ISSN 2477-9105
Número 34 Vol.1 (2025)
DOI: https://doi.org/10.47187/perf.v1i34.362
Perfiles
Fecha de recepción: 25-02-2025 · Fecha de aceptación: 16-06-2025 · Fecha de Publicación: 24-11-2025

INFLUENCIA DE LA NANOSÍLICE EXTRAÍDA DE CAPARAZONES DE CANGREJO EN LAS PROPIEDADES FÍSICO-MECÁNICAS DE LOS MORTEROS DE CEMENTO

Influence of nanosilica extracted from crab shells on the physical-mechanical properties of cement mortars

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RESUMEN

Esta investigación evalúa el uso de desechos de caparazones de cangrejo para mejorar morteros, incorporando nanosílice al 0.25% en peso del cemento como alternativa sostenible. Las nanopartículas, obtenidas de residuos biológicos, se aplicaron en cementos Tipo N y HS, formulados para mampostería y resistencia a sulfatos. La metodología incluyó síntesis de nanopartículas, ensayos de compresión uniaxial, pruebas de permeabilidad y análisis microestructural (XRD y SEM).

Los resultados muestran que la nanosílice incrementa resistencia y durabilidad, aunque la nanoquitina y nanopartículas de cal fueron más eficaces a largo plazo. La mezcla $C+S_{0.25}$% alcanzó 31.22 MPa a 90 días, similar a su control (31.63 MPa), mientras que $M+S_{0.25}$% aumentó un 3.2% frente a su referencia. El aumento de resistencia en edades tempranas (24 h-7 d) indica que la nanosílice acelera la hidratación y densifica la matriz, mejorando la cohesión interna.

Las pruebas de permeabilidad evidenciaron comportamiento hidrofóbico (ángulos >90°), reduciendo la absorción de agua y favoreciendo la durabilidad. Esta estrategia optimiza el desempeño del mortero y promueve la sostenibilidad mediante la reutilización de residuos, alineándose con la economía circular y la nanotecnología, demostrando la viabilidad de nanosílice derivada de cangrejo en materiales de construcción ecoeficientes.

Palabras claves: *Nanosílice, mortero, resistencia a la compresión, microestructura, sostenibilidad.*

ABSTRACT

This study evaluates the use of crab shell waste to enhance mortar properties by incorporating nanosilica at 0.25% by cement weight as a sustainable alternative. The nanoparticles, obtained from biological waste, were applied to Type N and HS cements, formulated for masonry and sulfate-resistant applications. The methodology included nanoparticle synthesis, uniaxial compression tests, permeability assessments, and microstructural analyses (XRD and SEM).

Results show that nanosilica improves compressive strength and durability, although nanochitin and calcium nanoparticles proved more effective in long-term performance. The C+$S_{0.25}$% mixture reached 31.22 MPa at 90 days, similar to its control (31.63 MPa), while M+$S_{0.25}$% achieved a 3.2% strength increase compared to its reference. The early-age strength gain (24 h–7 d) indicates that nanosilica accelerates cement hydration and densifies the matrix, improving internal cohesion.

Permeability tests revealed hydrophobic behavior (contact angles >90°), reducing water absorption and enhancing durability. This approach optimizes mortar performance while promoting sustainability through the reuse of waste materials. It aligns with circular economy principles and nanotechnology applications, demonstrating the feasibility of using crab-derived nanosilica in the development of eco-efficient construction materials.

Keywords: *Nanosilica, mortar, compressive strength, microstructure, sustainability.*

I. INTRODUCTION

The integration of nanoparticles in construction materials has gained significant attention due to their ability to enhance mechanical properties, durability, and sustainability. Among these, nanosilica has emerged as a key material for improving the performance of mortars and concretes, primarily by reducing porosity and increasing compressive strength (1). Additionally, nanosilica derived from crab shells represents an innovative strategy for developing more sustainable construction materials, aligning circular economy principles by repurposing industrial by-products and minimizing environmental impact (2).

The incorporation of nanosilica into cementitious matrices has been shown to promote the formation of calcium silicate hydrate (C-S-H) compounds, which accelerates hydration reactions and strengthens the internal microstructure of the mortar (2,3). Additionally, nanosilica acts as a nano-filler, improving aggregate distribution and optimizing material cohesion, which directly contributes to increased durability and resistance to external agents (4,5). Research on high-performance concrete (HPC) indicates that a 1.5% nanosilica replacement can increase compressive strength by 8.44% at 28 days, significantly enhancing cement hydration and matrix densification (6,7).

Moreover, functionalized hydrophobic nano-silica has demonstrated enormous potential in enhancing water repellency and corrosion resistance in cementitious materials. Research findings suggest that substituting 2% of the cement weight with nanosilica enhances the early-age strength of the material and mitigates chloride ion penetration, which is crucial in reinforced concrete applications, particularly in aggressive environments (8). Similarly, the integration of nano-iron and nanosilica in mortars has been found to enhance compressive and tensile strength, with nanoparticle dispersion playing a critical role in optimizing mortar performance and reducing porosity (9,10).

In addition to structural improvements, the use of nanosilica in concrete pavers has been explored as an alternative for sustainable infrastructure development. Research findings indicate that a 3% nanosilica replacement results in a 12% increase in compressive strength, while a combined micro- and nanosilica mix achieves a 23% improvement, demonstrating the feasibility of enhancing concrete durability while reducing cement consumption (11). Furthermore, bio-composite materials reinforced with nanosilica have shown promise in sustainable construction, as silica nanoparticles increase strength, durability, and environmental resistance, offering innovative solutions for eco-friendly material development (9).

In conclusion, the utilization of nanoparticles, particularly nanosilica, presents a significant opportunity to improve the durability, sustainability, and mechanical efficiency of cementitious materials. These advancements contribute to reducing environmental impact, optimizing material properties, and promoting a more resilient and eco-friendly construction industry. As research continues to evolve, the potential of nanotechnology in construction will further expand, providing innovative and high-performance solutions for the built environment.

II. MATERIALS AND METHODS

Crab Shell-Derived Nanosilica

Background

The incorporation of nanosilica into cementitious materials has been extensively studied due to its ability to enhance strength and durability in mortar. Recent research highlights that nanosilica obtained from crab shell waste is emerging as a sustainable and efficient alternative for optimizing cementitious mixtures. The pozzolanic activity of nanosilica enables it to act as a nano-filler, refining the cement matrix structure, reducing porosity, and promoting a more compact material. This effect translates into higher compressive strength, with studies reporting up to 40% improvement in nanosilica-modified mortars, indicating a notable reinforcement of mechanical properties (12).

Additionally, nanosilica contributes to reducing mortar permeability, which is essential in enhancing resistance against aggressive agents such as chlorides and sulfates. This property is particularly relevant for infrastructures exposed to marine or industrial environments (Morales *et al*. (13), the use of nanoparticles in cement-based composites contributes to reducing pollution and environmental impact during production processes. Experimental studies using X-ray diffraction (XRD) and scanning electron microscopy (SEM) have demonstrated that nanosilica induces the formation of additional hydration products, particularly calcium silicate hydrate (C-S-H) gel, which is crucial for mechanical resistance and long-term durability (14).

Moreover, the sustainability benefits of nanosilica are increasingly recognized in construction materials research. The utilization of nanosilica derived from industrial and biological waste sources contributes to lowering CO_2 emissions associated with cement production. This aspect aligns with the circular economy model, promoting eco-friendly alternatives that reduce the demand for natural resources while improving material performance (15).

Furthermore, nanosilica's nanometric scale and high specific surface area allow for superior cement hydration kinetics, accelerating reaction rates and improving early-age strength development. Studies have confirmed that its interaction with calcium hydroxide (CH) reduces the formation of voids, resulting in a denser, more homogeneous microstructure (14).

Lastly, the combination of nanosilica with other nanomaterials, such as nano-iron and nano-titania, has demonstrated additional improvements in flexural strength, impact resistance, and self-healing capabilities. These advancements suggest that nanosilica-enhanced mortars and concretes are crucial for developing high-performance and environmentally sustainable construction materials (16).

The integration of nanosilica, particularly when sourced from sustainable waste materials, presents a promising pathway for improving the mechanical, durability, and sustainability aspects of cement-based materials. Given the growing need for green construction solutions, ongoing research will continue to explore the optimal dosages and hybrid nanomaterial applications to further enhance the efficiency and ecological impact of modern construction practices.

Synthesis

Crab Shell

Figure 1 illustrates the process of obtaining crab flour from crab exoskeletons through four sequential steps:

1. **Crab exoskeleton:** Initial raw material consisting of crab shells.
2. **Washing and drying of crab exoskeleton:** The exoskeleton is cleaned to remove impurities and then dried to facilitate further processing.
3. **Crushing of crab exoskeleton:** The dried exoskeleton is mechanically ground using a milling device.
4. **Crab flour:** The final product is a fine powder obtained from the crushed exoskeleton, which can be used for various applications.

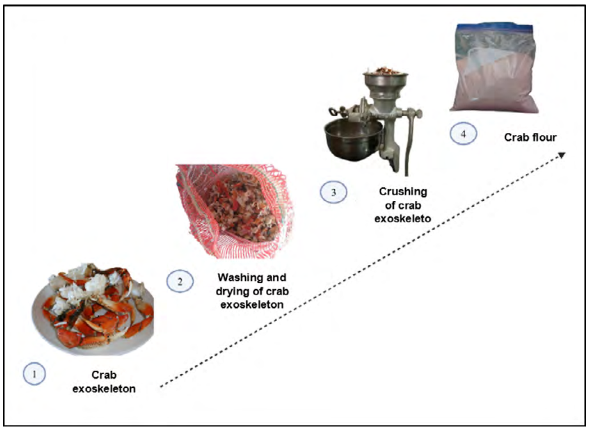


Figura 1. *Production of Raw Material of Crab*

Obtained Chemically

This study investigates the extraction of nanosilica from crab shell waste, aiming to optimize its synthesis for application in cementitious materials. The research prioritizes identifying an efficient methodology that enhances mechanical properties and durability while aligning with sustainable construction principles. A comprehensive evaluation is conducted, focusing on process feasibility, economic viability, and the quality of the extracted material to determine its potential for large-scale implementation. By refining this approach, nanosilica can be systematically integrated into cement-based composites, contributing to enhanced mechanical strength, reduced permeability, and a lower environmental footprint in modern construction.

Nanosilica from crab

For this procedure, the article "Estimation of chitin and chitin nitrogen in crab waste and derived products" (17) served as the basis. To accommodate specific conditions, adaptations were made to the method described in the article. This approach allows for the adjustment and optimization of the analysis process to obtain more precise and relevant results for the study.

In the Figure 2 outlines the process of obtaining nanosilica:

1. **Sample Weighing:** A portion of the dried and ground sample is weighed. The quantity will depend on the type of sample and the expected fiber content.
2. **Acid Digestion:** The sample is dissolved in a 1N hydrochloric acid solution in a digestion vessel at boiling temperature for a specified time (usually 60 minutes).
3. **Filtration:** After acid digestion, the mixture is filtered through a filtration crucible or filter cloth to collect the insoluble fraction, which should reach a neutral pH.
4. **Alkaline Digestion:** The collected residue is dissolved in a 5% sodium hydroxide (NaOH) solution at boiling temperature for a time similar to the acid digestion.
5. **Second Filtration:** The mixture is filtered again to collect the insoluble fraction in the alkaline solution.
6. **Washing:** The residue is washed with hot water until the filtrate is free of acids and alkalis.
7. **Drying and Weighing:** The residue is subjected to drying in an oven at a controlled temperature, typically between 105-110 °C, yielding a chitin residue contaminated with SiO_2.

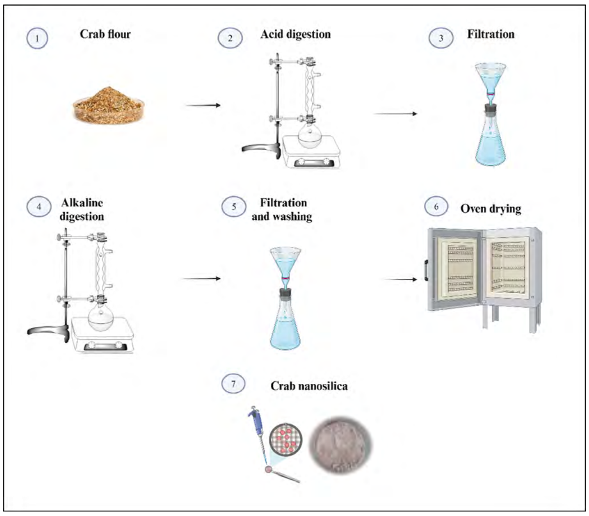


Figura 2. *Crab Nanosilica*

STRATEGIES AND MATERIALS USED

This research presents a comparative technical analysis of the physicochemical and mechanical characteristics of mortars formulated with nanosilica extracted from crab shell waste and two distinct cement types. The study aims to assess the influence of these variables on the overall performance of the mortar, providing insights into their potential applications in construction materials. The selected cements, Type N and Type HS, are known for their performance under various environmental conditions and resistance to aggressive agents. The nanosilica, obtained through a controlled synthesis process, is evaluated for its impact on mortar durability, strength, and permeability reduction. The research methodology involves assessing the influence of nanosilica on mortar performance, considering production complexity, cost efficiency, and final material quality.

The experimental phase includes the characterization of mortar through standardized tests for compressive strength, permeability, and microstructural analysis, employing X-ray diffraction (XRD) and scanning electron microscopy (SEM). This study aims to establish the technical feasibility of utilizing crab shell waste as a sustainable source of nanosilica in construction, improving mortar properties while contributing to eco-friendly construction practices. The comparison of results will determine the optimal cement-nanosilica combination, offering the best performance in terms of durability, mechanical strength, and practical implementation in construction projects.

Materials

The characterization was carried out through laboratory tests that comply with national INEN standards. To create mortar specimens, all materials used were characterized, and trial mixes were made until official mixes were defined based on their properties. It was necessary to detail the mix design and manufacturing process. The results were subsequently applied to mortar in both its fresh and hardened states. The experimental procedures encompassed flowability measurements, compressive strength testing, along with permeability assessments conducted. The resulting data underwent a thorough analysis and interpretation to ensure comprehensive evaluation.

The study focused on the characterization of mortars composed of fine aggregate (sand), cement, and nanosilica synthesized from crab shell waste, as outlined in Table 1. These materials were integral to assessing the performance and properties of the developed mixtures.

Table 1. *Components used*

Materials	Specification	Origin
Fine Aggregate	Quarry of Copeto	Toachi River
Cement	Type N Masonry Cement	Holcim
Cement	Portland Pozzolanic Type HS	UNACEM
Watter	Drinkable	Metropolitan District of Quito Water Network (EPMAPS)
Crab Nanosilica	Synthesized Material	Laboratory-Synthesized

Water

The amount and quality of water incorporated into the mortar mixture are fundamental factors in determining its mechanical characteristics and long-term durability. The use of clean, impurity-free water is vital for effective cement hydration, which directly affects the mortar's ability to meet the required mechanical standards. In contrast, water of inferior quality can adversely affect these properties and reduce the workability of the mixture.

Therefore, adherence to the NTE INEN 2617:2012 standard (18) is essential to ensure that construction water meets the necessary requirements. Compliance with this standard enhances the durability and integrity of structures, contributing to safer and more sustainable construction practices.

Fine Aggregate (Sand)

The Toachi River quarry, situated in the province of Santo Domingo de los Tsáchilas, is operated by Copeto Cía. Ltda., a company specializing in the extraction and supply of construction aggregates (19) . The quarry provides various types of sand, including washed sand, block sand, and natural sand, all of which meet the highest national quality standards (INEN, MOP) and comply with international regulations (ASTM) (20).

Table 2 outlines the physical characteristics of the fine aggregate, reporting a fineness modulus of 2.44. Furthermore, Figure 3 illustrates the particle size distribution curve, which aligns with the requirements established by NTE INEN 2536 (21) for its application in masonry mortar.

Table 2. *Characteristics of Fine Aggregate.*

Characteristics	Units	Results
Colorimetry	-	1
Fineness Modulus	-	2.44
Specific Gravity	g/cm³	2.70
Absorption Capacity	%	1.50

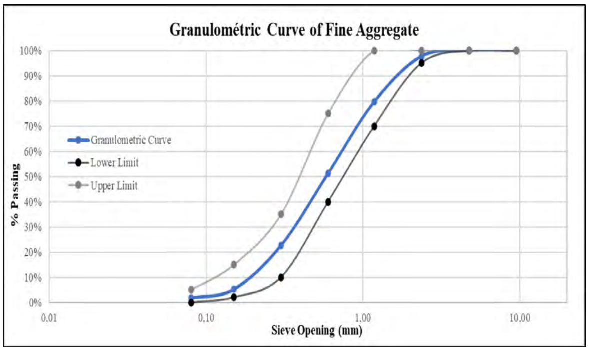


Figura 3. *Granulometric of aggregate*

Cement

Portland Pozzolanic Cement is a blended material composed of Portland Cement and pozzolanic additives, which are reactive compounds derived either from natural volcanic sources or synthetic processes **(22)**. This cement variant exhibits improved durability against chemical exposure, lower heat of hydration, and enhanced water resistance compared to standard Portland Cement.

In this study, Type N and HS Portland Cement, conforming to the specifications outlined in NTE 2380 **(23)**, **(24)**, was selected to evaluate its performance in mortar formulations.

"Maestro" Cement – Holcim Type N

Holcim "Maestro" Type N cement is an advanced, high-performance material specifically engineered for modern masonry applications. Its optimized formulation improves workability by up to 15% compared to traditional cements, effectively reducing material loss and rebound during application. Additionally, its superior waterproofing capability, ranging from 65% to 90%, makes it particularly well-suited for structures subjected to constant moisture exposure **(24)**.

"Campeón" Cement - UNACEM Type HS

"Campeón" HS cement is a high-sulfate-resistant hydraulic cement, certified under the NTE INEN 2380 standard to guarantee quality and reliability in construction. Its high fineness and precisely controlled composition enable the production of long-lasting concrete, particularly in aggressive environments with elevated sulfate concentrations in soils and water. This cement is especially suitable for applications such as mass concrete structures, soil stabilization, dams, and mortars that are easy to place and provide high-quality finishes **(25)**.

Ultrasound

Ultrasound has proven to be an efficient method for dispersing nanoparticles within cement-based matrices. Research has shown that ultrasonic treatment promotes a more uniform distribution of nanoparticles in the mortar, resulting in a denser and stronger microstructure. For instance, research **(26)** suggests that the application of ultrasound can promote the growth of C-S-H phases in cement paste, thereby improving mechanical strength and reducing porosity **(27)**.

Nanoparticles obtained from crab shell

The nanoparticles were synthesized at the UCE laboratories, where 1,000 grams of pure crab meal derived from exoskeletons were processed. Following a series of chemical treatments, 129 grams of crab-derived nanosilica were successfully extracted.

Composition of crab nanosilica through laboratory tests

Laboratory tests were performed to verify the composition of the nanoparticles extracted from crab exoskeletons and assess the effectiveness of the research. The analysis involved Energy Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and X-ray Diffraction (XRD). These characterization methods are essential for evaluating the properties and structural composition of the crab-derived nanoparticles.

Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) Testing

Crab Nanosilica

In Figure 4, the SEM images (a)-(b)-(c)-(d) offer a detailed analysis of the morphology and particle distribution within the sample, providing valuable insights into its structural characteristics. The combination of laminar and particulate structures, along with rough and fractured textures, suggests that the material has a complex nature, possibly with different phases: fibrous or porous. The variability in particle size and distribution may have significant implications for the material's physical and chemical properties, such as reactivity, porosity, and mechanical strength.

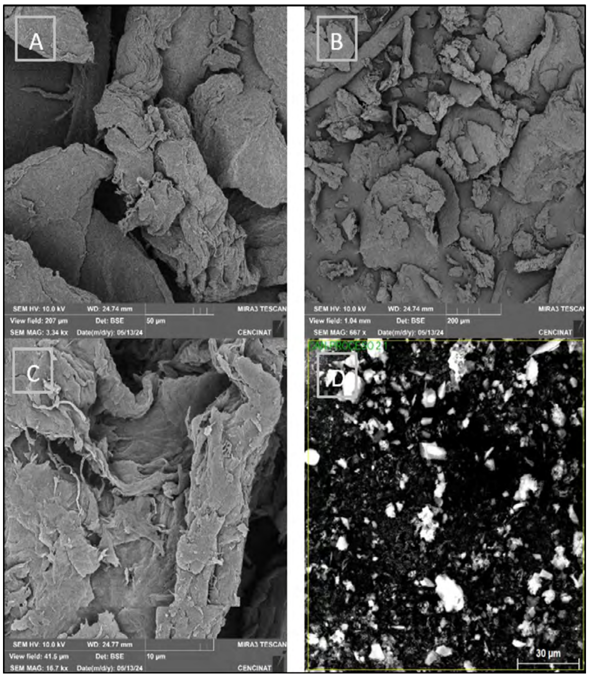


Figura 4. *Morphology and topography of crab nanosilica*

Energy dispersive spectroscopy (EDS) is an analytical technique used to characterize the chemical composition. It is particularly valuable in the analysis of nanomaterials, as it enables the detection and quantification of the elements present within the material.

As illustrated in Figure 5, the analysis identified and confirmed the presence of key elements:

carbon and oxygen as the predominant elements, evidenced by energy peaks in the 0.2 keV and 0.5 keV regions, respectively, corresponding to the K series. The analysis revealed the elemental composition of the sample, with carbon (60.34%) and oxygen (59.06%) being the predominant elements. Other elements such as sodium, magnesium, aluminum, silicon, phosphorus, potassium, calcium, titanium, and iron were present in smaller quantities. The relative atomic fraction percentages further confirm a uniform distribution of these elements within the sample, suggesting a well-distributed composition.

The findings indicate that the sample primarily consists of carbon and oxygen, which together represent a substantial portion of the total weight. Additionally, the presence of silicon, which constitutes 5.03% of the sample's weight, is particularly noteworthy. This suggests that the material may share similarities with silicon-containing compounds, such as silicates, likely due to the applied synthesis processes.

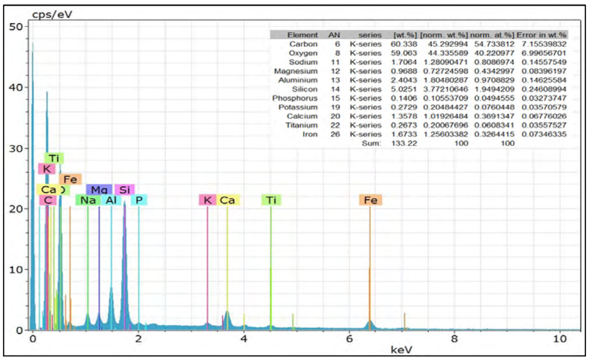


Figura 5. *Composition of crab nanosilica*

XRD Test

- **Crab nanosilica**

Figure 6 presents the XRD analysis illustrating a diffraction pattern typical of nanosilica synthesized from crab exoskeletons.

The analysis was conducted employing a diffractometer over a 2θ scanning range from 0 to 90°, at a scan rate of 0.02 degrees per second. Distinct diffraction peaks were observed around 2θ = 22° and 27°, characterized by their sharpness and high intensity, clearly indicating a high crystallinity level and confirming the presence of silica in its nanostructured form.

The XRD pattern identified as "CRAB NANOSILICA" distinctly features a prominent peak at approximately 22° on the 2θ scale, reflecting a high concentration of silica in nanostructured form. The accompanying minor peaks further suggest structural complexity and multiphase composition, characteristics typical of biologically derived materials and chemically synthesized composites. These findings are consistent with anticipated structural attributes for silica-mineral composites sourced from crab exoskeleton waste.

Moreover, the XRD spectrum exhibits several minor peaks distributed across the entire range, indicating the coexistence of multiple crystalline phases alongside possible amorphous structures within the material. The presence of these diverse phases emphasizes the significance of crystallinity in achieving the desired physical and chemical properties of nanosilica and related mineral compounds derived from crab exoskeletons.

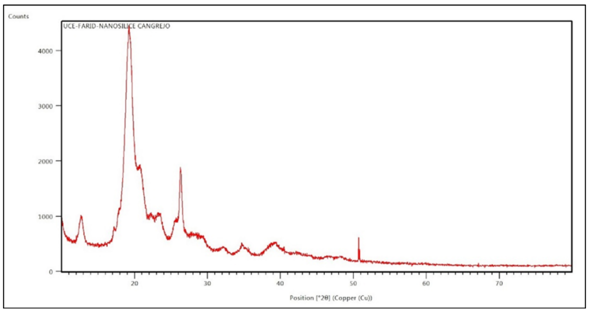


Figura 6. *Structural composition of crab nanosilica*

Transmission Electron Microscopy (TEM)

- Crab nanosilica

The TEM images presented in Figure 7 (a)-(b)-(c)-(d) confirm the successful synthesis of materials exhibiting typical nanometric structures, characterized by agglomerations and morphological variations.

In Figure 7A, a small agglomeration of particles with irregular morphology is observed. The variations in contrast suggest differences in thickness or density, indicating the coexistence of both large and small structures within a more uniform matrix. Figure 7B displays a large agglomeration of particles with a dense and complex structure. The irregular shape and regions of greater opacity imply the presence of multiple layers or phases in the material, suggesting potential interactions between the particles and the matrix. Figure 7C depicts a more compact agglomeration of particles with well-defined edges, suggesting a higher degree of structural organization within the sample. The structure exhibits an irregular morphology with a uniform opacity distribution, indicating consistency in material synthesis.

In contrast, Figure 7D presents a small agglomeration of particles with a simpler and less dense structure compared to Figure 7B. The variations in contrast suggest differences in composition or particle thickness, indicating a well-distributed composite structure.

The observed contrast variations among the particles correspond to nanoscale topographical differences, typically encountered in advanced materials synthesized via chemical or mechanical routes. Such nanoscale features are especially significant in applications requiring enhanced electrical conductivity and extensive surface area, including flexible electronic components and supercapacitor technologies.

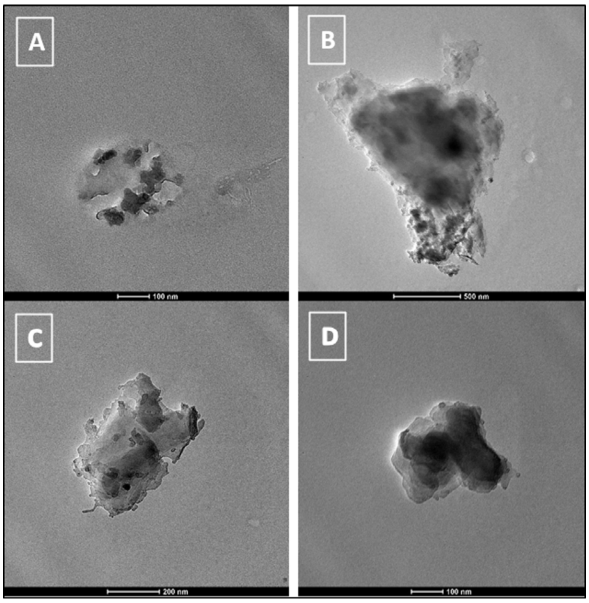


Figura 7. *Nanometric composition of crab silica*

Table 3. *Quantity of Specimens for the Compression Strength Test.*

No.	Mix Name	Description	Test	Number of Specimens
1	M	Standard Mix	Simple Compressive Strength.	30
2	$M+S_{0.25\%}$	Standard Mix + 0.25% Nanosilica		30
3	C	Standard Mix		30
4	$C+S_{0.25\%}$	Standard Mix + 0.25% Nanosilica		30
		Total		**120**

Table 4. *Quantity of Specimens for the Permeability Test.*

No.	Mix Name	Description	Test	Number of Specimens
1	M	Standard Mix	Permeability	1
2	$M+S_{0.25\%}$	Standard Mix + 0.25% Nanosilica		1
3	C	Standard Mix		1
4	$C+S_{0.25\%}$	Standard Mix + 0.25% Nanosilica		1
		Total		**4**

Methodological Design

The experimental methodology adopted in this study follows the guidelines established by INEN standards to guarantee precision and reproducibility in mortar and cement testing through systematically standardized protocols. Particularly, NTE INEN 2518 outlines optimal practices related to material proportioning and mixing procedures, ensuring consistent and reliable test outcomes. The correct dosage is verified using containers of known volume, with sand quantities adjusted as needed to maintain mortar consistency. To ensure homogeneity, the recommended procedure involves initially combining most of the water with a portion of the sand and cementitious materials rapidly and uniformly. Subsequently, the remaining components are gradually introduced into the mixture. Additionally, the standard prescribes a mixing duration of between 3 to 5 minutes after the final addition of water. The methodology also includes a retempering step to compensate for water loss through evaporation, thus preserving the mortar's required workability prior to application in construction processes (28).

Additionally, the procedure requires precise determination of material proportions, as outlined in Table 5. These proportions are essential for the proper preparation of specimens, which are critical in the design and evaluation phases of mortar production. Ensuring accurate material ratios enhances the reliability of test results, validating the performance and consistency of the mortar formulations.

Table 5. *Quantity of materials used for preparing 6 specimens*

Mix Name	W/C Ratio	Cement	Fine Aggregate	Water	Nanoparticles
		g	g	g	g
M	0.625	500.0	1596.2	312.5	-
$M+S_{0.25\%}$	0.625	498.7	1596.2	312.5	1.3
C	0.584	500.0	1596.2	292.0	-
$C+S_{0.25\%}$	0.584	498.7	1596.2	292.0	1.3

M: Control mortar mix with “Maestro cement” containing 0% nanoparticles.

$M+S_{0.25\%}$: Control mortar mix with 0.25% silicon nanoparticles.

C: Control mortar mix with “Campeón” cement containing 0% nanoparticles.

C+S0.25%: Control mortar mix with 0.25% silicon nanoparticles.

The mortar mixes were formulated while maintaining a constant w/c ratio; however, the nanoparticle content was adjusted based on percentages relative to the cement weight, as detailed in Table 3.

The formulation and proportioning of mortar constitute essential stages involving meticulous selection of materials, precise mixing procedures, and rigorous testing protocols. Mortar quality significantly influences the durability and functional performance of masonry constructions. By adhering to the specified methodology, mortars are designed to meet established standards tailored to particular applications, ensuring an optimal combination of mechanical strength and adequate workability.

Permeability Test

Drop Method

Several investigations have employed drop tests to evaluate permeability properties in mortars and concretes (29) , for example, conducted studies utilizing contact angle measurements to examine the wettability of mortar constituents, including cement and aggregates. Their results demonstrated that the contact angle directly correlates with the absorption characteristics of these granular materials, significantly affecting critical mortar properties such as workability and mechanical performance upon hardening. Additionally, the drop test effectively predicts the resistance of cementitious materials to penetration by liquids, such as water and aggressive chemical agents, establishing it as an essential criterion for durability assessment in concrete and mortar applications (30).

Lower contact angles indicate higher wettability, which corresponds to greater permeability. This relationship occurs because smaller contact angles facilitate the spreading of liquid—typically water—over the mortar surface, thus enhancing its ability to penetrate the material’s microstructure (29). Hence, the contact angle is critical in distinguishing between hydrophobic and hydrophilic surfaces, offering insights into the effectiveness of surface modifications applied to nanosilica particles (8).

III. RESULTS

Uniaxial compressive strength

This test is among the most widely used methods for evaluating the compressive strength of mortar. It is typically conducted using mortar cubes, commonly with dimensions of 50 mm, to assess the material’s capacity to resist compressive loads. The INEN 488 standard is frequently utilized as the reference methodology for this assessment (30).

Table 6 summarizes the compressive strength results obtained for the different mortar mixtures evaluated, recorded across multiple curing periods. These data provide insight into the mechanical performance evolution of each formulation over time.

Table 6. *Summary of Compressive Strength of Mortars.*

Mix Name	24 hours	3 days	7 days	28 days	56 days	90 days
	MPa	MPa	MPa	MPa	MPa	MPa
M	1.11	3.00	4.85	6.55	7.16	7.88
$M+S_{0.25\%}$	1.13	2.94	4.31	6.45	7.52	8.13
C	4.12	12.41	15.90	23.67	28.36	31.63
$C+S_{0.25\%}$	5.21	12.74	15.76	24.00	28.57	31.22

Table 6 illustrates the evolution of compressive strength in mortar mixes incorporating nanosilica ($S_{0.25}$%), compared to conventional mixes (M and C) across different curing periods, ranging from 24 hours to 90 days.

Among the tested formulations, the C+$S_{0.25}$% mix consistently exhibited the highest compressive strength at all curing ages, reaching 31.22 MPa at 90 days, which represents a slight increase relative to the unmodified C mix (31.63 MPa). In the case of Type N cement, the nanosilica-modified mix (M+$S_{0.25}$%) demonstrated superior long-term performance, achieving a compressive strength of 8.13 MPa at 90 days, reflecting a 3.2% improvement compared to the reference mix (M: 7.88 MPa).

The observed early-age strength development (between 24 hours and 7 days) in the C+$S_{0.25}$% mix suggests a favorable interaction between nanosilica and cement hydration products, resulting in matrix densification and enhanced particle cohesion. These findings confirm that the incorporation of nanosilica contributes to higher compressive strength and long-term durability, establishing it as an effective nanomaterial for optimizing the mechanical performance of cement-based mortars.

Compressive Strength Analysis Against Time

Figure 8 depicts a comparative analysis of the compressive strength development over time for mortar samples formulated with "Campeón" Cement, evaluating differences between the reference composition (C) and the modified mixture incorporating 0.25% nanosilica (C+$S_{0.25}$%) over various curing ages (24 hours to 90 days).

At early curing stages (24 hours to 7 days), both formulations exhibit comparable strength development, suggesting that nanosilica does not significantly influence the initial hydration process. However, from 28 days onwards, the C+$S_{0.25}$% mix demonstrates a gradual increase in compressive strength, surpassing the reference mix and reaching 31.22 MPa at 90 days, representing a slight improvement compared to the unmodified C mix (31.63 MPa).

This trend indicates that nanosilica contributes to the enhancement of microstructural integrity within the cementitious matrix, leading to greater durability and improved long-term mechanical performance. These results verify that the incorporation of nanosilica significantly improves compressive strength and matrix densification, supporting its suitability as an effective additive in the development of high-performance cement-based materials.

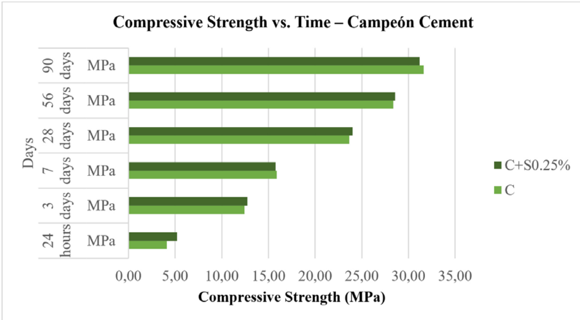


Figura 8. *Compressive Strength vs Time – "Campeón" Cement*

Figure 9 illustrates the compressive strength evolution of mortars prepared with Campeón Cement, comparing the reference mix (C) and the modified mix incorporating 0.25% nanosilica (C+$S_{0.25}$%) over various curing periods (24 hours to 90 days).

During early curing stages (24 hours to 7 days), both formulations exhibit similar trends with minor variations, suggesting that nanosilica does not significantly impact the initial hydration process. However, beyond 28 days, the C+$S_{0.25}$% mix shows a gradual increase in compressive strength, reaching 31.22 MPa at 90 days, which represents a slight decrease compared to the unmodified mix (31.63 MPa).

The mechanical behavior at later curing ages indicates that nanosilica enhances hydration reactions, matrix densification, and particle cohesion, contributing to greater durability and long-term strength stability. These findings confirm that nanosilica improves the long-term compressive strength and durability of cementitious materials, reinforcing its potential as a viable additive for high-performance construction applications.

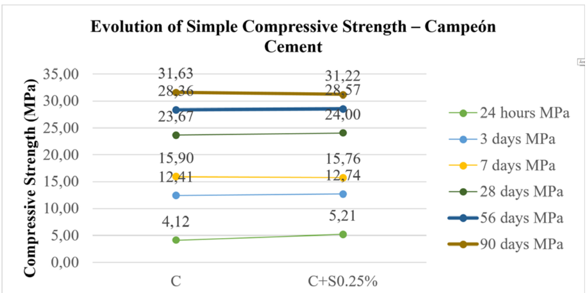


Figura 9. *Evolution of Compressive Strength – "Campeón" Cement*

Figure 10 presents a comparative analysis of compressive strength for mortars made with Maestro Cement (M) and a mortar incorporating 0.25% nanosilica (M+$S_{0.25}$%), evaluated at curing periods of 24 hours, 3, 7, 28, 56, and 90 days. At early curing ages (24 hours to 7 days), both mortar formulations exhibit comparable performance, indicating that nanosilica does not significantly influence the initial hydration process. However, from 28 days onwards, the $M+S_{0.25}\%$ mix demonstrates superior compressive strength compared to the reference mix, attaining 8.13 MPa at 90 days, which represents a slight increase relative to the M mix (7.88 MPa). The improved mechanical performance observed at later ages suggests that nanosilica contributes to matrix densification and enhanced particle cohesion, ultimately leading to greater long-term durability. These findings confirm that nanosilica promotes the progressive development of compressive strength, establishing it as an effective nanomaterial for enhancing the mechanical properties of cement-based mortars.

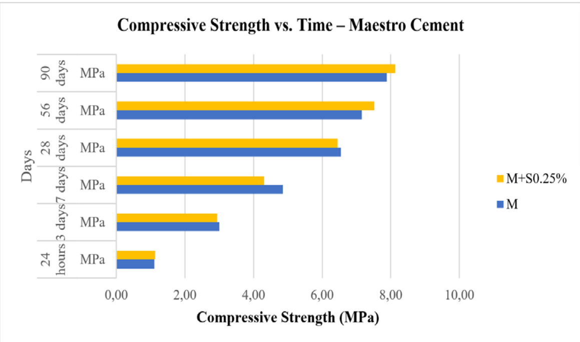


Figura 10. *Compressive Strength vs Time – "Maestro" Cement*

Figure 11 illustrates the progression of compressive strength in mortars formulated with Maestro Cement, comparing a control mixture (M) with a mixture containing 0.25% nanosilica (M+S0.25%). Results indicate that the inclusion of nanosilica consistently enhances compressive strength at advanced curing ages (28, 56, and 90 days)

During early curing ages (24 hours to 7 days), both mixes exhibit similar trends with minor variations, suggesting that nanosilica does not significantly influence initial strength development. However, beyond 28 days, the $M+S_{0.25}\%$ mix demonstrates a progressive increase in compressive strength, reaching 8.13 MPa at 90 days, indicating a slight increase relative to the reference sample (7.88 MPa).

The enhancement in compressive strength observed at advanced curing periods suggests that nanosilica facilitates more effective cement hydration and improved microstructural densification. Consequently, these characteristics lead to superior durability and enhanced long-term mechanical integrity. The results thus confirm nanosilica's effectiveness in elevating the long-term compressive strength of mortars, validating its potential as a valuable additive for improving cementitious materials within sustainable construction contexts.

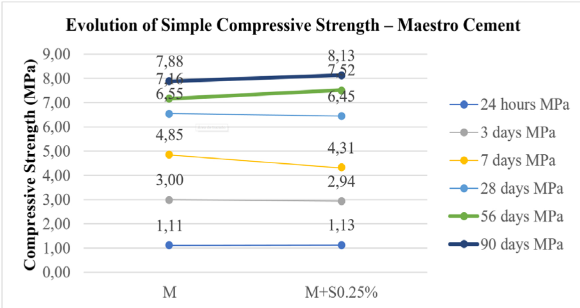


Figura 11. *Evolution of Compressive Strength – "Maestro" Cement*

Scanning Electron Microscopy (SEM)

SEM micrographs reveal a densely compacted granular microstructure with uniformly distributed particles of diverse sizes. This homogeneous particle arrangement indicates reduced porosity, a key attribute enhancing the mechanical integrity and long-term durability of the material.

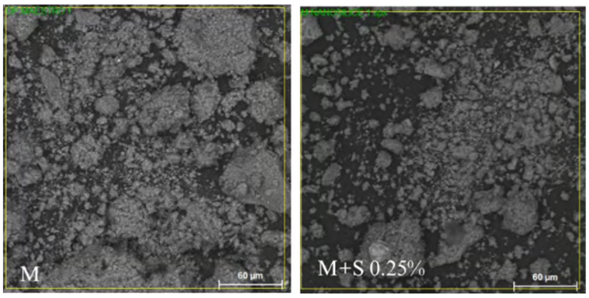


Figura 12. *Morphology and topography of type N cement mixtures with Crab Nanoparticles.*

- **$M+S_{0.25}\%$:** Nanosilica exhibits high pozzolanic reactivity, facilitating the formation of additional C-S-H gel, which enhances both mechanical strength and durability.

A comparable analysis was performed on mixtures containing Campeón cement, revealing significant densification improvements and porosity reduction attributed to the incorporation of nanomaterials.

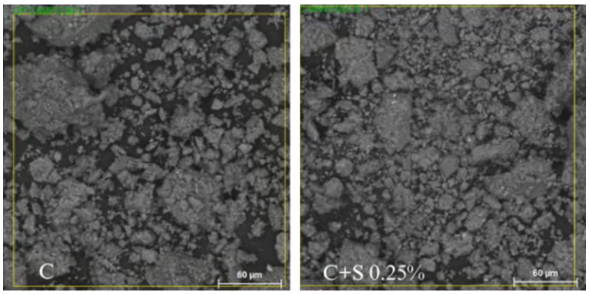


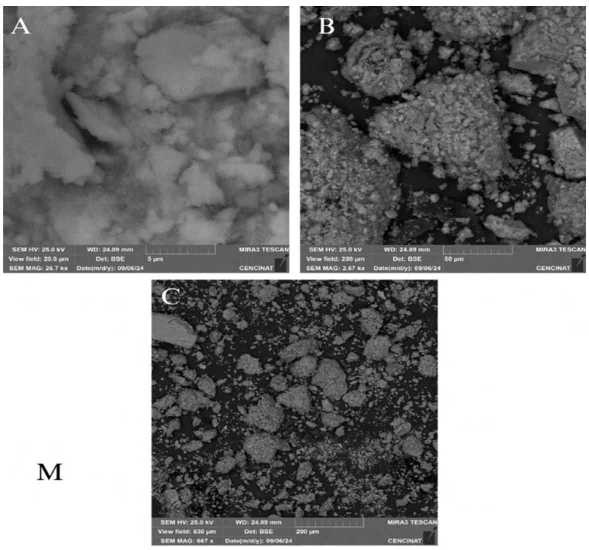
Figura 13. *Morphology and topography of type HS cement mixtures with Crab Nanoparticles.*

- **C:** Displays a heterogeneous microstructure with particles of varying sizes, a typical characteristic of cementitious mixtures.
- **C+S$_{0.25}$%:** The uniform dispersion of nanosilica enhances the formation of C-S-H gel, leading to improved strength and durability of the concrete.

Transmission Electron Microscopy (TEM)

TEM micrographs provide detailed information regarding the atomic-scale distribution within nanomaterials, allowing precise characterization of their structural composition, ranging from 5 µm to 200 µm. These images reveal a more uniform dispersion of particles and a notable decrease in porosity. The resulting denser and more compact microstructure indicates enhanced durability and greater long-term resistance to both mechanical stress and environmental factors.

The nanometric structure observed through TEM is presented in Figure 14 and Figure 15, evidencing matrix densification and enhanced particle cohesion due to nanosilica incorporation



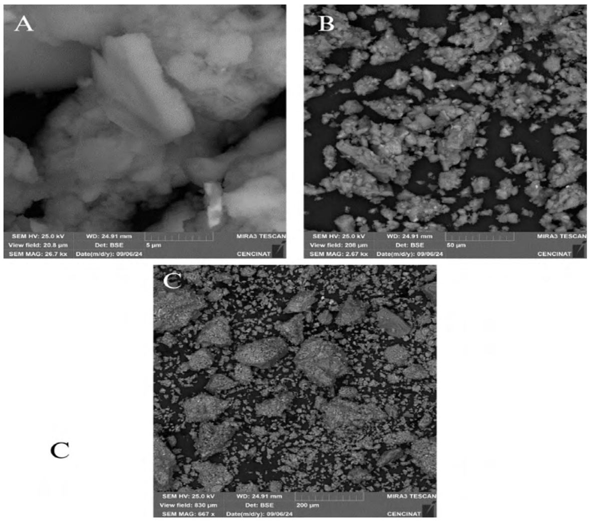


Figura 14. *Nanometric Composition of mixtures.*

- **Base Samples M and C: A** loosely packed and porous microstructure, indicating low particle cohesion and diminished mechanical strength.

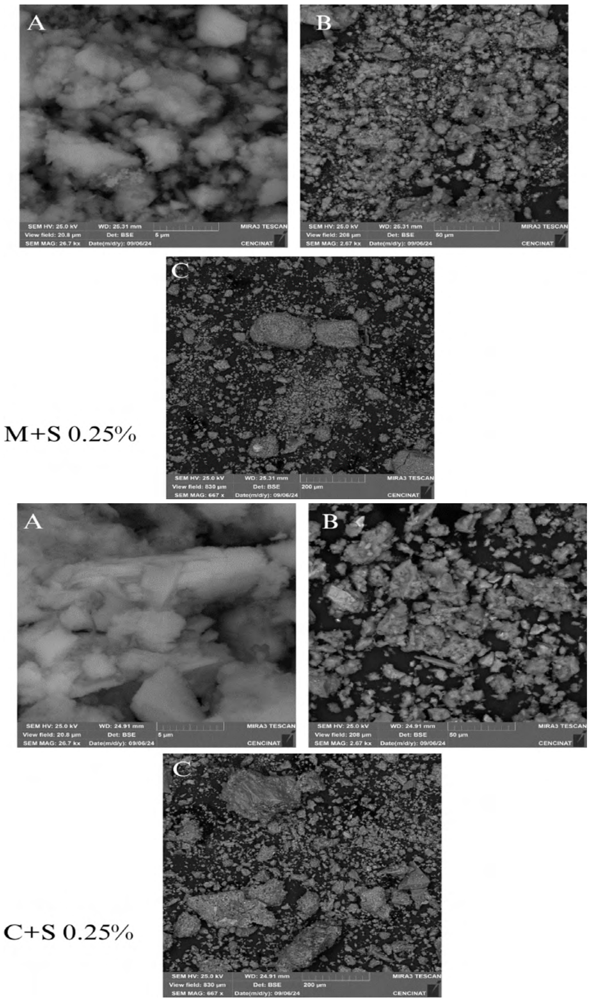


Figura 15. *Figure 15. Nanometric Composition of mixtures with Crab Particles.*

- **C+S$_{0.25}$% and C+S$_{0.75}$%:** A highly compact and cohesive microstructure, resulting in a significant reduction in porosity.

X-Ray Diffraction (XRD)

XRD analysis facilitates the **identification and validation of crystalline phases** present in the mixtures.

The XRD patterns of the modified mixtures are shown in Figure 16 and Figure 17, confirming the presence of additional crystalline phases associated with C–S–H formation.

- **Samples with nanosilica:** Higher presence of crystalline phases and pozzolanic reactivity, reinforcing material strength and stability.

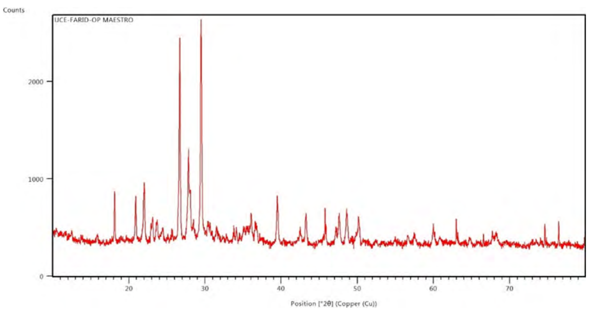


Figura 16. *Nanometric composition of mixtures of type N cement.*

- **M+S$_{0.25}$%: A** Multiple additional peaks identified between 40º and 50º (2θ), indicating an increased presence of crystalline phases. Nanosilica enhances pozzolanic reactivity, improving structural cohesion and material strength.

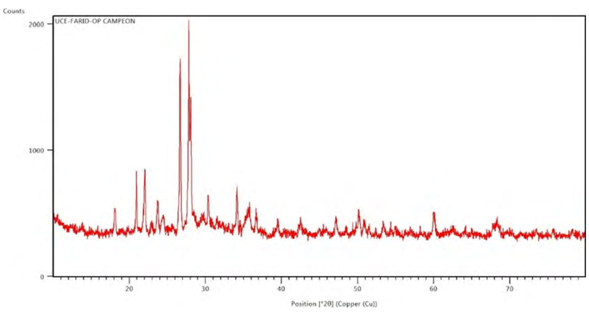


Figura 17. *Nanometric composition of mixtures of type HS cement.*

- **C+S$_{0.25}$%:** Multiple well-defined peaks detected between 28º and 50º (2θ), suggesting a higher presence of crystalline phases and better structural organization. Nanosilica promotes the formation of additional hydration products, enhancing mechanical strength.

Permeability

Figure 18 presents the contact angle analysis for mortars prepared with Campeón Cement (A) and Campeón Cement modified with 0.25% nanosilica (C+S$_{0.25}$%) (B).

A slight reduction in the contact angle is observed in the nanosilica-modified mix, indicating increased wettability and higher water absorption compared to the reference mixture. This suggests a moderate decrease in the hydrophobicity of the cementitious matrix, which may influence porosity and permeability against aggressive agents.

These findings indicate that while nanosilica enhances mechanical properties, its effect on surface hydrophobicity can vary depending on cement composition and curing conditions. However, the nano-filler effect of nanosilica contributes to matrix densification, which may still lead to improved long-term durability and resistance to degradation in cementitious materials. Additionally, the particle dispersion properties of nanosilica can optimize hydration reactions, potentially enhancing the structural integrity of mortar under diverse environmental conditions.

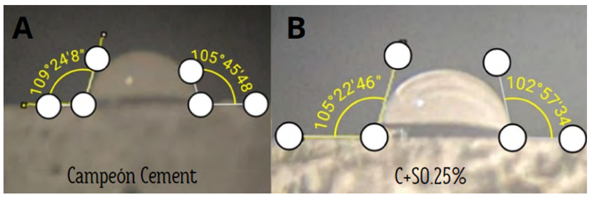


Figura 18. *Permeability Assessment Using the Drop Method Test – "Campeón" Cement*

Figure 19 illustrates the contact angle measurements obtained using the drop method on various mortar samples modified with nanosilica, compared to a reference cement sample. The contact angle serves as a key parameter for assessing surface hydrophobicity, reflecting the material's ability to repel water.

Figure 19 presents a detailed contact angle analysis comparing Maestro Cement (A) with the nanosilica-modified Maestro Cement 0.25% nanosilica (M+S$_{0.25}$%) (B). The nanosilica-enhanced sample demonstrates a significant rise in contact angle compared to the reference cement, reflecting increased hydrophobicity and reduced water absorption. This finding indicates that the inclusion of nanosilica effectively improves the water-repellent characteristics of the mortar, thereby potentially lowering permeability and enhancing its resistance against moisture-induced deterioration.

These results verify nanosilica's role as a hydrophobic modifier, effectively minimizing water interaction with the cement matrix. Such behavior likely enhances material durability by restricting moisture penetration, especially beneficial for structures subjected to harsh environmental exposure. Moreover, nanosilica incorporation may also strengthen adhesion and cohesion within the cementitious matrix, further improving mechanical performance over extended periods.

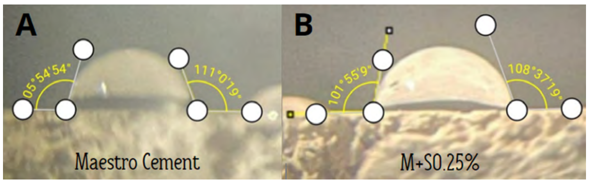


Figura 19. *Permeability Assessment Using the Drop Method – "Maestro" Cement*

Table 7 summarizes the permeability test results obtained using the drop method for the four mortar samples, emphasizing the impact of different nanoparticles on the material's behavior and performance.

Table 7. *Contact Angle Measurement.*

Mix Type	Contact Angle	Surface Type
M	108.3°	Hydrophobic > 90°
M+$S_{0.25\%}$	105.0°	Hydrophobic > 90°
C	107.5°	Hydrophobic > 90°
C+$S_{0.25\%}$	104.1°	Hydrophobic > 90°

The results indicate that all mixtures exhibit hydrophobic surfaces, suggesting low water absorption. However, the Campeón Cement mix with 0.25% nanosilica (C+$S_{0.25}$%) records the lowest contact angle (104.1°) compared to the reference mix (107.5°). This slight reduction implies a minor decrease in water repellency, potentially leading to increased permeability relative to the unmodified cementitious matrix.

These findings suggest that while nanosilica improves mechanical properties, its effect on surface hydrophobicity is minimal. Nevertheless, its matrix densification effect may still contribute to long-term durability and resistance to environmental degradation. The decrease in contact angle suggests a moderate increase in wettability, which could impact capillary absorption. Further analysis is required to determine its influence on moisture resistance and performance under aggressive environmental conditions.

Sustainability Analysis

The reduction of CO_2 emissions associated with the utilization of nanoparticles derived from crab waste was evaluated. In Ecuador, approximately 8.0 tons of crab waste are generated annually, contributing to an estimated 3,856 tons of CO_2 emissions.

An analysis was conducted to assess the CO_2 emissions resulting from the production of mortars incorporating these nanoparticles, aiming to quantify their potential environmental benefits in terms of carbon footprint reduction.

. CO_2 Emissions Associated with Crab Waste							
Chemical production	Price	Mortar components			TOTAL	CO_2 from Decomposition	% CO_2 Emissions
		Crab waste flour	Chemical process	CEMENT			
	$/kg	kg CO_2	kg CO_2	kg CO_2	kg CO_2	kg CO_2	%
Nanosílica	418.4	167	504	900	1571	3856	41%

Table 8. *Table 8. Sustainability Assessment and CO_2 Emissions Associated with Crab-Derived Nanosilica.*

Environmental Impact of Nanomaterials

The sustainability analysis (Table 8) shows a 41% reduction in CO_2 emissions resulting from the use of nanosilica derived from crab shell waste.

- Nanosilica derived from crab waste contributes to a 41% reduction in CO_2 emissions.

The chemical production process and mortar components, including cement replacement, significantly lower the carbon footprint compared to conventional materials.

The findings highlight a beneficial effect on reducing greenhouse gas emissions, thereby promoting circular economy principles through the recycling of waste materials within the construction sector. This strategy simultaneously advances environmental sustainability and enhances the mechanical properties of cementitious composites.

IV. DISCUSSION

The results obtained demonstrate that the incorporation of nanosilica synthesized from crab shells has a positive effect on the physical–mechanical properties and durability of the evaluated cement mortars. The addition of 0.25% nanosilica by cement weight produced significant increases in compressive strength, particularly at early ages, which is associated with the acceleration of cement hydration and the formation of a denser and more homogeneous cementitious matrix. This behavior is consistent with findings reported in the literature on the use of silica nanoparticles, where their high specific surface area acts as nucleation sites for C–S–H gel, promoting pore filling and reducing capillary porosity.

Microstructural analysis through SEM and XRD confirmed the presence of hydration products with a more uniform distribution, as well as a reduced amount of free portlandite, suggesting more efficient consumption of the available calcium hydroxide. Additionally, permeability tests and contact angle values greater than 90° confirmed a hydrophobic behavior, which contributes to reduced water absorption and, consequently, greater material durability against aggressive agents.

From a sustainability perspective, the utilization of biological waste as a raw material for obtaining nanosilica represents a viable alternative aligned with the principles of the circular economy, reducing the carbon footprint and minimizing the environmental impact associated with the production of construction materials. However, it is recognized that variability in the chemical composition of the waste and the need to optimize synthesis processes may influence the reproducibility of the results.

Future studies should focus on assessing the performance of these mortars under more severe exposure conditions, as well as analyzing the interaction of nanosilica with other additives and supplementary materials, in order to broaden its application range and ensure consistent long-term performance.

V. CONCLUSIONS

Nanosilica demonstrates superior long-term efficiency, contributing significantly to the mechanical enhancement of mortar over extended curing periods.

The incorporation of nanosilica derived from crab shell waste not only improves mortar performance but also serves as a sustainable strategy, aligning with circular economy principles by repurposing industrial byproducts and reducing environmental impact. Compressive strength results confirm that nanosilica-modified mortars achieve high mechanical resistance, competing closely with other nanoparticle-enhanced formulations, ensuring structural durability.

Water absorption tests indicate that nanosilica reduces permeability, maintaining absorption levels below 10%, which is crucial for enhancing moisture resistance and durability in aggressive environments. Hydrophobicity measurements reveal that nanosilica-modified mortars exhibit contact angles exceeding 90°, confirming improved water repellency, which helps mitigate corrosion risks and chemical degradation.

The obtained results confirm that nanosilica meets the requirements of the NTE INEN 2518 standard, validating its technical feasibility for integration into high-performance structural applications.

Future research should explore alternative sources of nanosilica from biological waste, optimizing its processing methods to enhance its efficiency and applicability in sustainable construction. The use of nanosilica in mortar formulations contributes to reducing cement consumption, lowering CO_2 emissions, and fostering the development of eco-friendly construction solutions.

Overall, nanosilica proves to be a viable nanomaterial for improving mechanical strength, water resistance, and long-term performance in cementitious composites, reinforcing its importance in sustainable and high-performance construction applications.

VI. AGRADECIMIENTOS

We would like to express our deepest gratitude to the Nanoscience and Nanotechnology Center of the Armed Forces University (ESPE) and its laboratory team for their valuable technical support and dedication throughout the development of this project. We also extend our thanks to the staff of the Ecuadorian Institute of Cement and Concrete (INECYC) for their collaboration in conducting specialized tests. Likewise, we are grateful to the laboratories of Material Testing and Chemical Sciences at the Central University of Ecuador (UCE) for their commitment and professionalism in the sample analysis. Finally, we express our gratitude to the Ministry of the Interior for their support, which was essential for the successful completion of this research.

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