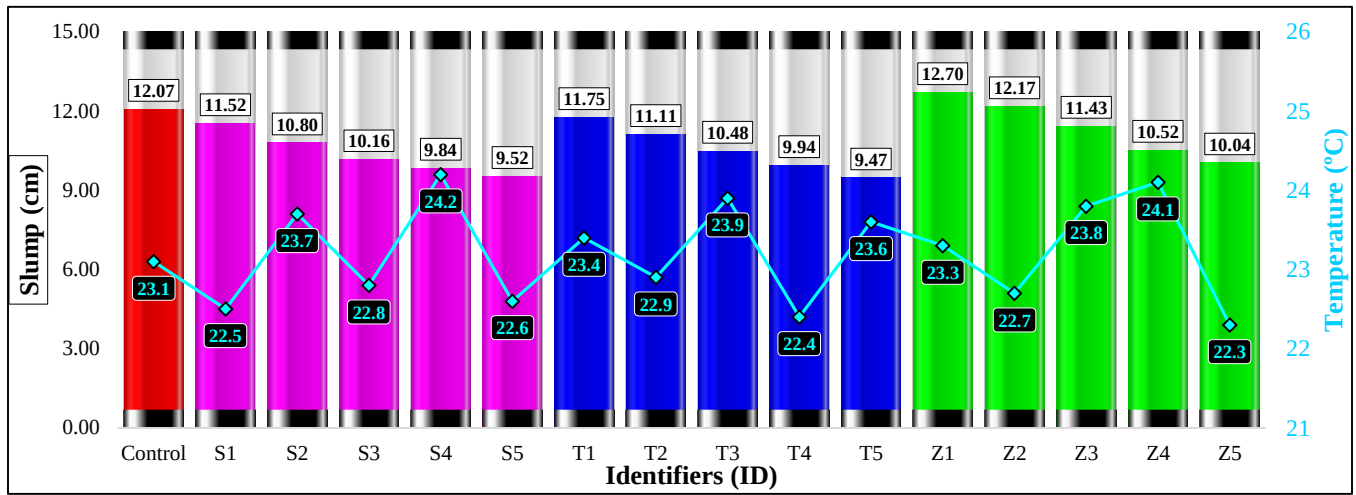


Fig. 16 Behaviour of fresh concrete: slump and temperature

4.2. Compressive Strength

Table 4 presents the compressive strength (F'c) results of the concrete at 28 days for the standard mix and for the modified mixes, incorporated as an additive to the concrete. The standard mix achieved 297.23 kg/cm², a value associated with the typical strength development of a conventional cementitious matrix at the final test age. In the case of nano-SiO₂, compressive strengths of 356.58 kg/cm² were recorded when 1% nano-SiO₂ was incorporated, 364.19 kg/cm² with 2%, 379.41 kg/cm² with 3%, 371.12 kg/cm² with 4% and 343.86 kg/cm² with 5%, with peak performance observed at 3% nano-SiO₂; this behaviour was explained by the combined action of the nanometric filler effect, the nucleation of hydration products and the pozzolanic reaction of the nano-silica, which consumed Ca(OH)₂ and promoted greater formation of C-S-H gel, refining the porosity and increasing the continuity of the matrix. The decrease observed when the content was increased to 4% and 5% nano-SiO₂ was interpreted as a loss of reinforcement efficiency, attributable to nanoparticle agglomeration, a higher effective water demand and the presence of areas with less homogeneous dispersion, which limited the additional gain in strength. For

nano-TiO₂, the compressive strengths at 28 days were 316.02 kg/cm² with 0.5% nano-TiO₂, 324.68 kg/cm² with 1%, 331.93 kg/cm² with 1.5%, 348.84 kg/cm² with 2% and 340.11 kg/cm² at 2.5%, showing a maximum at 2% nano-TiO₂, consistent with an improvement due to paste densification and strengthening of the ITZ associated with the ultrafine filler and the nucleating effect; however, upon exceeding this dosage, the slight reduction observed at 2.5% was consistent with agglomerations and local defects that reduced the efficiency of the microstructural reinforcement. In the case of nano-ZnO, strengths of 305.14 kg/cm² were obtained with 0.3% nano-ZnO, 311.47 kg/cm² with 0.4%, 320.55 kg/cm² with 0.5%, 335.92 kg/cm² with 0.6% and 327.73 kg/cm² with 0.7%, with peak performance identified at 0.6% nano-ZnO; this result was attributed to a favourable balance between the nanoscale filler and the improvement in the internal continuity of the matrix, whilst the decrease observed when the content was increased to 0.7% was consistent with a more pronounced effect of ZnO on the hydration kinetics and/or a greater propensity for agglomeration at high contents, reducing the net strength gain at 28 days.

Table 4. Compressive strength results

ID	Compressive Strength F'c Design – 280 (kg/cm ²)			% Variation with respect to Control at 28 Days
	7 days	14 days	28 days	
Control	204.31	255.52	297.23	-
S ₁	258.48	309.41	356.58	19.97%
S ₂	265.99	316.60	364.19	22.53%
S ₃	280.50	329.63	379.41	27.65%
S ₄	273.60	321.93	371.12	24.86%
S ₅	245.37	299.53	343.86	15.69%
T ₁	221.71	271.94	316.02	6.32%
T ₂	228.31	278.54	324.68	9.24%
T ₃	234.63	286.92	331.93	11.67%
T ₄	252.82	304.30	348.84	17.36%
T ₅	239.41	295.82	340.11	14.43%
Z ₁	211.75	261.99	305.14	2.66%
Z ₂	216.52	267.43	311.47	4.79%
Z ₃	225.33	275.58	320.55	7.85%
Z ₄	236.41	291.05	335.92	13.02%
Z ₅	230.61	282.87	327.73	10.26%

Figure 17 shows the 28-day compressive strength of each mixture relative to the standard (297.23 kg/cm²), quantifying the relative effect of each nano-additive and its dosage. For nano-SiO₂, the greatest increase was obtained with 3% (379.41 kg/cm²), equivalent to a 27.65% increase compared to the standard, whilst the smallest increase within the group occurred at 5% (343.86 kg/cm²), corresponding to an increase of 15.69%, confirming that overdosing reduced the reinforcement efficiency. For nano-TiO₂, the best performance was observed at 2% (348.84 kg/cm²), with an increase of 17.36%, and the lowest at 0.5% (316.02 kg/cm²),

with an increase of 6.32%, demonstrating a dosage-dependent response with an optimum at 2%. For nano-ZnO, the greatest increase was recorded at 0.6% (335.92 kg/cm²), equivalent to an increase of 13.02%, whilst the smallest was observed at 0.3% (305.14 kg/cm²), representing an increase of 2.66%, showing more moderate improvements compared to nano-SiO₂ and nano-TiO₂. Overall, at 28 days, the optimal concentrations were determined to be 3% nano-SiO₂, 2% nano-TiO₂ and 0.6% nano-ZnO, as these yielded the greatest increases in strength without any evidence of penalties associated with overdosing.

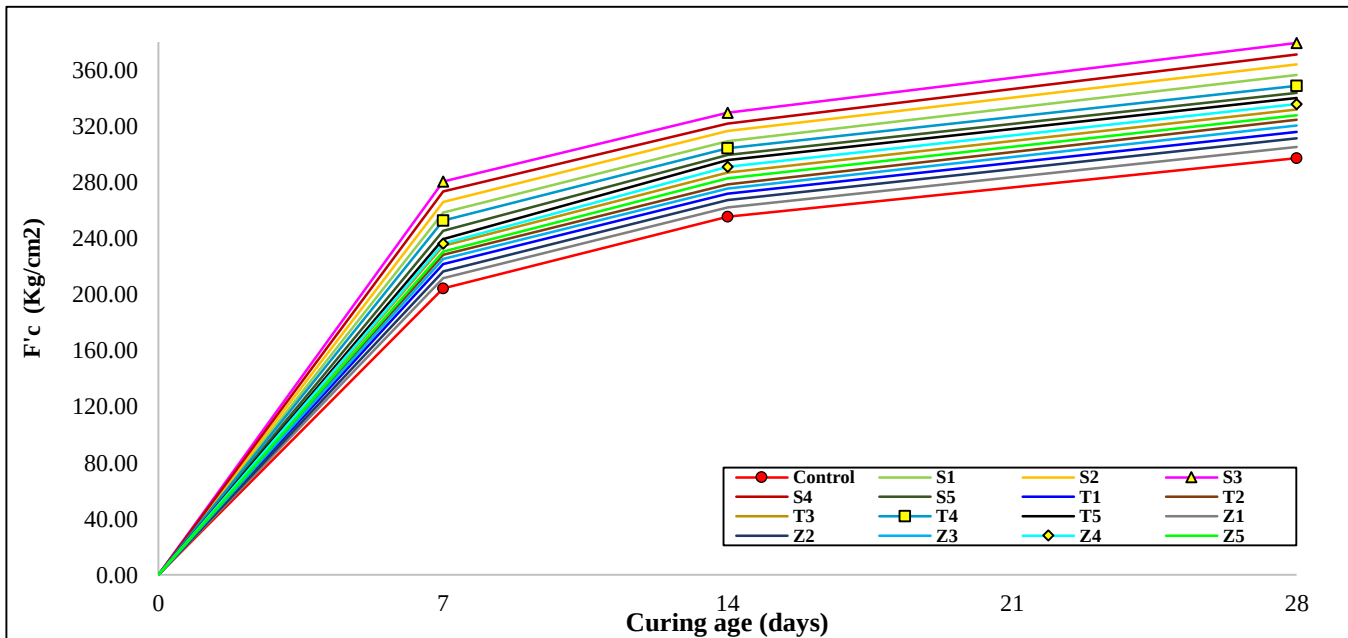


Fig. 17 Evolution of concrete compressive strength with age

4.3. Flexural Strength

Table 5 presents the flexural strength of the concrete at 28 days for the control mix and the modified mixes. The conventional mix achieved 50.46 kg/cm², a value representative of the typical behaviour of rigid pavements. With nano-SiO₂, strengths of 58.79 kg/cm² (1%), 61.78 kg/cm² (2%), 69.08 kg/cm² (3%), 66.12 kg/cm² (4%) and 55.41 kg/cm² (5%) were obtained, with an optimum observed at 3%.

This increase is attributed to matrix densification, pore refinement and improvement of the interfacial transition zone (ITZ) due to the pozzolanic and nucleating effect; whereas the reduction at higher dosages is related to particle agglomeration and increased water demand. In the case of nano-TiO₂, values

of 53.93 kg/cm² (0.5%), 56.64 kg/cm² (1%), 60.18 kg/cm² (1.5%), 64.88 kg/cm² (2%) and 63.22 kg/cm² (2.5%) were recorded, reaching a maximum at 2%, associated with the filler effect, nucleation of hydration products and improvement of the ITZ, with a slight final reduction due to agglomeration.

For nano-ZnO, the values were 51.64 kg/cm² (0.3%), 52.53 kg/cm² (0.4%), 54.71 kg/cm² (0.5%), 57.50 kg/cm² (0.6%) and 55.92 kg/cm² (0.7%), with the best performance at 0.6%, due to the balance between nanometric filling and microstructural continuity, while the decrease at higher content is attributed to effects on hydration kinetics and a greater tendency to agglomerate.

Table 5. Flexural strength results

ID	Flexural Strength (kg/cm ²)			% Variation with respect to Control at 28 Days
	7 days	14 days	28 days	
Control	35.31	43.19	50.46	-
S ₁	41.39	49.74	58.79	16.51%
S ₂	43.58	52.13	61.78	22.43%
S ₃	49.03	57.33	69.08	36.89%
S ₄	46.70	55.71	66.12	31.03%
S ₅	39.41	47.53	55.41	9.80%
T ₁	38.22	46.60	53.93	6.87%
T ₂	40.29	48.48	56.64	12.24%
T ₃	42.37	51.11	60.18	19.27%
T ₄	45.30	54.21	64.88	28.57%
T ₅	44.31	53.11	63.22	25.29%
Z ₁	36.52	44.69	51.64	2.34%
Z ₂	37.14	45.36	52.53	4.10%
Z ₃	38.68	46.97	54.71	8.42%
Z ₄	40.88	49.15	57.50	13.95%
Z ₅	39.85	47.88	55.92	10.81%

Figure 18 compares the flexural strength of the concrete at 28 days for the mixtures modified with nanoparticles with that of the standard mixture, which reached a value of 50.46 kg/cm², thereby enabling the relative effect of each type of nano-additive and its dosage to be identified. For nano-SiO₂, the greatest increase was observed when 3% nano-SiO₂ was incorporated, achieving a strength of 69.08 kg/cm², which represented a 36.89% increase compared to the standard, constituting the highest performance among all the mixtures evaluated; this behaviour demonstrated the high efficiency of nano-silica in improving the flexural response of the concrete pavement.

In contrast, the smallest increase within this group was recorded with 5% nano-SiO₂, with a strength of 55.41 kg/cm², equivalent to an increase of 9.80%, indicating that overdosing significantly reduced the efficiency of the reinforcement. For nano-TiO₂, the best performance was achieved with 2% nano-TiO₂, where the strength was 64.88 kg/cm², corresponding to an increase of 28.57% compared to the control; by contrast,

the smallest increase occurred with 0.5% nano-TiO₂, which reached 53.93 kg/cm², equivalent to an increase of 6.87%, showing that at low dosages the effect of the nano-additive was limited. In the case of nano-ZnO, the highest strength was obtained with 0.6% nano-ZnO, reaching 57.50 kg/cm², which represented a 13.95% increase compared to the control; in contrast, the smallest increase was observed with 0.3% nano-ZnO, with a value of 51.64 kg/cm², equivalent to an increase of 2.34%, demonstrating that this nano-additive exhibited more moderate improvements compared to nano-silica and nano-TiO₂.

The figure confirmed that, at 28 days, the optimal flexural strength performance corresponded to 3% nano-SiO₂, 2% nano-TiO₂ and 0.6% nano-ZnO, whilst the lowest efficiencies were associated with dosages far removed from these optimal levels, reinforcing the importance of controlling the nanoparticle content to maximise the flexural performance of the concrete pavement.

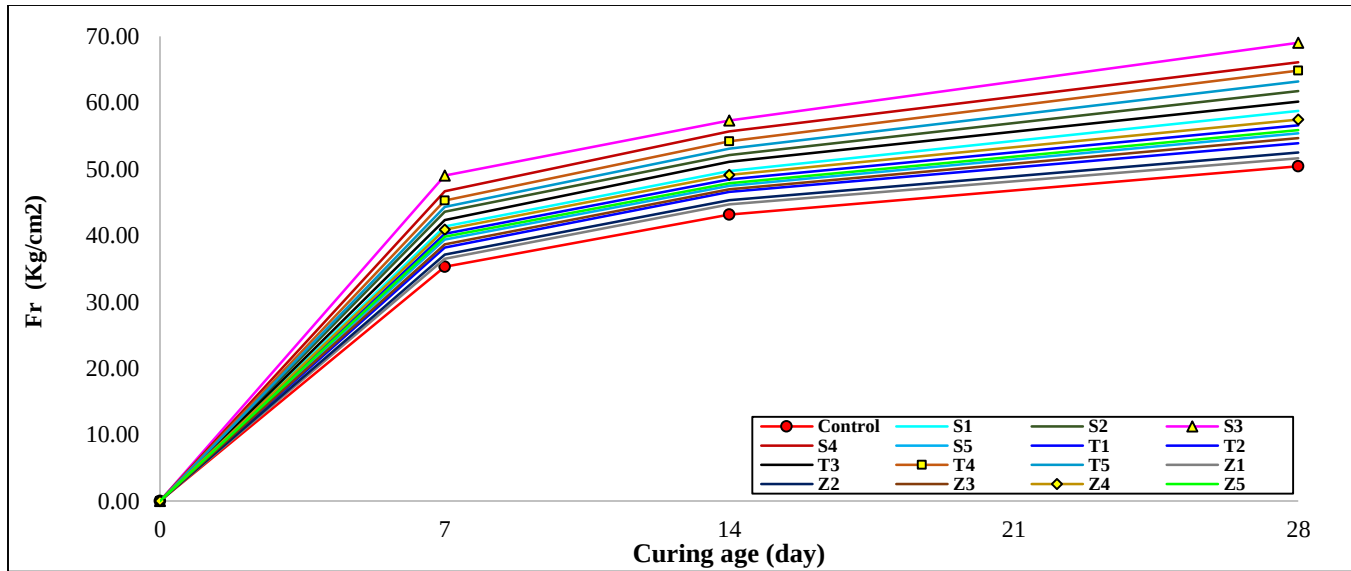


Fig. 18 Development of concrete flexural strength

4.4. Freeze-Thawing of Concrete

Table 6 shows the results for the relative dynamic modulus of elasticity (RDME), together with the values for the resonant frequency, initial and final mass, and the number of cycles reached during the freeze-thaw test. The standard mix exhibited an RDME of 66.09%, reflecting a significant loss of dynamic stiffness after 275 cycles, a behaviour associated with increased pore connectivity and the progressive generation of microcracks due to the expansion of frozen water in the unmodified cementitious matrix. In the case of the mixtures with nano-SiO₂, the 1% addition (S1) achieved an RDME of 82.01%, demonstrating an improvement attributed to the nanometric filler effect and the acceleration of hydration. At 2% (S2), the RDME increased to 86.47%, whilst at 3% (S3) the maximum value of 92.58% was obtained, a result consistent with optimal densification of the microstructure, greater formation of C-S-H gel and a reduction in critical capillary porosity. When the dosage was increased to 4% (S4) and 5% (S5), the RDME fell to 80.98% and 73.97%, respectively, which was attributed to nanoparticle agglomeration and a less homogeneous distribution, both of

which limited the reinforcement's effectiveness against cyclic damage. For mixtures containing nano-TiO₂, additions of 0.5% (T1) and 1% (T2) yielded RDME values of 70.03% and 72.08%, showing moderate improvements over the control; with 1.5% (T3) the RDME increased to 81.98%, and with 2% (T4) a high value of 91.50% was achieved, associated with effective refinement of the porous system and strengthening of the ITZ. The addition of 2.5% (T5) produced an RDME of 88.52%, slightly lower, indicating the onset of a loss of efficiency due to overdosing. As for nano-ZnO, mixture Z1 (0.3%) exhibited an RDME of 78.01%, whilst Z2 (0.4%) and Z3 (0.5%) reached 82.52% and 85.01%, respectively, a behaviour linked to a progressive improvement in internal compactness and more controlled hydration. The best performance was observed at 0.6% (Z4), with an RDME of 88.04%, attributable to a favourable balance between the nanometric filling effect and the mitigation of microcracking damage; however, upon increasing the dosage to 0.7% (Z5), the RDME decreased to 79.98%, reflecting greater interference by ZnO in the hydration kinetics and a reduction in microstructural stability under severe thermal cycles.

Table 6. Results of concrete freeze-Thaw testing

ID	Final Cycle	Initial Weight (kg)	Final Weight (kg)	Initial Frequency (Hz)	Final Frequency (Hz)	RDME (%)
Control	275	7.232	7.044	3790	3081	66.09
S1	295	7.282	7.165	3834	3472	82.01
S2	300	7.202	7.116	3750	3487	86.47
S3	300	7.188	7.130	3885	3738	92.58
S4	300	7.264	7.141	3855	3469	80.98
S5	292	7.183	7.025	3901	3355	73.97
T1	286	7.221	7.019	3904	3267	70.03
T2	291	7.244	7.092	3868	3284	72.08
T3	300	7.208	7.093	3818	3457	81.98

T4	300	7.263	7.201	3729	3567	91.50
T5	300	7.265	7.189	3752	3530	88.52
Z1	281	7.297	7.158	3760	3321	78.01
Z2	289	7.205	7.097	3755	3411	82.52
Z3	300	7.223	7.129	3820	3522	85.01
Z4	300	7.240	7.168	3775	3542	88.04
Z5	300	7.266	7.135	3747	3351	79.98

Figure 19 shows a comparison of the RDME of the mixtures modified with nanoparticles relative to the reference mixture, which achieved a value of 66.09%. Compared to this control, mixture S1 increased the RDME by 24.1%, S2 by 30.8%, and S3 by 40.1%, the latter representing the largest increase observed, which confirmed the highly beneficial effect of 3% nano-SiO₂ on resistance to freeze-thaw deterioration. In contrast, S4 and S5 showed more moderate increases of 22.5% and 11.9%, respectively, indicating a loss of efficiency at higher concentrations. For nano-TiO₂, the

increases relative to the control were 6.0% (T1), 9.1% (T2) and 24.0% (T3), reaching a maximum increase of 38.4% with T4, whilst T5 showed an increase of 33.9%, confirming that the optimal dosage was around 2%. In the case of nano-ZnO, the increases in RDME were 18.0% (Z1), 24.9% (Z2) and 28.6% (Z3), with the best performance achieved with Z4, showing an increase of 33.2%, whilst Z5 showed a smaller increase of 21.0%, once again demonstrating the adverse effect of overdosing.

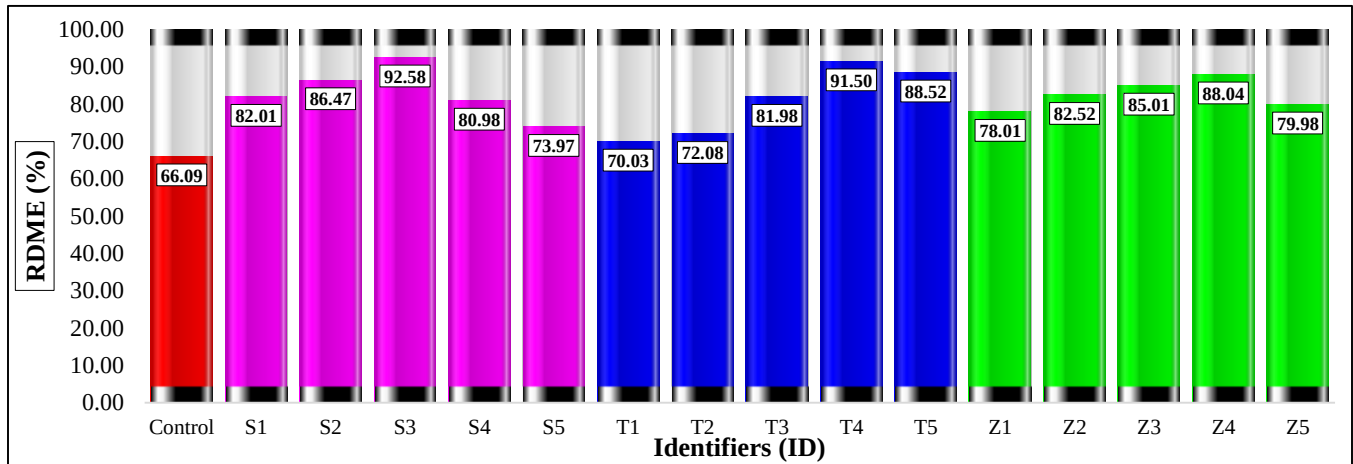


Fig. 19 Relative Dynamic Modulus of Elasticity (RDME) of concrete following freeze-thaw cycles

4.5. Sorptivity Test

Table 7 shows the initial and secondary sorptivity coefficients determined using the ASTM C1585 test, which enabled the assessment of water transport by capillary absorption in hardened concrete. The standard mix exhibited an initial sorptivity of 0.003785 mm/s^{1/2} and a secondary sorptivity of 0.001179 mm/s^{1/2}, a behaviour associated with a more interconnected porous network and the absence of microstructural refinement mechanisms, which facilitated both the initial ingress of water and its propagation in subsequent stages. In the mixtures with nano-SiO₂, the S1 addition recorded 0.003286 and 0.001040, whilst S2 reached 0.003273 and 0.001029, demonstrating a sustained reduction in capillary transport attributed to the nanometric filler effect and the pozzolanic reaction of the nano-silica, which promoted the additional formation of C-S-H gel and the obstruction of capillary pores. Mixture S3 exhibited the lowest values in the group, with 0.003034 for initial sorptivity and 0.000825 for secondary sorptivity, reflecting an optimal

dosage that led to more effective refinement of the porous network and reduced capillary connectivity in both the early and late stages. In S4, values of 0.003121 and 0.000948 were obtained, with the densification effect persisting, albeit with lower efficiency than in S3, whilst S5 achieved 0.003633 and 0.001016, showing a partial loss of the benefit due to overdosing, attributed to nanoparticle agglomeration and less homogeneous dispersion in the cementitious matrix. For the mixtures with nano-TiO₂, T1 showed 0.003605 and 0.001066, indicating a moderate reduction compared to the control; T2 and T3 reached 0.003386 / 0.001029 and 0.003168 / 0.000968, respectively, reflecting a progressive improvement in capillary control associated with the nucleating effect and the strengthening of the interfacial transition zone. Mixture T4 recorded 0.003016 and 0.000908, demonstrating the best performance of the TiO₂ group, consistent with a dosage that favoured the refinement of the porous system without generating heterogeneities; however, in T5 the values increased to 0.003254 and 0.001016, indicating a decrease in

efficiency due to an excess of nano-additive. In the case of nano-ZnO, Z1, Z2 and Z3 exhibited initial sorptivities of 0.003766, 0.003709 and 0.003519, and secondary sorptivities of 0.001164, 0.001156 and 0.001144, respectively, showing limited reductions compared to the standard. The Z4 mixture achieved values of 0.003396 and 0.001131, representing the best performance within the ZnO group; however, Z5 recorded 0.003623 and 0.001149, again showing a loss of effectiveness, a behaviour consistent with the influence of ZnO on the hydration kinetics, which can delay the formation of dense cementitious products and favour the persistence of interconnected pores in later stages.

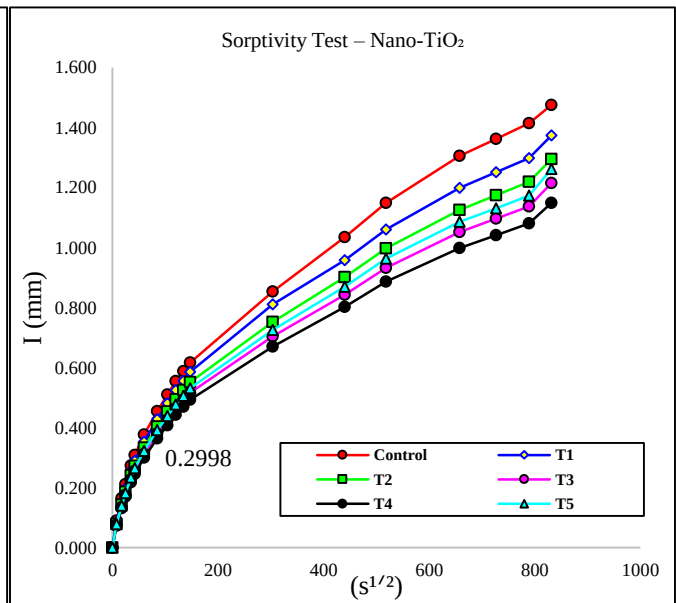
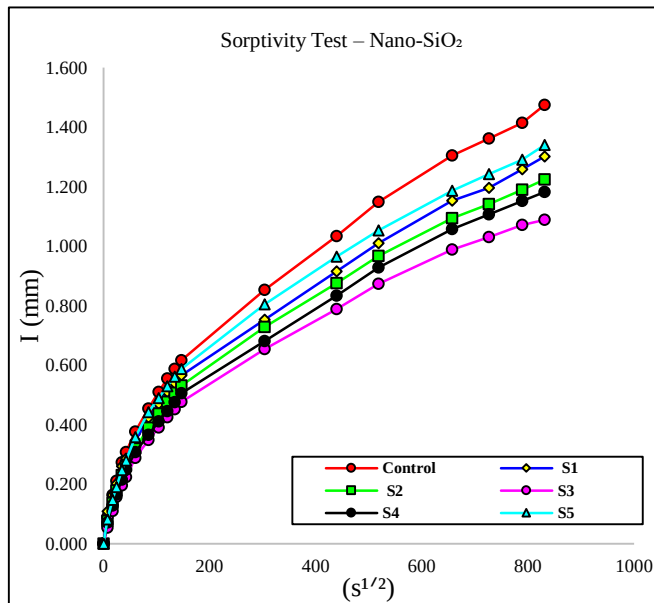
Table 7. Sorptivity test results

ID	Initial Sorptivity (mm/s ^{1/2})	Secondary Sorptivity (mm/s ^{1/2})
Control	0.003785	0.001179
S1	0.003286	0.001040
S2	0.003273	0.001029
S3	0.003034	0.000825
S4	0.003121	0.000948
S5	0.003633	0.001016
T1	0.003605	0.001066
T2	0.003386	0.001029
T3	0.003168	0.000968
T4	0.003016	0.000908
T5	0.003254	0.001016
Z1	0.003766	0.001164
Z2	0.003709	0.001156
Z3	0.003519	0.001144
Z4	0.003396	0.001131
Z5	0.003623	0.001149

Figure 20 shows a comparison of the sorptivity of the mixtures modified with nanoparticles relative to the reference mixture, which had an initial sorptivity of 0.003785 mm/s^{1/2} and a secondary sorptivity of 0.001179 mm/s^{1/2}. In the nano-SiO₂ group, the initial sorptivity decreased by 13.2% (S1), 13.5% (S2), 19.8% (S3) and 17.5% (S4), whilst S5 showed a reduction of only 4.0%, indicating a loss of efficiency due to overdosing; in the secondary stage, the reductions were 11.8%, 12.7%, 30.0% and 19.6% for S1, S2, S3 and S4, respectively, confirming S3 as the optimal dosage as it exhibited the greatest simultaneous decrease in both stages. For nano-TiO₂, the initial sorptivity was reduced by 4.8% (T1), 10.5% (T2), 16.3% (T3), 20.3% (T4) and 14.0% (T5), whilst the secondary sorptivity decreased by 9.6%, 12.7%, 17.9%, 23.0% and 13.8%, respectively, with T4 identified as the most efficient addition in the group due to its lower initial sorptivity and a significant reduction in the secondary stage.

In the case of nano-ZnO, the reductions in initial sorptivity were limited, reaching 0.5% (Z1), 2.0% (Z2), 7.0% (Z3), 10.3% (Z4), and 4.3% (Z5), whereas secondary sorptivity decreased only between 1.3% and 4.1%. This indicates that, although ZnO partially contributed to reducing initial water ingress, it did not achieve effective control of long-term capillary transport.

The figure confirmed that the optimum dosages for minimising capillary water transport were S3 (3% nano-SiO₂) as the best overall performance, and T4 (2% nano-TiO₂) as the best within its group, due to their greater ability to densify the cementitious matrix and reduce pore connectivity, a critical aspect for the durability of rigid pavements exposed to moisture and aggressive agents.



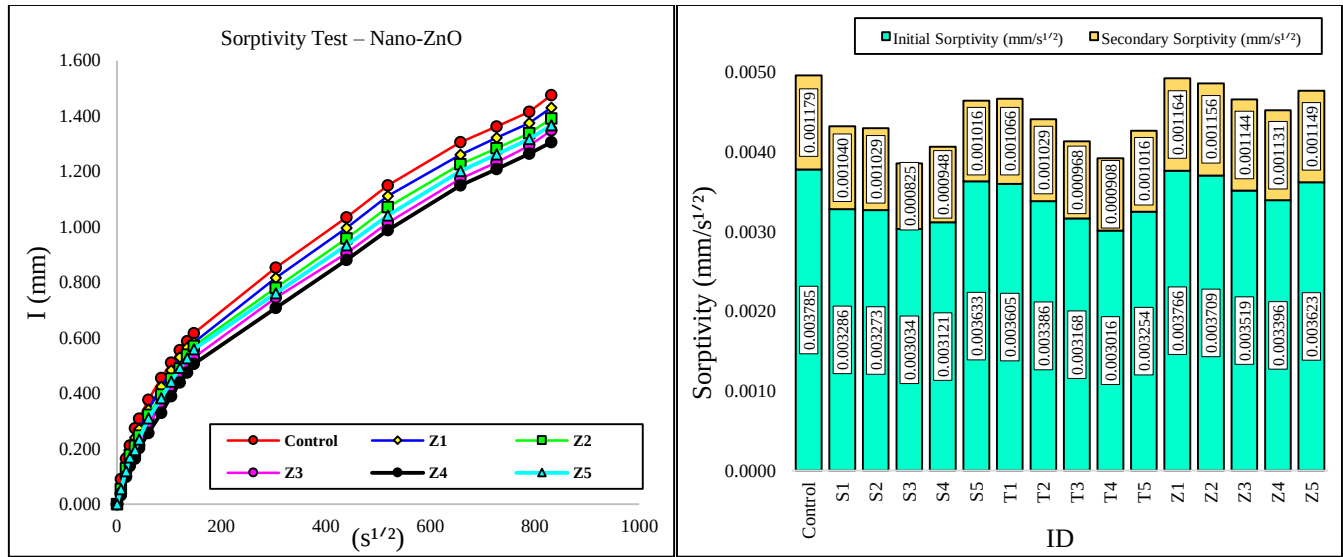


Fig. 20 Comparison of the sorptivity behaviour of concrete

5. Discussion

In Saudi Arabia, the incorporation of nano-SiO₂ in concrete was evaluated through compressive strength, flexural strength, and durability tests associated with absorption and damage under freeze–thaw cycles. It was reported that dosages between 2% and 4% increased compressive strength by up to 44% at 28 days, with an optimum at 3%, whereas higher contents progressively reduced strength. In terms of flexural performance, increases of up to 39% compared to the control were reported, attributed to improvements in the Interfacial Transition Zone (ITZ). Furthermore, capillary absorption and sorptivity were significantly reduced (by up to 40%), and the relative dynamic modulus remained above 90% after 300 cycles for optimal dosages [29]. In the present study, the control concrete achieved a compressive strength of 297.23 kg/cm² and a flexural strength of 50.46 kg/cm² at 28 days, with sustained improvements observed with nano-SiO₂ up to an optimum of 3%. At this dosage, compressive strength reached 379.41 kg/cm² and flexural strength 69.08 kg/cm², corresponding to increases of 27.65% and 36.89% relative to the control, respectively; beyond 3%, a gradual decline in performance was observed. Regarding durability, the control exhibited a relative dynamic modulus of 66.09% after 275 cycles, whereas the 3% nano-SiO₂ mixture maintained 92.58% and completed 300 cycles without critical deterioration. Additionally, sorptivity decreased from 0.003785 to 0.003034 (initial) and from 0.001179 to 0.000825 (secondary), indicating reduced capillary transport. Both studies identified an optimum dosage close to 3% nano-SiO₂ and a loss of performance at higher contents, associated with changes in pore refinement and in the quality of the C–S–H gel and the ITZ.

In China, nano-TiO₂ was applied superficially to concrete paving blocks, comparing its particulate and nanotubular forms through compressive strength, abrasion, and

photocatalytic NO removal tests. The control concrete achieved 44.50 MPa, a value similar to that obtained with particulate nano-TiO₂ (44.51 MPa), whereas nanotubular nano-TiO₂ increased strength up to 45.98 MPa. In terms of abrasion, the nanotubular system (3.85) outperformed both the control (3.57) and the particulate form (2.94). Furthermore, the photocatalytic efficiency of nanotubular nano-TiO₂ reached approximately 50% NO removal after 110 minutes of UV irradiation, exceeding that of the particulate material (35%) and maintaining stability after surface polishing [34]. In the present study, the incorporation of nano-TiO₂ led to a progressive increase in performance up to an optimum dosage of 2%, achieving 348.84 kg/cm² in compressive strength and 64.88 kg/cm² in flexural strength, corresponding to increases of 17.36% and 28.57%, respectively. When the dosage was increased to 2.5%, a relative decrease in performance was observed. Regarding durability, the mixture with 2% nano-TiO₂ maintained a relative dynamic modulus of 91.50% after 300 freeze–thaw cycles, compared to 66.09% for the control, and reduced initial and secondary sorptivity to 0.003016 and 0.000908, respectively. Both studies confirmed improvements with nano-TiO₂, attributed to the form and method of incorporation: the nanotubular form applied superficially provided localised reinforcement and enhanced photocatalytic activity, whereas the particulate form within the matrix densified the C–S–H gel, improving strength and durability, albeit with a risk of agglomeration at higher dosages.

In India, the effect of nano-ZnO incorporated into concrete as a partial cement replacement (0.5%, 0.75%, and 1.0%) was evaluated, maintaining a water–cement ratio of 0.45. The control concrete achieved 30.98 MPa at 28 days, whereas the optimum dosage of 0.5% nano-ZnO increased compressive strength to 33.30 MPa, representing an increase of 6.32%, with reductions observed at higher contents. The indirect tensile strength showed a slight increase from 2.74

MPa to 2.85 MPa, equivalent to a 3.86% improvement. In terms of durability, acid attack using 1% HCl showed a moderate reduction in mass loss compared to the control, although lower than that achieved with nano-SiO₂. Additionally, Ultrasonic Pulse Velocity (UPV) tests classified the nano-ZnO concrete as having moderate quality, indicating limited microstructural improvement [42]. In the present study, the incorporation of nano-ZnO resulted in progressive increases in both mechanical properties up to an optimum dosage of 0.6%, reaching compressive strength values of 335.92 kg/cm², corresponding to a 13.02% increase, and flexural strength of 57.50 kg/cm², equivalent to a 13.95% increase. For higher contents, a decline in mechanical performance was observed. Regarding durability, the mixture with 0.6% nano-ZnO maintained a relative dynamic modulus of 88.04% after 300 freeze–thaw cycles, compared to 66.09% for the control concrete, and exhibited a moderate reduction in both initial and secondary sorptivity. Both studies identified a low optimum range for nano-ZnO, with moderate improvements attributed to its non-cementitious nature and the formation of Zn(OH)₂. The differences were associated with dispersion and concrete type, with greater effectiveness observed against physical damage than chemical deterioration.

6. Conclusion

Firstly, the results confirmed that the incorporation of oxide nanoparticles in concrete for rigid pavements constitutes a viable alternative to improve material performance from a microstructural perspective. The application of standardised tests demonstrated that the refinement of the pore system and the strengthening of the interfacial transition zone translate into improvements associated with both strength and durability under service conditions. In this regard, the study provides useful criteria for the design of higher-performance concretes subjected to flexural loading and environments exposed to water ingress and cyclic damage. The main contribution of the study lies in the comparative evaluation of nano-SiO₂, nano-TiO₂ and nano-ZnO under the same concrete composition and experimental framework, allowing their relative effectiveness and optimum incorporation ranges to be identified. In particular, the most efficient dosages were 3% nano-SiO₂, 2% nano-TiO₂, and 0.6% nano-ZnO, as they exhibited simultaneous improvements compared to the control concrete.

Secondly, the behaviour of concrete incorporating nano-SiO₂ demonstrated that its effectiveness does not rely solely on its high reactivity, but on the balance between matrix densification and adequate dispersion to avoid heterogeneities. The results confirmed the existence of an optimum threshold, as an excess of ultrafine particles may promote agglomeration, increase the effective water demand, and generate discontinuities within the cementitious gel. In rigid pavements, nano-silica proved to be particularly effective when applied within controlled ranges, enhancing

mechanical performance while simultaneously reducing capillary transport, which is critical for durability. In this regard, the optimum dosage was 3% nano-SiO₂ (S3), with increases of 27.65% in compressive strength and 36.89% in flexural strength, as well as an improvement of 40.1% in the Relative Dynamic Modulus of Elasticity (RDME) compared to the control, indicating greater resistance to internal deterioration due to freeze–thaw action. Additionally, sorptivity decreased by 19.8% in its initial component and by 30.0% in the secondary component, demonstrating a more effective and sustained pore refinement.

Thirdly, the incorporation of nano-TiO₂ exhibited behaviour characteristic of nano-additives with a predominantly physical action, where the improvement is mainly associated with the nucleation of hydration products and ultrafine filling, which compacts the paste and strengthens the interfacial transition zone, without a significant pozzolanic contribution. The results indicated that the benefit is maximised when the dosage allows a homogeneous distribution of the nanomaterial, as overdosing may induce microstructural discontinuities and reduce reinforcement efficiency. In the context of rigid pavements, nano-TiO₂ proved to be an effective alternative for enhancing strength and stability against cyclic damage, provided that its content is properly controlled. In particular, the optimum dosage corresponded to 2% nano-TiO₂ (T4), with increases of 17.36% in compressive strength and 28.57% in flexural strength, as well as an improvement of 38.4% in the Relative Dynamic Modulus of Elasticity (RDME) compared to the control. Additionally, reductions of 20.3% in initial sorptivity and 23.0% in secondary sorptivity were recorded, consistent with a less connected pore system and a more stable ITZ against water ingress.

Fourthly, nano-ZnO exhibited a response highly dependent on dosage, consistent with its ability to modify hydration kinetics and the early microstructural development of concrete. The results indicated that its favourable effect is achieved only under an appropriate balance between nano-filler compaction and controlled interference in hydration, as higher contents reduce its efficiency due to heterogeneities and alterations in the formation of cementitious phases. In rigid pavements, ZnO proved to be a moderately effective and selective improvement strategy, particularly against cyclic physical damage mechanisms, although it requires strict control of dosage and mixing. In this regard, the most efficient condition was 0.6% nano-ZnO (Z4), with increases of 13.02% in compressive strength and 13.95% in flexural strength, as well as an improvement of 33.2% in the Relative Dynamic Modulus of Elasticity (RDME) compared to the control. However, its influence on capillary transport was more limited, with reductions of 10.3% in initial sorptivity and 4.1% in secondary sorptivity, confirming a lower contribution to durability associated with late-stage absorption compared to nano-silica and nano-TiO₂.

Finally, the study provides applicable evidence for optimising concrete intended for rigid pavements through nano-modification, demonstrating that performance depends not only on the type of nanoparticle but also on the optimum incorporation range and the microstructural mechanisms governing moisture transport, internal integrity, and flexural response. As a practical limitation, the research suggests that actual field performance may be influenced by dispersion quality, variations in local materials, and curing conditions. The experimental programme did not include combined environmental exposures, direct advanced microstructural characterization, field-scale validation, or quantitative service-life and maintenance modelling. Therefore, future studies should further investigate incorporation methods (e.g.,

dispersing agents, premixes, and sonication), pilot-scale validation, and evaluation under repeated fatigue conditions representative of traffic loading.

Accordingly, its application is recommended in the design of more durable, rigid pavements in environments where water ingress and cyclic damage govern service life, prioritising controlled dosage ranges and mixing protocols that ensure homogeneity and reproducibility.

Conflicts of Interest

We declare that there is no conflict of interest regarding the publication of this article.

References

- [1] Anno Mahfuda, Suprpto Siswosukarto, and Bambang Suhendro, "The Influence of Temperature Variations on Rigid Pavement Concrete Slabs," *Journal of the Civil Engineering Forum*, vol. 9, no. 2, pp. 139-150, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [2] Kristina Bayraktarova et al., "Characterisation of the Climatic Temperature Variations in the Design of Rigid Pavements," *International Journal of Pavement Engineering*, vol. 23, no. 9, pp. 3222-3235, 2022. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [3] Emad Alshammari et al., "Thermal and Flood Resiliency Evaluation of Rigid Pavement Using Various Pavement Characteristics," *CivilEng*, vol. 6, no. 2, pp. 1-14, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [4] Nathaly Deysi Rojas Oncebay, "Analysis of the Development of the Properties of Concrete with Nanosilica Exposed to Freeze-Thaw Cycles in Huancaayo-2022," Thesis, National University of Central Peru, pp. 1-376, 2024. [[Publisher Link](#)]
- [5] San Luo et al., "Impact of Freeze–Thaw Cycles on the Long-Term Performance of Concrete Pavement and Related Improvement Measures: A Review," *Materials*, vol. 15, no. 13, pp. 1-19, 2022. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [6] Hanglin Luo et al., "Freeze–Thaw Deterioration Mechanisms of Concrete Under Low-Temperature High-Frequency Cycling in High-Altitude Mountainous Regions," *Case Studies in Construction Materials*, vol. 23, pp. 1-17, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [7] Xiao Guan et al., "Effect of Freeze-thaw Damage and Pore Structure on Capillary Water Absorption of Gangue-based Concrete," *KSCE Journal of Civil Engineering*, vol. 28, no. 6, pp. 2315-2328, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [8] Jun Zhao et al., "Dynamic Behavior and Damage Mechanisms of Concrete Subjected to Freeze–Thaw Cycles," *Buildings*, vol. 15, no. 12, pp. 1-14, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [9] Jingtao Wang, Fengxia Han, and Qing Liu, "Research on Freeze–Thaw Damage and Life Prediction Model of Polyethylene Fiber–Reinforced Cementitious Composites based on Reliability Analysis," *Case Studies in Construction Materials*, vol. 20, pp. 1-16, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [10] Lantian Xu et al., "A New Concrete Freeze–Thaw Damage Model Based on Hydraulic Pressure Mechanism and Its Application," *Materials*, vol. 18, no. 15, pp. 1-21, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [11] Temperatures as Low as -15 Degrees Celsius: Frost Recorded in High-Altitude Areas of Puno, Arequipa, and Tacna, Perú 21, 2024. [Online]. Available: <https://peru21.pe/peru/hasta-15-grados-bajo-cero-registran-heladas-en-zonas-altas-de-puno-arequipa-y-tacna-puno-heladas-en-peru-helada-en-puno-tacna-arequipa-senamhi-frio-noticia/>
- [12] Senamhi: These are the Regions in Peru that Experience Sub-Zero Temperatures and are Under an Orange Alert, La República, 2025. [Online]. Available: <https://larepublica.pe/sociedad/2025/07/09/senamhi-estas-son-las-regiones-en-peru-que-soportan-temperaturas-bajo-cero-y-poseen-alerta-naranja-742284>
- [13] Senamhi Reports that 4 Regions are Experiencing Frost and Sub-Zero Temperatures, The Trade, Peru, El comercio, 2026. [Online]. Available: <https://elcomercio.pe/peru/senamhi-reporta-que-4-regiones-soportan-heladas-y-temperaturas-bajo-cero-ultimas-noticia/>
- [14] Infobae, Frost in Puno Freezes Lagoons and Hits the Population: Senamhi Reports Temperatures as Low as -16°C, 2025. [Online]. Available: <https://www.infobae.com/peru/2025/07/09/heladas-en-puno-congelan-lagunas-y-golpean-a-la-poblacion-senamhi-reporta-temperaturas-de-hasta-16c-bajo-cero/>
- [15] Frost hits Pasco: Dead Animals and Communities at Risk from Temperatures as Low as -10 Degrees Celsius, Infobae, 2025. [Online]. Available: <https://www.infobae.com/peru/2025/07/15/helada-golpea-a-pasco-animales-muertos-y-comunidades-en-riesgo-por-temperaturas-de-hasta-10-grados-bajo-cero/>
- [16] Soner Guler, and Zehra Funda Akbulut, "A Comprehensive Review of Concrete Durability in Freeze-Thaw Conditions: Mechanisms, Prevention, and Mitigation Strategies," *Structures*, vol. 75, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]

- [17] Quan Ma et al., “Frost Resistance and Improvement Techniques of Recycled Concrete: A Comprehensive Review,” *Frontiers in Materials*, vol. 11, pp. 1-19, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [18] Feiyang Yi et al., “Experimental Assessment of Freeze-Thaw Deterioration in Compression-Cast Fiber-Reinforced Concrete,” *Journal of Building Engineering*, vol. 98, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [19] Qinglong Zhang et al., “The Effect of Modification with Nano-Alumina, Nano-Silica, and Polypropylene Fiber on the Frost Resistance of Concrete,” *Buildings*, vol. 15, no. 21, pp. 1-19, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [20] Aniss Zaoui et al., “Fine-Tuning the Microstructure of Concrete Composite: Impact of Surface-Coated Nanocellulose on the Increase of Strength and Decrease of Strength Variation in Concrete,” *Construction and Building Materials*, vol. 484, pp. 1-18, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [21] Shinhyu Kanget al., “Determining the Air-Entraining Admixture Dosage in Concrete with Non-Traditional Coal Ash,” *Materials and Structures*, vol. 56, no. 1, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [22] Lori E. Tunstall, M. Tyler Ley, and George W. Scherer, “Air Entraining Admixtures: Mechanisms, Evaluations, and Interactions,” *Cement and Concrete Research*, vol. 150, 2021. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [23] Dongtao Xia et al., “Damage Characteristics of Hybrid Fiber Reinforced Concrete under the Freeze-Thaw Cycles and Compound-Salt Attack,” *Case Studies in Construction Materials*, vol. 18, pp. 1-18, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [24] Robin Deb, Sharaniya Visvalingam, and Yaghoob Amir Farnam, “Assessment of Real-Time Freeze-Thaw Durability of Concrete Pavements Containing Low-Temperature Phase Change Materials,” *International Journal of Pavement Research and Technology* 2025, pp. 1-22, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [25] Cihang Huang et al., “Effect of Colloidal Nano Silica on the Freeze-Thaw Resistance and Air Void System of Portland Cement Concrete,” *Journal of Building Engineering*, vol. 86, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [26] Chao Xu, Lin Deng, and Dingtao Yang, “Mechanical Properties of Nano-TiO₂-Modified Concrete Under Freeze-Thaw Environment,” *Nanomaterials* 2025, vol. 15, no. 16, pp. 1-15, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [27] Ruize Zhao et al., “Study on the Freeze-Thaw Resistance of Concrete Pavements in Seasonally Frozen Regions,” *Materials*, vol. 17, no. 8, pp. 1-19, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [28] Mohit Kansotiya, Gyanendra Kumar Chaturvedy, and Umesh Kumar Pandey, “Improving Concrete Performance with Nano-SiO₂: A Review,” *AIP Conference Proceedings: International Conference on Emerging Materials, Smart Manufacturing, and Computational Intelligence*, Rajpura, India, vol. 3227, no. 1, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [29] Fadi Althoey et al., “Impact of Nano-Silica on the Hydration, Strength, Durability, and Microstructural Properties of Concrete: A State-of-the-art Review,” *Case Studies in Construction Materials*, vol. 18, pp. 1-22, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [30] Haneen AlTawaiha, Fadi Alhomaidat, and Tamer Eljufout, “A Review of the Effect of Nano-Silica on the Mechanical and Durability Properties of Cementitious Composites,” *Infrastructures*, vol. 8, no. 9, pp. 1-18, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [31] Hammad Khalid et al., “An Experimental Investigation of Mechanical Properties of Concrete Composites Reinforced with PET Fibers as Per ASTM Standard,” *Sustainable Chemistry for the Environment*, vol. 10, pp. 1-8, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [32] Ashmita Mohanty et al., “Impact of Nanomaterials on the Mechanical Strength and Durability of Pavement Quality Concrete: A Comprehensive Review,” *Eng*, vol. 6, no. 4, pp. 1-35, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [33] Dan Huang, Mirian Velay-Lizancos, and Jan Olek, “A Comparative Study of the Impact of Nano-TiO₂ and Nano-silica on the Durability of Concretes Cured at Different Temperatures,” *Procedia Structural Integrity*, vol. 67, pp. 61-79, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [34] Chengqi Meng et al., “Photocatalytic Concrete Paving Block Reinforced by TiO₂ Nanotubes for no Removal,” *Journal of Materials Science*, vol. 55, no. 29, pp. 14280-14291, 2020. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [35] Garima Rawat, Sumit Gandhi, and Yogesh Iyer Murthy, “Durability Aspects of Concrete Containing Nano- Titanium Dioxide,” *Materials Journal*, vol. 120, no. 2, pp. 25-35, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [36] R. Mohanraj, and Nitin Sharma, “Characterization of Concrete with Nano-TiO₂ Particles using Compression and FTIR Methods,” *Procedia Structural Integrity*, vol. 70, pp. 358-364, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [37] Yeon-Tae Kim, Jong-Won Lee, and Sung-Lin Yang, “Influence of Nano SiO₂/Al₂O₃-TiO₂ Powder on Strength and Durability Properties of Concrete,” *KSCE Journal of Civil Engineering*, vol. 30, no. 2, pp. 1-15, 2026. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [38] Bruno Oliveira Bica, and João Victor Staub de Melo, “Concrete Blocks Nano-Modified with Zinc Oxide (ZnO) for Photocatalytic Paving: Performance Comparison with Titanium Dioxide (TiO₂),” *Construction and Building Materials*, vol. 252, 2020. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [39] Ana Silvia Gonzalez Garcia et al., “Assessment of Lightweight Concrete Properties with Zinc Oxide Nanoparticles: Structural and Morphological Analyses,” *Applied Sciences*, vol. 14, no. 11, pp. 1-14, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [40] Ahmed Hamed El-Sayed Salama et al., “Enhanced Cement Properties through Nano-ZnO: A Comprehensive Analysis using ANOVA and DOE Techniques,” *Innovative Infrastructure Solutions*, vol. 10, no. 11, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]

- [41] Mohammad Alharthai et al., "Synthesis and Characterisation of ZnO Nanoparticles and its Influence on the Engineering Properties of lightweight foamed concrete," *Archives of Civil and Mechanical Engineering*, vol. 25, no. 2, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [42] Chittaranjan B. Nayak et al., "Effect of SiO₂ and ZnO Nano-Composites on Mechanical and Chemical Properties of Modified Concrete," *Iranian Journal of Science and Technology*, vol. 46, no. 2, pp. 1237-1247, 2022. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [43] Surya Teja Swarna et al., "The Analysis and Design of Jointed Plain Concrete Pavements with Wider Slabs," *Road Materials and Pavement Design*, vol. 25, no. 12, pp. 2664-2684, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [44] Saima Yaqoob, Johan Silfwerbrand, and Romain Gabriel Roger Balieu, "A Parametric Study Investigating the Dowel Bar Load Transfer Efficiency in Jointed Plain Concrete Pavement Using a Finite Element Model," *Buildings*, vol. 14, no. 4, pp. 1-20, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [45] Weiwei Wang et al., "A Case Study of Pavement Foundation Support and Drainage Evaluations of Damaged Urban Cement Concrete Roads," *Applied Sciences*, vol. 14, no. 5, pp. 1-17, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [46] Daniel D. Akerele, Federico Aguayo, and Lingzi Wu, "Portland Limestone Cement in Concrete Pavement and Bridge Decks: Performance Evaluation and Future Directions," *Buildings*, vol. 15, no. 5, pp. 1-33, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [47] Ministry of Transport and Communications, Director's Resolution No. 10-2014-MTC/14: Soils, Geology, Geotechnics and Pavements, 2014. [Online]. Available: <https://www.gob.pe/institucion/mtc/normas-legales/4441297-10-2014-mtc-14>
- [48] *ASTM C131-06: Standard Test Method for Resistance to Degradation of Small-Size Coarse Aggregate by Abrasion and Impact in the Los Angeles Machine*, ASTM International, pp. 1-4, 2006. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [49] Jirayut Suebsuk et al., "Performance of Sustainable Cement Mortar and Concrete Containing Recycled Asphalt Pavement (RAP) under Tropical Temperature," *Case Studies in Construction Materials*, vol. 23, pp. 1-26, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [50] Ministry of Transport and Communications, "Highway Manuals: Especificaciones Técnicas Generales Para Construcción, pp. 1-1274, 2013. [[Google Scholar](#)] [[Publisher Link](#)]
- [51] Huaming Li et al., "Advances in the Mechanism and Application of Nanoparticles in Concrete Property Modification," *Inorganics*, vol. 13, no. 9, pp. 1-35, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [52] Tao Meng et al., "Effect of Different Particle Sizes of Nano-SiO₂ on the Properties and Microstructure of Cement Paste," *Nanotechnology Reviews*, vol. 9, no. 1, pp. 833-842, 2020. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [53] Hemn Unis Ahmed, Azad A. Mohammed, and Ahmed Salih Mohammed, "Effectiveness of Nano-SiO₂ on the Mechanical, Durability, and Microstructural behavior of Geopolymer Concrete at Different Curing Ages," *Archives of Civil and Mechanical Engineering*, vol. 23, no. 2, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [54] R. M. Ashwini, Kanaka Durga Sambhana, and M. Manjunatha, "Enhancing Concrete Performance and Sustainability with Nano TiO₂: Engineering, Microstructural, and Life Cycle Evaluation," *Iranian Journal of Science and Technology, Transactions of Civil Engineering*, vol. 50, pp. 2687-2702, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [55] Qing-Hai Luo, and Sheng-En Fang, "Influence of Ultrafine Metakaolin and Nano-TiO₂ on the Durability and Microstructure of Seawater Sea - Sand Concrete," *Construction and Building Materials*, vol. 473, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [56] Penglong Zhao et al., "Durability Enhancement Mechanism and Service Life Prediction Model of Nano-Modified Concrete for Buildings in Chloride-Rich Environments," *Journal of Building Engineering*, vol. 119, 2026. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [57] Arzu Çağlar, and Hakan Çağlar, "Enhancement of Fracture Toughness and Mechanical Properties of Ultra-High Performance Concrete with ZnO Nanoparticles under Mixed-Mode Loading Conditions: A Multi-Scale Numerical Investigation," *Engineering Fracture Mechanics*, vol. 329, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [58] Abiola Usman Adebajo et al., "Optimizing Nano-TiO₂ and ZnO Integration in Silica-based High-Performance Concrete: Mechanical, Durability, and Photocatalysis Insights for Sustainable Self-Cleaning Systems," *Construction and Building Materials*, vol. 446, 2024. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [59] *ASTM C143-78 Standard Test Method for Slump of Portland Cement Concrete*, ASTM International, pp. 1-2, 2017. [[Publisher Link](#)]
- [60] Sallal R. Abid et al., "Underwater Abrasion of Steel Fiber-Reinforced Self-Compacting Concrete," *Case Studies in Construction Materials*, vol. 11, pp. 1-17, 2019. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [61] André Pico-Moliner et al., "Incorporation of Recycled Concrete and Mixed Recycled Aggregate as Coarse Aggregate Replacements in Structural Grade Concrete," *Construction and Building Materials*, vol. 511, 2026. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [62] *ASTM C1064/C1064M-17: Standard Test Method for Temperature of Freshly Mixed Hydraulic-Cement Concrete*, ASTM International, pp. 1-3, 2017. [[CrossRef](#)] [[Publisher Link](#)]
- [63] Geovanny Solano-Vinueza, and Jorge Albuja-Sánchez, "Development and Field Based Validation of a Predictive Model for Concrete Compressive Strength using Fresh State Properties in a Large Scale Construction Project," *Case Studies in Construction Materials*, vol. 24, pp. 1-17, 2026. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [64] Radi Al-Rashed, and Maher Al-Jabari, "Managing Thermal Effects in Waterproofed Concrete with Multi-Crystallization Enhancer," *CEMENT*, vol. 10, pp. 1-13, 2022. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]

- [65] ASTM C39/C39M-23: *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*, ASTM International, pp. 1-8, 2023. [[CrossRef](#)] [[Publisher Link](#)]
- [66] Wajeeha Mahmood, Tehmina Ayub, and Asad-ur-Rehman Khan, “Mechanical Properties and Corrosion Resistance of Recycled Aggregate Concrete Exposed to Accelerated and Natural Marine Environment,” *Journal of Building Engineering*, vol. 66, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [67] Fouad Ismail Ismail et al., “The Behavior of Graphene-Nanoplatelets-based High-Performance Concrete under Ambient Curing,” *Structures*, vol. 47, pp. 694-708, 2023. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [68] ASTM C78/C78M-22: *Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)*, ASTM International, pp. 1-9, 2010. [[CrossRef](#)] [[Publisher Link](#)]
- [69] Nakin Suksawang et al., “Using Post-Cracking Strength to Determine Flexural Capacity of Ultra-Thin Whitetopping (UTW) Pavements,” *Construction and Building Materials*, vol. 240, 2020. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [70] Ronak Motiani et al., “Innovative Reinforcement Techniques for 3D-printed Concrete: The Impact of Shape Memory Alloys on Flexural Strength and Crack Mitigation,” *Next Materials*, vol. 9, pp. 1-10, 2025. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [71] ASTM C666-97 *Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing*, ASTM International, pp. 1-6, 2017. [[CrossRef](#)] [[Publisher Link](#)]
- [72] Bahram M. Taheri et al., “Experimental Evaluation of Freeze–Thaw Durability of Pervious Concrete,” *Journal of Building Engineering*, vol. 33, 2021. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [73] Mohammed T. Albahtiti et al., “Effects of Prestressing and Saw-Cutting on the Freeze-Thaw Durability,” *Cement and Concrete Composites*, vol. 104, pp. 1-9, 2019. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [74] Peiman Azarsa, and Rishi Gupta, “Novel Approach to Microscopic Characterization of Cryo Formation in Air Voids of Concrete,” *Micron*, vol. 122, pp. 21-27, 2019. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [75] ASTM C1585-20: *Standard Test Method for Measurement of Rate of Absorption of Water by Hydraulic-Cement Concretes*, ASTM International, pp. 1-6, 2020. [[CrossRef](#)] [[Publisher Link](#)]
- [76] Hakan Çağlar et al., “Investigating the Compressive and Corrosion Resistance Properties of Ultra-High-Performance Fiber-Reinforced Concrete Enhanced with Layered Double Hydroxide Nanoparticles,” *Construction and Building Materials*, vol. 508, 2026. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [77] Amir Hossein Yadak Yaraghi et al., “Evaluation of Test Procedures for Durability and Permeability Assessment of Concretes Containing Calcined Clay,” *Journal of Building Engineering*, vol. 58, 2022. [[CrossRef](#)] [[Google Scholar](#)] [[Publisher Link](#)]
- [78] Sikatop®–107 Seal Per: Cementitious Mortar for Waterproofing and Protecting Concrete, Sika 2025. [Online]. Available: <https://per.sika.com/es/construccion/76277/morteros-impermeabilizantes/sikatop-107-sealper.html>