Incorporation of Nanochitin in Cement Mortars: An Approach to Enhancing Durability and Sustainability

Incorporación de Nanoquitosano en Morteros de Cemento: Un Enfoque para Mejorar la Durabilidad y la Sostenibilidad

INVESTIGACIÓN Y DESARROLLO FIGEMPA

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ABSTRACT

This study explores the use of nanochitin extracted from crab shell waste to enhance the mechanical and durability properties of cement-based mortars. Nanochitin, a biopolymer derived from chitin, has been identified as a promising nanomaterial additive that improves compressive strength, cohesion, and workability of cementitious composites. The methodology involved the synthesis, characterization, and incorporation of nanochitin in mortars using Type N and Type HS cements. The assessment of mechanical performance was conducted through uniaxial compression tests, permeability analysis, and microstructural characterization using Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD). The results indicate that nanochitin enhances hydration, contributing to an optimized cementitious matrix. The modified mortars exhibited higher compressive strength, reaching 9.18 MPa at 90 days in Type N cement. Furthermore, nanochitin demonstrated superior rheological properties, allowing for improved workability and water retention, particularly in arid environments. This study highlights the sustainability benefits of repurposing crab shell waste, aligning with circular economy principles and advancing the development of eco-friendly construction materials.

Palabras claves: Nanochitin, mortar, internal cohesion, rheology, sustainability.

RESUMEN

Este estudio explora el uso de nanoquitosano extraído de residuos de caparazones de cangrejo para mejorar las propiedades mecánicas y de durabilidad de morteros a base de cemento. El nanoquitosano, un biopolímero derivado de la quitina, ha sido identificado como un aditivo nanomaterial prometedor que mejora la resistencia a la compresión, la cohesión y la trabajabilidad de los compuestos cementicios. La metodología incluyó la síntesis, caracterización e incorporación del nanoquitosano en morteros utilizando cementos Tipo N y Tipo HS. La evaluación del desempeño mecánico se llevó a cabo mediante ensayos de compresión uniaxial, análisis de permeabilidad y caracterización microestructural mediante Microscopía Electrónica de Barrido (SEM) y Difracción de Rayos X (XRD). Los resultados indican que el nanoquitosano mejora la hidratación, contribuyendo a una matriz cementicia optimizada. Los morteros modificados mostraron una mayor resistencia a la compresión, alcanzando los 9.18 MPa a los 90 días con cemento Tipo N. Además, el nanoquitosano demostró propiedades reológicas superiores, permitiendo una mejor trabajabilidad y retención de agua, especialmente en ambientes áridos. Este estudio destaca los beneficios en términos de sostenibilidad del reaprovechamiento de residuos de caparazones de cangrejo, en línea con los principios de economía circular y promoviendo el desarrollo de materiales de construcción ecológicos.

Keywords: Nanoquitosano, mortero, cohesión interna, reología, sostenibilidad.

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INTRODUCTION

The integration of nanoparticles in construction materials has led to significant advancements in mechanical performance, durability, and sustainability. Among these nanomaterials, nanosilica has been extensively studied for its ability to enhance compressive and tensile strength, improve microstructure, and accelerate cement hydration. Research findings indicate that replacing 1.5% of cement with nanosilica optimizes early-age strength and long-term durability, making it a valuable additive in high-performance concrete (HPC) (Alvansaz *et al.*, 2022b).

The use of biopolymer-based nanomaterials, such as nanochitin, has emerged as an eco-friendly alternative in construction. Biopolymers have been recognized as viable alternatives for optimizing construction materials due to their mechanical properties and contribution to sustainability (Alvansaz Yazdi *et al.*, 2014). Nanochitin, derived from crustacean exoskeletons, has been incorporated into cement mortars to improve internal cohesion and rheology, leading to lower porosity and greater resistance to external agents (Lee *et al.*, 2023). Studies indicate that nanochitin enhances water retention capacity in cementitious mixtures, thereby promoting hydration and contributing to uniform hardening (Haider *et al.*, 2022b). Furthermore, its incorporation improves mortar workability without compromising mechanical performance (Ma and Li, 2013). The reduction of cracks and improved adhesion between components position nanochitin as a promising material for developing more durable and resilient construction materials (Zhang *et al.*, 2023).

Another critical innovation is the application of hydrophobic nanosilica in cementitious materials, which has been shown to enhance corrosion resistance and improve overall durability. Research suggests that replacing 2% of cement with nanosilica increases hydration rates, mechanical strength, and water repellency, which is essential for prolonging the lifespan of concrete structures (Alvansazyazdi et al., 2023). Nanotechnology in concrete has also advanced through the incorporation of nanomaterials such as nano-SiO_2, TiO_2, and CNTs, which contribute to reducing microporosity, enhancing cement hydration, and improving structural integrity. These nanoparticles act as reinforcing agents, significantly improving crack resistance and mechanical performance, making them highly effective for modern high-performance concrete applications (Alvansaz *et al.*, 2022b).

Furthermore, nano- and microsilica have been integrated into concrete pavers as partial cement replacements, demonstrating substantial improvements in compressive strength and durability. Studies show that replacing 3% of cement with nanosilica increases compressive strength by 12%, while a combined mix of 15% microsilica and 3% nanosilica results in a 23% strength improvement, contributing to sustainable construction practices and efficient material utilization (Alvansaz *et al.*, 2022a).

These advancements in nanoparticle applications, particularly in nanosilica, nanochitin, and hybrid nanomaterials, underscore the potential of nanotechnology in optimizing construction materials. Their ability to enhance strength, improve durability, and reduce environmental impact highlights their importance in developing high-performance and sustainable infrastructure.

Crab Shell-Derived Nanochitin

Background

The use of nanoparticles in construction has evolved significantly in recent years, enabling improvements in mechanical properties, durability, and sustainability of cementitious materials. One of the most studied approaches is the incorporation of silica nanoparticles in high-strength concrete, which has led to reductions in porosity, improvements in compressive strength, and a decrease in cement consumption, thereby contributing to a lower environmental impact (Morales *et al*., 2020).

The application of nano and microsilica in high-performance concrete (HPC) has demonstrated significant structural improvements. In particular, replacing 15% of cement with microsilica has been found to provide greater mechanical strength enhancements than a combination of 15% microsilica and 3% nanosilica, promoting a reduction in cement consumption and fostering sustainable construction practices (Tapia Vargas *et al.*, 2024).

Other nanomaterials, such as nano-iron, have been evaluated in cement mortars, showing benefits in terms of increased mechanical strength and reduced permeability. However, its incorporation may affect the workability of the mixture due to particle agglomeration, highlighting the need for appropriate dispersion techniques to optimize its performance (Alvansanzyazdi *et al.*, 2024c). Additionally, the use of nano-silica combined with polypropylene fibers and 4D metallic fibers has been explored to minimize cracking in high-performance concrete. Nano-silica densifies the cement matrix, reducing microcracking and improving the durability of structures. The inclusion of fibers further enhances the concrete's ability to withstand tensile and flexural stresses, preventing premature failures (Alvansazyazdi *et al.*, 2024a).

Recent studies have also highlighted the importance of nano-silica in cement mortars, demonstrating that its incorporation significantly improves compressive strength and reduces permeability. The addition of 0.75% nano-silica in mortars has resulted in a 61% increase in compressive strength at 7 days, while a 1.25% dosage has shown a 14% improvement at 28 days, solidifying its role as a key material in the optimization of the construction industry (Alvansazyazdi *et al.*, 2024b)

In the field of biopolymers, nanochitin, a material derived from crustacean exoskeletons, has emerged as a promising additive for improving cement mortars. Recent research has demonstrated that nanochitin enhances mechanical strength, internal cohesion, and adhesion in cementitious materials, positioning it as a key resource for the development of sustainable construction technologies(Alvansaz Yazdi *et al.*, 2014). Its biodegradable nature and renewable origin further reinforce its significance in eco-friendly construction (Lee *et al.*, 2023; Haider *et al.*, 2022b).

A study conducted by the University of Washington revealed that incorporating nanochitin into cementitious mixtures can increase compressive strength by up to 30%, due to the interaction between chitin and cement hydration products (Fu *et al.*, 2022). Additionally, nanochitin acts as a reinforcing agent at the nanostructural level, promoting better distribution of the cementitious phase and reducing the internal porosity of the mortar (Žižlavský *et al.*, 2020; Zhang *et al.*, 2023).

The use of nanochitin in the construction industry is also associated with improved mortar workability, allowing for more homogeneous mixtures with greater water retention. This factor is particularly crucial in arid climates, where rapid moisture loss can negatively impact the strength and durability of the material. Furthermore, electron microscopy studies have revealed that nanochitin contributes to a more compact cement microstructure, effectively reducing capillary absorption and water permeability (Aher *et al.*, 2023; Haider *et al.*, 2022a).

These advancements in nanotechnology applied to construction highlight the potential of nanomaterials and biopolymers to enhance structural performance, sustainability, and efficiency of cementitious materials, positioning them as innovative solutions for the construction industry.

Synthesis Methods

Crab Shell Waste

The transformation of crab exoskeletons into powder involves a systematic approach, as illustrated in Figure 1.

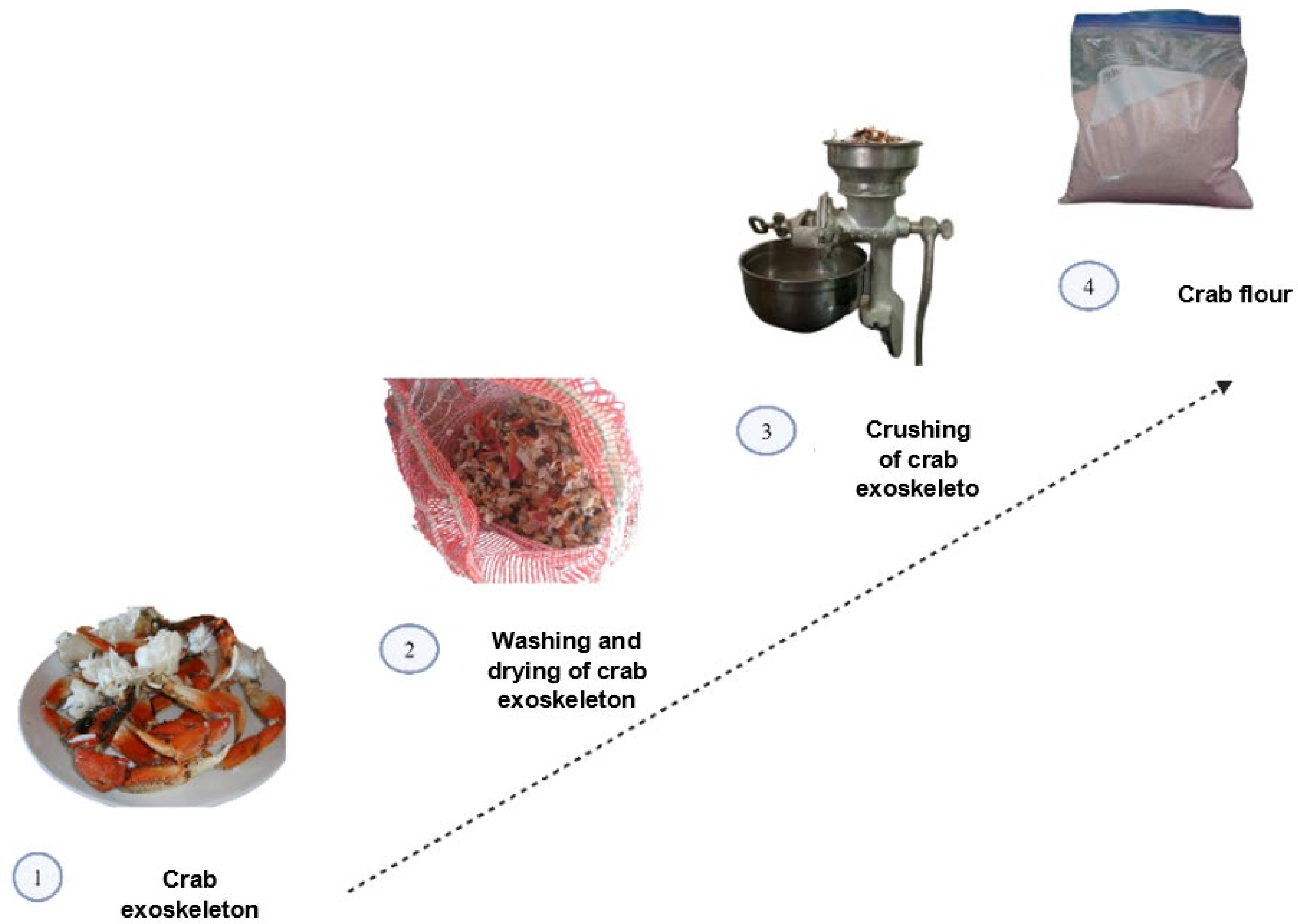


FIGURE 1
Obtaining of Crab flour

The primary phases of this methodology include:

1. **Selection of Raw Material:** Crab shells with minimal residual organic matter are carefully chosen to ensure high-quality processing.
2. **Cleaning and Dehydration:** The selected shells undergo an intensive washing process using water to eliminate any organic impurities and external contaminants. Subsequently, they are placed in a well-ventilated area for natural air drying over a period of five days to achieve optimal moisture reduction.
3. **Size Reduction:** Once dried, the shells are subjected to a grinding process using a manually operated cast-iron mill, ensuring the production of a fine, homogeneous powder suitable for further applications.

Chemical Methods

The chemical synthesis of nanochitosan from crab shell waste requires an optimized extraction process to enhance the mechanical performance and durability of mortars, supporting advancements in sustainable construction. This study focuses on a comprehensive technical evaluation of various extraction methods to identify the most efficient approach.

The primary aim is to assess the process in terms of complexity, cost-effectiveness, and material quality, ensuring its feasibility for large-scale production and its suitability for practical applications in the construction industry. Through this analysis, the study seeks to establish a sustainable and scalable method for incorporating nanochitosan into cementitious materials.

Nanochitin from crab

This experimental procedure was adapted from the methodology outlined by Haider *et al*. (2022b). Adjustments were implemented to accommodate the specific characteristics of the sample, ensuring the experimental approach yielded relevant and applicable results.

Nanochitin Extraction Process (Figure 2)

1. **Suspension Preparation:** A suspension was prepared by dispersing 50 g of oven-dried crab meal in 2500 ml of distilled water. To facilitate the reaction process, 5g of sodium bromide (NaBr) was introduced into the solution.

2. **Oxidation Process:** The reaction was initiated by adding 603 ml of sodium hypochlorite (NaClO) at a concentration of 9.5 mmol per gram of chitin. The pH of the mixture was carefully maintained between 10.0 and 11.0 through the gradual addition of 0.5 M sodium hydroxide (NaOH), creating an optimal alkaline environment for the reaction. The process was sustained for 24 hours at room temperature.

3. **Reaction Termination and Purification:** The oxidation reaction was halted by the addition of ethanol, and the pH was subsequently adjusted to a neutral value (pH 7.0) using hydrochloric acid (HCl). The mixture then underwent a three-day dialysis process against distilled water to eliminate residual impurities and undesired by-products.

4. **Mechanical Disintegration and Drying:** The purified suspension was subjected to mechanical disintegration using an Ultra-Turrax homogenizer (IKA) to achieve the desired particle size reduction. Finally, the processed material was dried in an oven at a controlled temperature ranging between 105 and 110 °C, ensuring optimal purity and stability of the final nanochitin product.

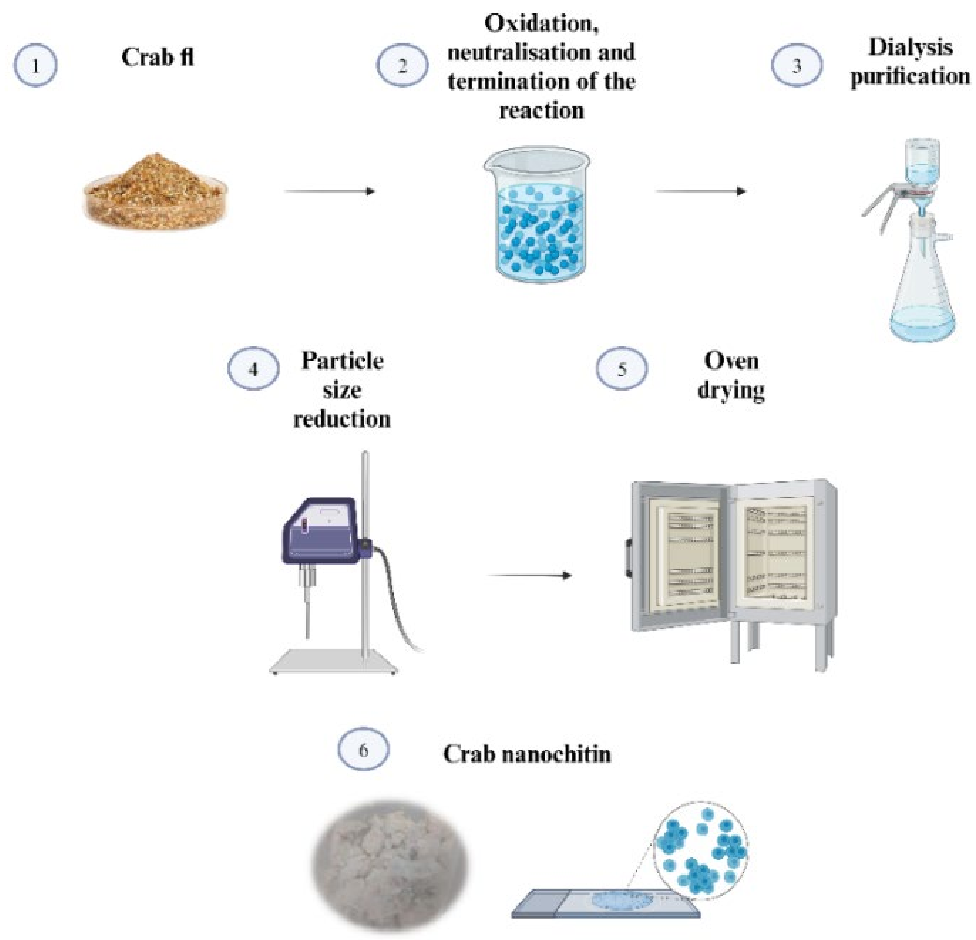


FIGURE 2
Scheme for obtaining nanochitin

MATERIALS AND APPLIED METHODOLOGY

This study presents a comparative technical analysis of the physicochemical and mechanical properties of mortars enhanced with nanochitin derived from crab shell waste. To ensure durability under diverse environmental conditions, Type N and Type HS cements were selected as the binding materials. The primary objective of this research is to evaluate the impact of nanochitin on improving mortar performance and to explore its feasibility as a sustainable construction material.

The nanochitin utilized in this study was synthesized through a controlled extraction and processing methodology specifically designed for crab shell waste. The research framework includes an evaluation of the production process complexity, cost-efficiency, and the quality of the final product. Furthermore, the study investigates the influence of nanochitin on cement hydration kinetics and its role in reducing mortar porosity.

To determine the effectiveness of nanochitin in enhancing mortar properties, samples incorporating this nanomaterial underwent a series of standardized assessments, including compressive strength testing, permeability evaluations, and microstructural characterization using advanced techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM). These analyses provide critical insights into the cohesion and long-term durability of nanochitin-modified mortars.

This research aims to establish the viability of integrating nanochitin from crab shell waste into the construction sector, aligning with sustainable waste management strategies while improving the structural performance of cement-based materials. Through a systematic comparative analysis, the study seeks to identify the optimal nanochitin-cement formulation that maximizes both mechanical efficiency and environmental sustainability in construction applications.

Materials

The characterization of mortar specimens was performed through a series of controlled laboratory tests, strictly following the national standards established by INEN. To develop the final mortar formulations, each material underwent an initial characterization phase involving iterative trial mixtures. This approach ensured the optimization of material properties before determining the most suitable formulations. The mixing process and dosage parameters were meticulously recorded to guarantee consistency and repeatability in subsequent evaluations.

The experimental program included key performance assessments such as flowability analysis, compressive strength testing, and permeability evaluation. The data obtained from these tests were systematically analyzed and interpreted to assess the effects of nanochitin incorporation on mortar properties. The fundamental materials used in this study consisted of fine aggregate (sand), cement, and nanochitin derived from crab shell waste, as specified in Table 1. These components played a critical role in the comprehensive evaluation of mortar behavior, contributing to the understanding of its structural and durability characteristics.

TABLE 1

Components

Materials	Specification
Fine Aggregate	Quarry of Copeto - Toachi River
Cement Maestro	Type N Masonry Cement - Holcim
Cement Campeón	Portland Pozzolanic Type HS - UNACEM
Watter	Metropolitan District of Quito Water Network (EPMAPS)
Crab Nanochitin	Materia synthesized in the laboratory

Fine aggregate

The Toachi River quarry, situated in the province of Santo Domingo de los Tsáchilas, is managed by Copeto Cía. Ltda., a company specializing in the extraction of construction aggregates (COPETO, 2024). This company supplies washed sand, block sand, and natural sand, among other materials, all of which comply with the highest national quality standards (INEN, MOP) and international standards (ASTM) (Défaz Paredes and Simbaña Cóndor, 2013).

Table 2 presents the characteristics of the fine aggregate, with a fineness modulus value of 2.44. Additionally, Figure 3 illustrates the particle size distribution curve, which complies with the limits established in NTE INEN 2536 (INEN, 2010a) for use in masonry mortar.

TABLE 2

Properties of Fine Aggregate

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

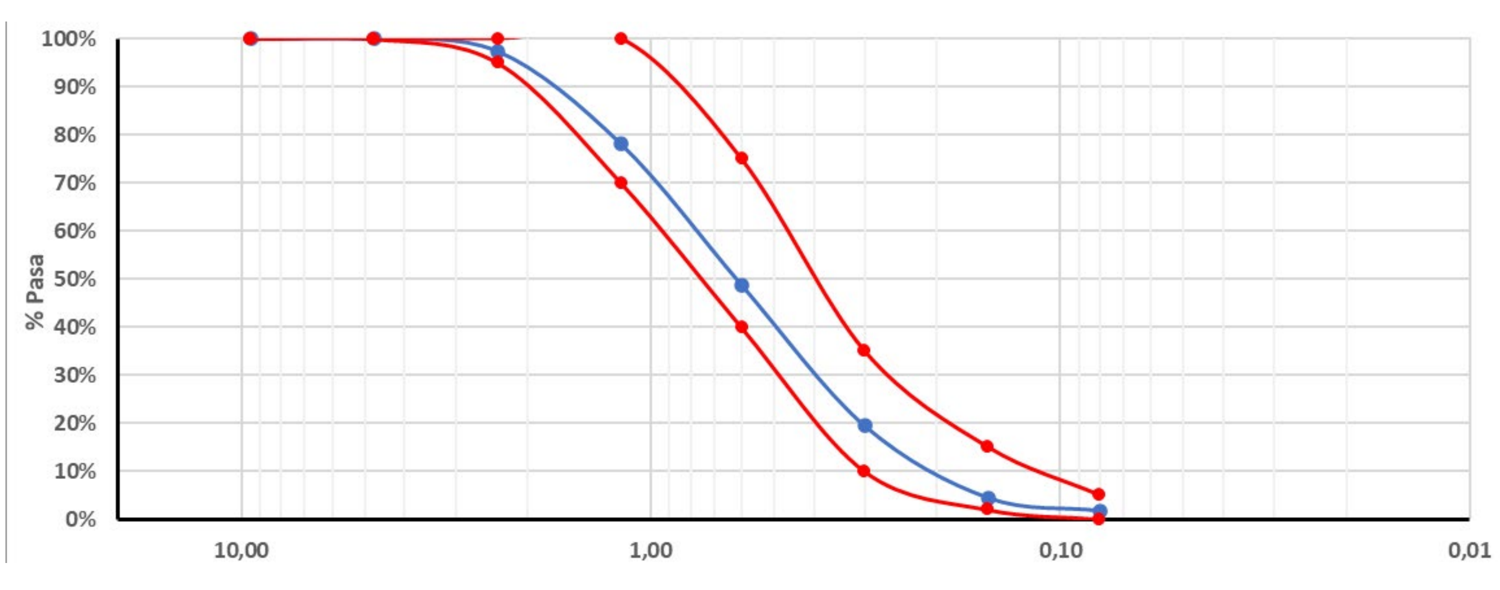


FIGURE 3

Granulometric Distribution of the Aggregate Material

Pozzolanic cement

Portland pozzolanic cement is a blend of Portland cement and pozzolanic materials, which are reactive compounds that may originate from volcanic sources or other artificial materials (Pozzolanic Cement - Construmatica, no date). This type of cement offers enhanced resistance to chemical agents, a lower heat of hydration, and improved water resistance compared to conventional Portland cement. In this study, Type N and Type HS Portland cement were utilized, both of which comply with the requirements established in NTE 2380 (INEN, 2011).

Type N Cement – Holcim "Maestro" cement

Holcim "Maestro" Type N cement is a modern, high-quality material designed for contemporary masonry applications. Its specialized formulation enhances material handling by up to 15% compared to other cements, significantly minimizing waste and rebound during application. Furthermore, due to its high waterproofing capacity, ranging from 65% to 90%, it is particularly suitable for use in structures exposed to continuous moisture (Holcim, 2022).

UNACEM Type HS Cement – UNACEM "Campeón" cement

"Campeón" HS cement is a hydraulic cement with high sulfate resistance, certified under the NTE INEN 2380 standard to guarantee quality and reliability in construction. Its fine particle size and controlled composition enable the production of durable concrete over time, particularly in aggressive environments with elevated sulfate concentrations in soils and water. This cement is especially suitable for mass concrete applications, soil stabilization, dam construction, and mortars that offer ease of placement and high-quality finishes (Cemento Selvalegre, 2025).

Water

The quantity and quality of water in the mortar mix are of utmost importance for the development of mechanical properties and the durability of the mortar. Pure, impurity-free water allows for proper cement hydration, which is crucial for achieving the desired mechanical properties. Poor-quality water can lead to catastrophic effects on these properties and negatively influence the workability of the mix. Therefore, it is essential to comply with the provisions of the NTE INEN 2617:2012 standard (INEN, 2012), for construction water to ensure the durability and integrity of the structure, thereby supporting safe and sustainable construction practices.

Ultrasound

Ultrasound has been identified as an effective technique for dispersing nanoparticles in cementitious matrices. It has been shown that ultrasound improves the distribution of nanoparticles in mortar, resulting in a denser and stronger microstructure. For example, research Hielscher Ultrasonics, 2024), indicates that the use of ultrasound can increase the size of C-S-H (calcium silicate hydrate) phases in the cement paste, contributing to better strength and reduced porosity (Marcondes *et al.*, 2015).

Nanoparticles obtained from crab

The synthesis of nanoparticles was carried out in the laboratories of the Central University of Ecuador, where 170 grams of pure crab powder were obtained from exoskeletons. Through subsequent chemical processes, 117 grams of crab-derived nanochitin were extracted.

Composition of Crab nanoparticles by laboratory tests

To verify the elemental composition and structural characteristics of the nanoparticles extracted from crab shell waste, a series of laboratory analyses were conducted to ensure the accuracy and reliability of the research findings. The characterization process employed advanced analytical techniques, including Energy Dispersive Spectroscopy (EDS), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), and X-ray Diffraction (XRD).

These methodologies are essential for assessing the physicochemical properties, morphology, and crystallographic structure of the synthesized nanoparticles. The obtained data provide critical insights into their composition, stability, and potential applicability in construction materials and other industrial sectors.

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

Background

The use of nanochitin in the construction industry is also associated with improved mortar workability, allowing for more homogeneous mixtures with greater water retention. This factor is particularly crucial in arid climates, where rapid moisture loss can negatively impact the strength and durability of the material. Furthermore, electron microscopy studies have revealed that nanochitin contributes to a more compact cement microstructure, effectively reducing capillary absorption and water permeability (Aher *et al.*, 2023; Haider *et al.*, 2022a).

• Crab nanochitin

Energy Dispersive Spectroscopy (EDS) is a technique used for the elemental and chemical characterization of a sample under analysis. It is especially valuable in the study of nanomaterials, as it enables the identification and quantification of the elements present.
As shown in Figure 4, the analysis revealed that oxygen is the predominant element, evidenced by an energy peak in the 0.5 keV region corresponding to the K series. Additionally, less pronounced energy peaks were observed from the K series of carbon, magnesium, phosphorus, and calcium, between 0 and 4 keV. The quantitative composition expressed as weight percentage (wt.%) reveals that oxygen constitutes 69.17%, carbon 48.42%, magnesium 1.15%, phosphorus 0.73%, and calcium 21.62%. The relative atomic fraction percentage is 48.23% for oxygen, 44.97% for carbon, 0.53% for magnesium, 0.26% for phosphorus, and 6.02% for calcium, suggesting a fairly uniform distribution of elements in the sample.

The results show that the sample primarily contains oxygen and carbon, which together constitute approximately 98% of the total weight, suggesting that it could be an organic composite material, possibly related to crab chitin. The substantial presence of oxygen and carbon suggests the possible existence of oxygenated organic compounds. Conversely, calcium, magnesium, and phosphorus appear in considerably lower concentrations, indicating a minimal contribution to the overall material composition. The data exhibit high accuracy, with relatively low error margins, ensuring the reliability of the obtained results.

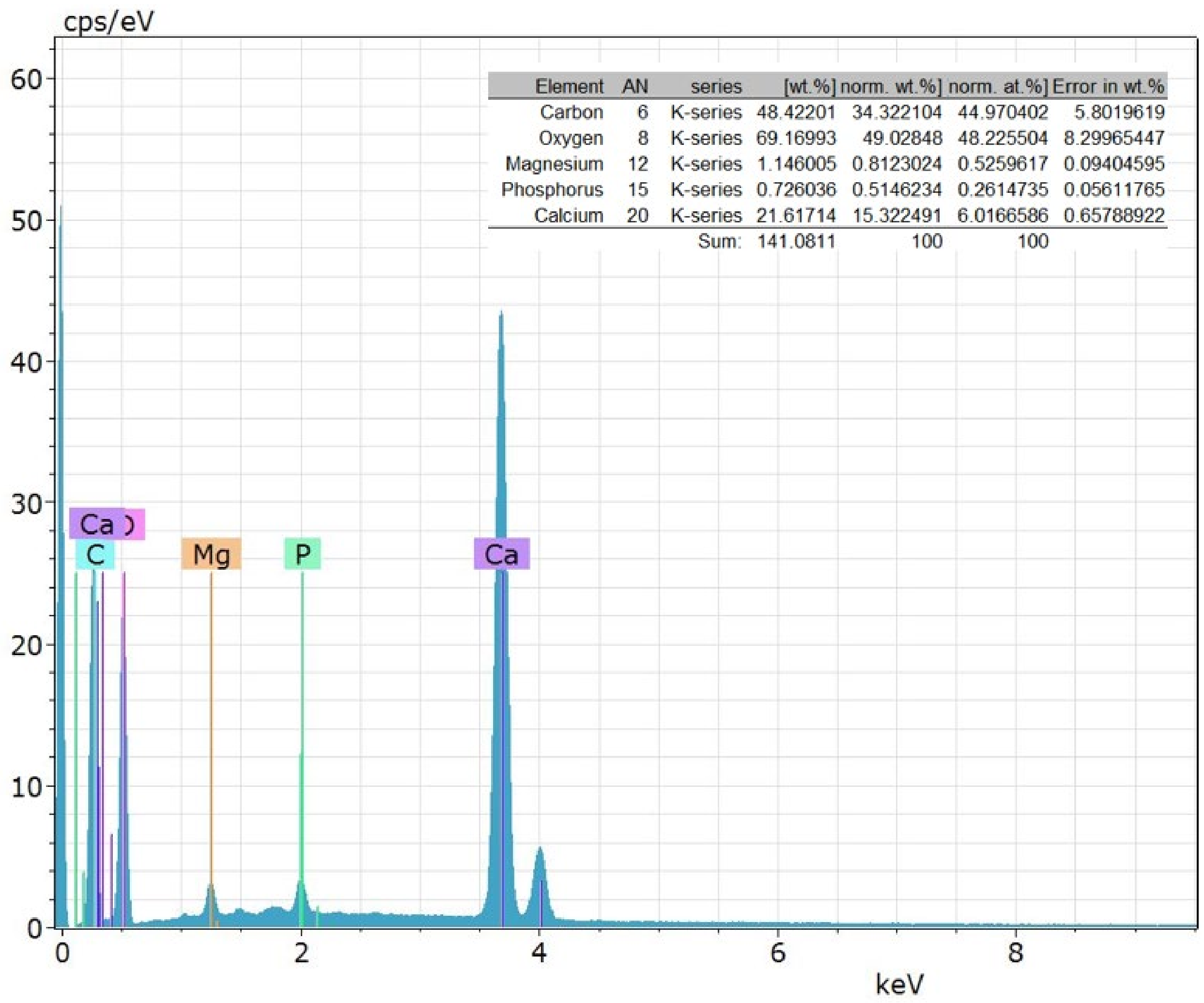


FIGURE 4
Composition of crab nanochitin

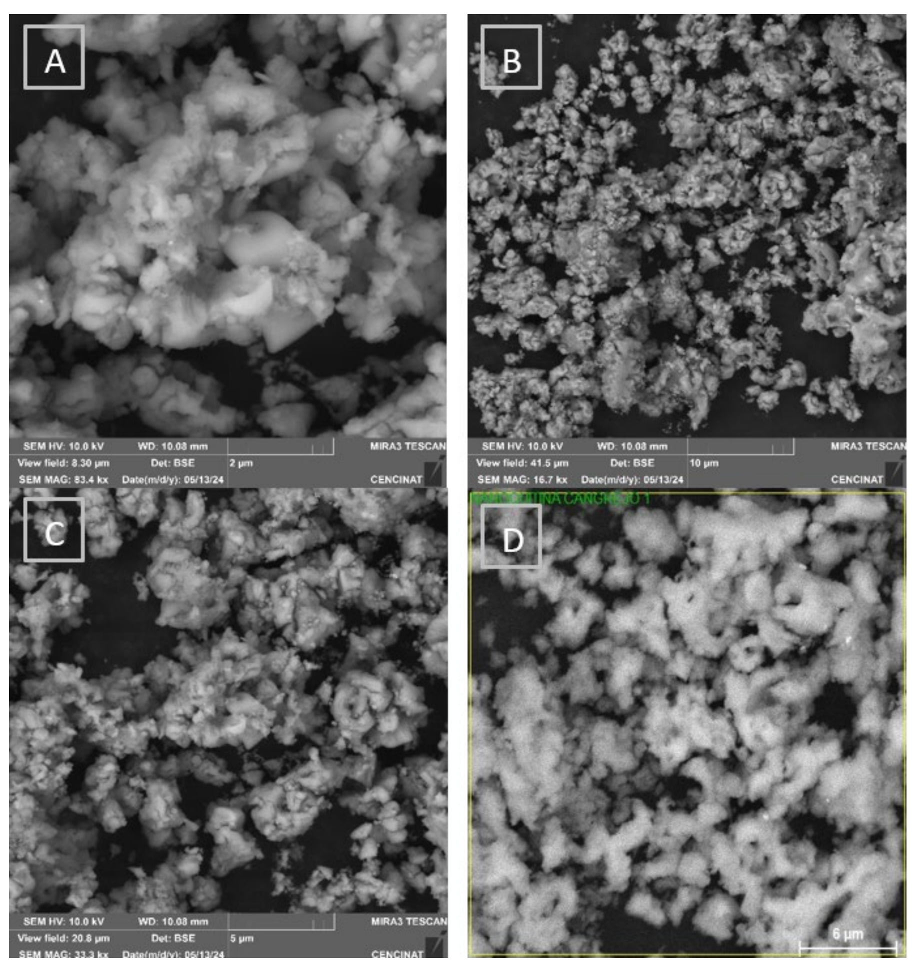


FIGURE 5
Morphology and topography of crab nanochitin

In Figure 5, the SEM images (a)-(b)-(c)-(d) provide detailed information on the morphology, showing irregular shapes and textured surfaces, as well as the non-uniform distribution of particles, with areas of higher concentration and others of broader dispersion within the sample. The granular structure and tendency to agglomerate may have significant implications for the physical and chemical properties of the material, such as reactivity and stability. The high resolution of image A reveals fine details, while the images with lower magnification (b, c, and d) offer a broader view of particle distribution and agglomeration.

Transmission Electron Microscopy (TEM)

• Crab Nanochitin

The images captured using transmission electron microscopy (TEM) in Figure 6 (a)-(b)-(c)-(d) confirm the successful synthesis of materials with typical nanometric characteristics, including dispersed particles and fibrous networks.

In Figure 6A, a large polygonal particle surrounded by numerous smaller particles and a fibrous network is observed, indicating the coexistence of large and small structures within a fibrous matrix. Figure 6B shows a uniform dispersion of spherical particles over a fibrous network, suggesting good distribution and possible interaction between the particles and the matrix. Figure 6C reveals a structure similar to that in Figure 6A, with a large polygonal particle and a network of smaller particles, indicating consistency in the material synthesis. Finally, Figure 6D presents a dense fibrous network with dispersed spherical particles, suggesting a well-distributed composite structure.

The areas of varying contrast within these networks and particles are consistent with the variations in topology at the nanoscale, common in the production of advanced materials by methods such as chemical or mechanical synthesis. These properties are crucial for applications that demand high conductivity and extensive surface area, including flexible electronic devices and supercapacitors.

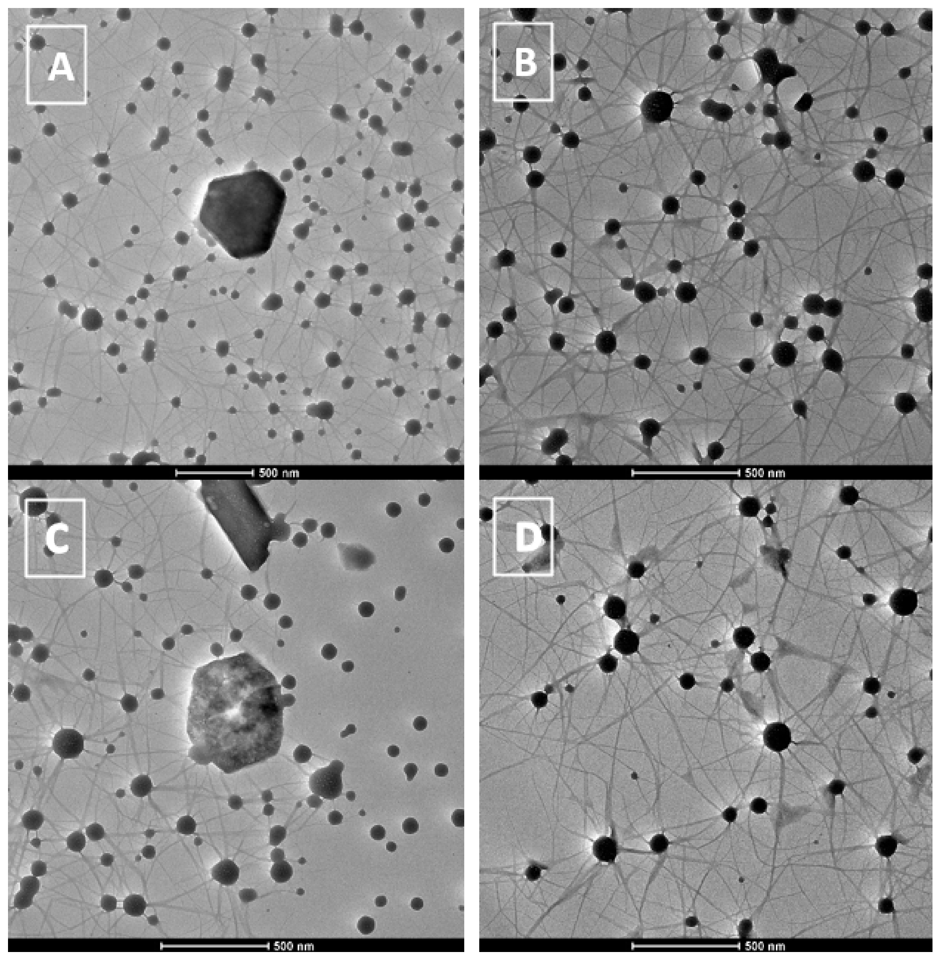


FIGURE 6

Nanometric composition of chitin

X-ray Diffraction (XRD)

• Crab nanochitin

In Figure 7, the X-ray Diffraction (XRD) analysis shows a pattern characteristic of chitin obtained from crab exoskeletons. The test was conducted on a diffractometer using a scanning range from 0 to 90° on the 2θ scale, at a speed of 0.02 degrees per second. Notable peaks can be observed near angles 2θ = 20°, 30°, 40°, 50°, 60°, and 70°. The sharp and highly intense nature of these peaks indicates a high degree of crystallinity and the presence of multiple crystalline phases, consistent with the material's complex structure.

The preferential orientation in the crystalline planes contributes to the peak intensity and the organized structure of the material. These results demonstrate that the crystalline quality is essential for applications that rely on the specific physical and chemical properties of chitin and the mineral compounds from crab exoskeletons.

The XRD pattern of "CRAB NANOCHITIN" reveals the presence of multiple crystalline phases, with a primary peak observed near 40° in 2θ, indicating a high concentration of a specific component, likely a calcium-based compound. The presence of additional minor peaks suggests that the sample comprises multiple crystalline phases, a characteristic commonly found in complex biological materials such as crab exoskeletons. These findings align with the expected crystalline structure of composite materials containing typical minerals from crab exoskeletons.

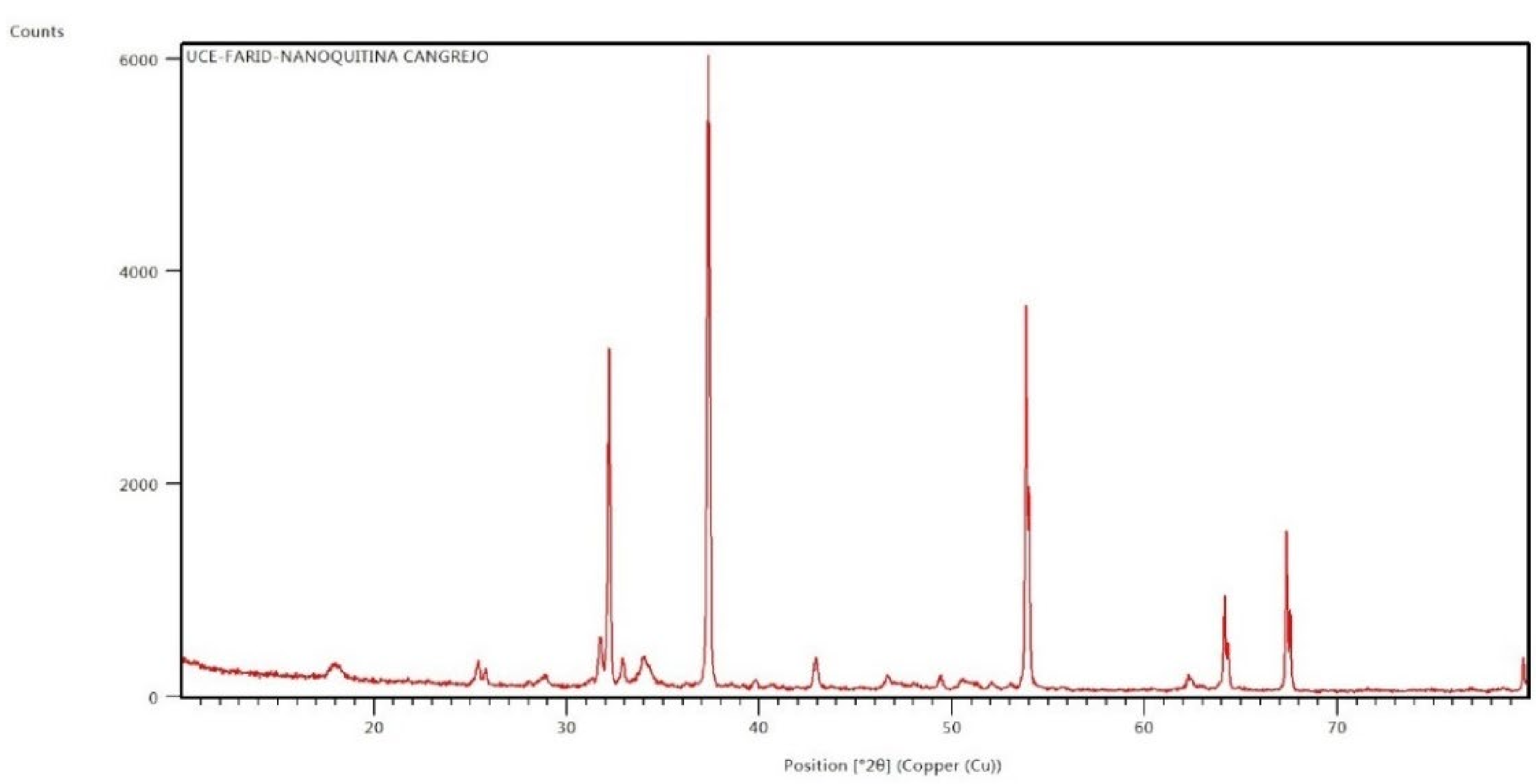


FIGURE 7

Structural composition of crab nanochitin

METHODOLOGY

The experimental design methodology outlined in the INEN standards focuses on ensuring the accuracy and reproducibility of mortar and cement tests through standardized procedures. Specifically, NTE INEN 2518 (INEN, 2010b), emphasizes the practices for proper dosing and mixing of materials. The appropriate dosage is confirmed using containers of known volume, adjusting the sand quantities as needed to maintain the desired consistency of the mortar. To achieve a homogeneous mixture, it is recommended to initially mix the majority of the water with a portion of the sand and cementitious material rapidly and uniformly, followed by the gradual incorporation of the remaining components. Furthermore, it is specified that the mixing process should continue for 3 to 5 minutes after the final addition of water. The methodology also accounts for retempering the mortar to compensate for water loss due to evaporation, ensuring the material maintains the proper consistency before its application in construction.

The procedure requires the precise determination of the proportions of the various materials used, as presented in Table 3. These proportions are crucial for the correct preparation of the specimens, which play a key role in the design and evaluation phases of the mortar, thereby ensuring the accuracy and reliability of the results obtained in the tests.

The mortar mixes were designed by maintaining a constant water/cement ratio; however, the number of nanoparticles was adjusted according to the percentages relative to the weight of the cement, as indicated in Table 3.

TABLE 3

Quantity of material for specimens

Name	W/C Ratio	Cement	Fine Aggregate	Water	Nanoparticles
		g	g	g	g
M	0.625	500.0	1596.2	312.5	-
M+$Q_{0.75\%}$	0.625	496.2	1596.2	312.5	3.8
C	0.584	500.0	1596.2	292.0	-
C+$Q_{0.75\%}$	0.584	496.2	1596.2	292.0	3.8

M: Control mortar mix with "Maestro cement" containing 0% nanoparticles.
M+Q0.75%: Control mortar mix with 0.75% chitin nanoparticles.
C: Control mortar mix with "Campeón" cement containing 0% nanoparticles.
C+Q0.75%: Control mortar mix with 0.75% chitin nanoparticles.

The dosing and design of the mortar are fundamental processes that require careful selection of materials, proper mixing, and thorough testing. The quality of mortar plays a crucial role in determining the durability and performance of masonry structures. The proposed methodology ensures that the mortar formulations comply with the required standards for specific applications, achieving an optimal balance between workability and mechanical properties.

Mortar mix tests

The prepared specimens were subjected to a simple compression test. For this test, the volume of material required for 50 mm edge cubes must be calculated. Table 4 and Table 5 specify the number of specimens prepared for the compression test.

TABLE 4

Quantify of specimens for the compression strength test

N°	Name	Description	Test	Number of Specimens
1	M	Standard Mix	Simple Compressive Strength	30
2	M+$Q_{0.75\%}$	Standard Mix + 0.75% Nanochitin		30
3	C	Standard Mix		30
4	C+$Q_{0.75\%}$	Standard Mix + 0.75% Nanochitin		30
		Total		120

TABLE 5

Quantify of specimens for the permeability test

Nº	Name	Description	Test	Number of Specimens
1	M	Standard Mix	Permeability	1
2	M+$Q_{0.75\%}$	Standard Mix + 0.75% Nanochitin		1
3	C	Standard Mix		1
4	C+$Q_{0.75\%}$	Standard Mix + 0.75% Nanochitin		1
		Total		**4**

In the Figure 8 presents the experimental processes and results related to the enhancement of mortar properties using nanoparticles synthesized from crab exoskeletons. The figure highlights the application of nanochitin in various mortar mixes based on "Maestro" cement.

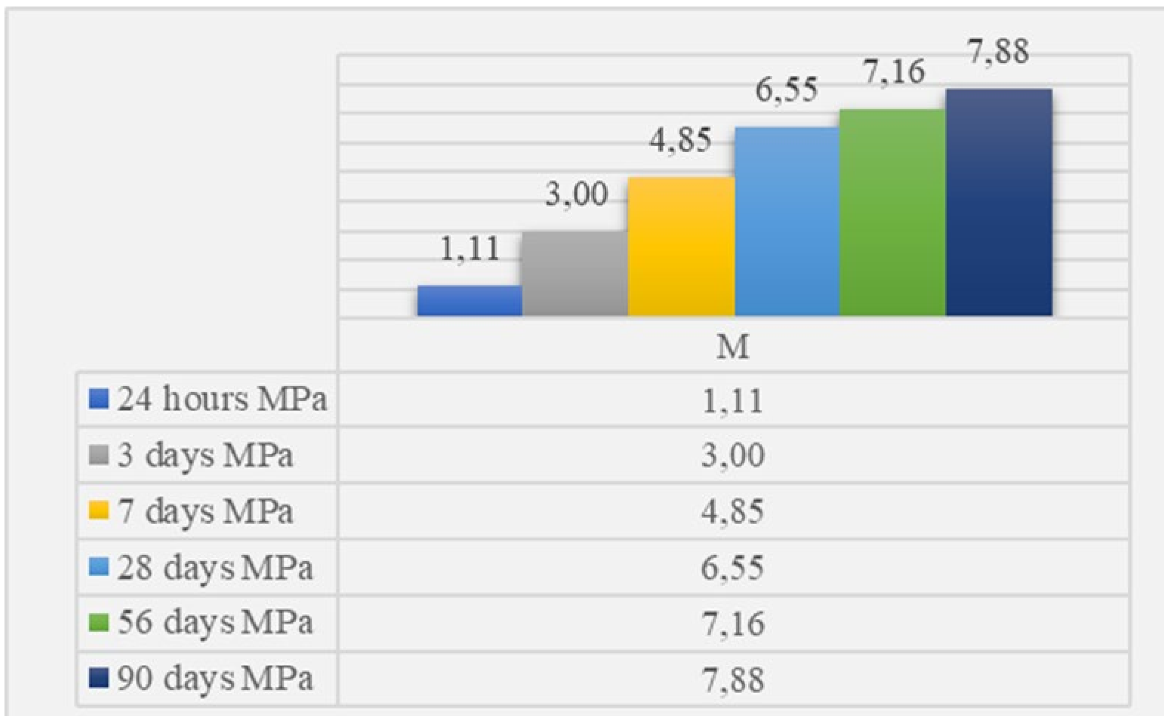


FIGURE 8

Standar Mortar (M)

The use of nanochitin derived from crab waste in mortars significantly enhances the mechanical properties of the material. Nanochitin proves to be effective in increasing compressive strength, demonstrating the best performance among the evaluated nanomaterials. Additionally, the ultrasonic dispersion process and flow tests confirm the viability of these mixes for construction applications, ensuring uniform distribution and enhanced mechanical properties. This approach not only optimizes mortar performance but also promotes sustainability through the reuse of biological waste.

In Figure 9 presents the experimental processes and results related to the enhancement of mortar properties using nanochitin synthesized from crab exoskeletons. The figure highlights the application of nanochitin in various mortar mixes based on "Campeón" cement. Figure 9 shows that the incorporation of nanochitin derived from crab exoskeletons significantly improves the properties of mortar, particularly in terms of compressive strength. XRD characterization, along with flow and ultrasonic tests, ensures that nanochitin is well-distributed and active within the mortar matrix. Among the evaluated nanomaterials, nanochitin demonstrates the greatest increase in compressive strength, reaching values close to 33 MPa. These improvements suggest that nanochitin synthesized from biological waste is an effective and sustainable solution for optimizing the performance of construction materials, aligning with the principles of nanotechnology and the circular economy (Figure 10 and Figure 11).

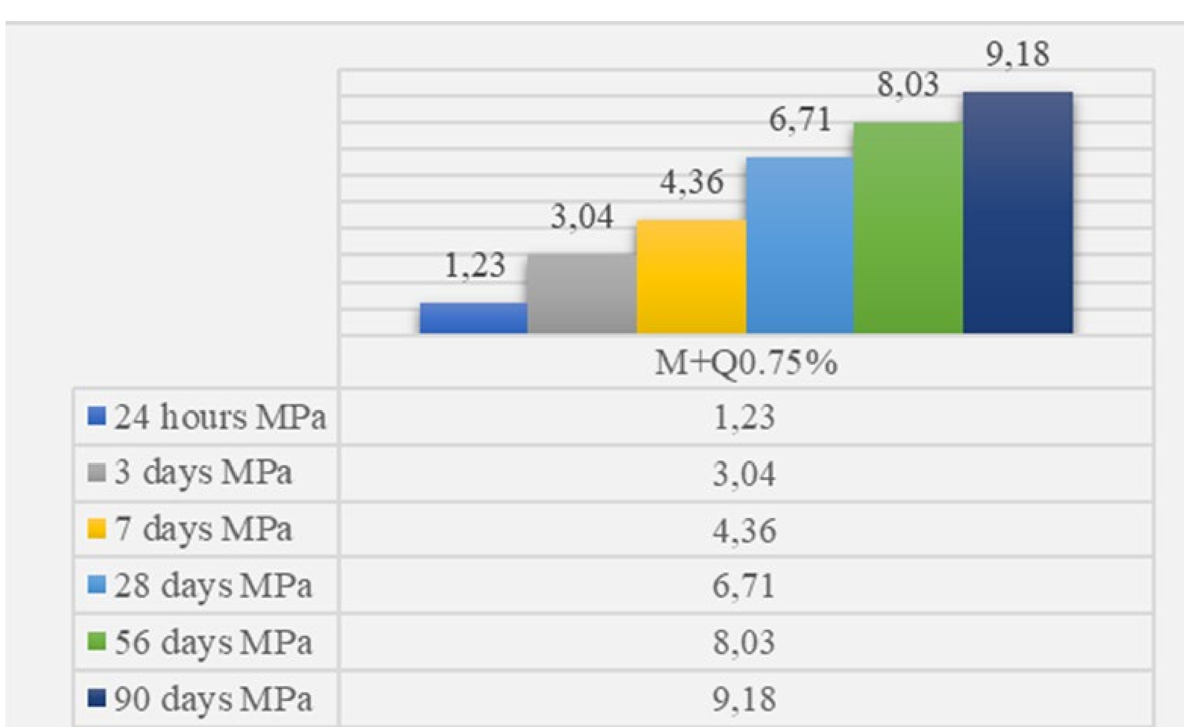


FIGURE 9

Mortar – Nanochitin 0.75%

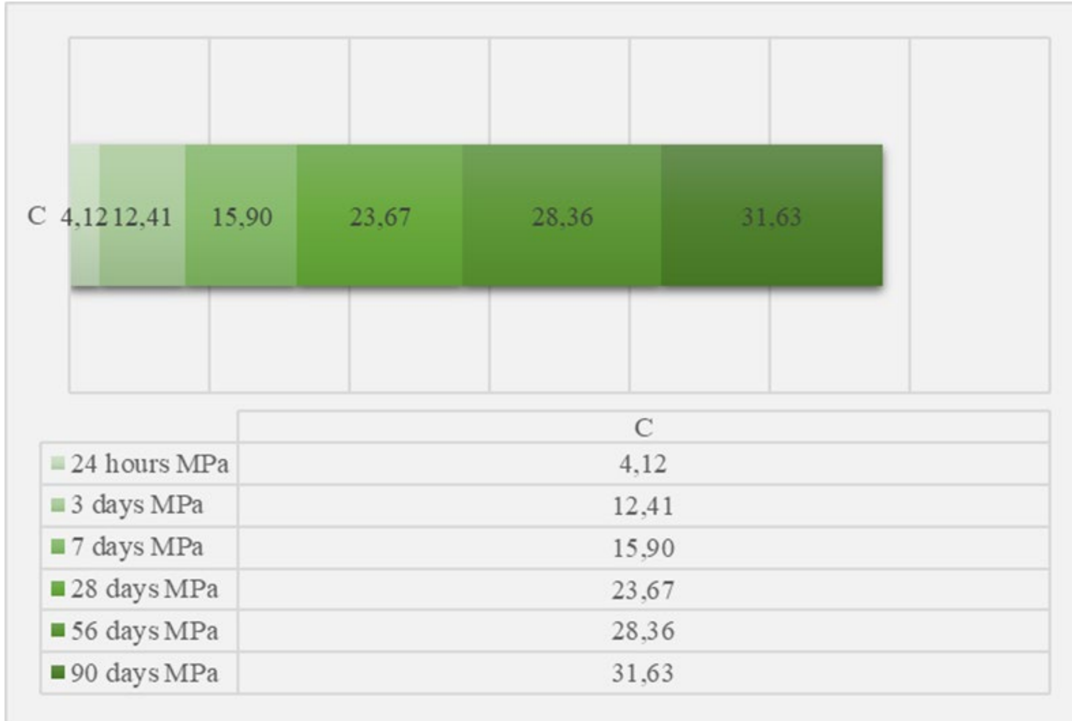


FIGURE 10

Standar Mortar (C)

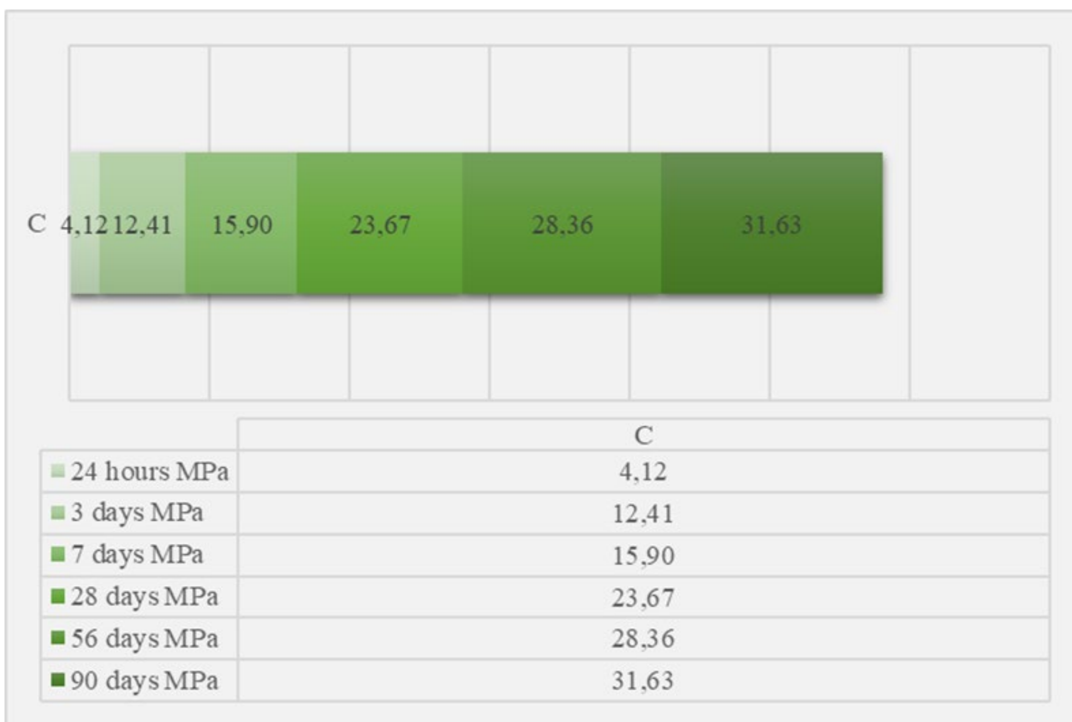


FIGURE 11

Mortar – Nanochitin 0.75%

Permeability test

Drop Method

Several studies have employed the drop test to assess the permeability of mortars and concretes. For instance, Klein *et al.* (2012) utilized contact angle measurements to analyze the wettability of granular materials that constitute mortar, including cement and aggregates. Their findings indicated that the contact angle is directly related to the absorption capacity of these materials, influencing key properties such as workability and the mechanical strength of hardened mortar. Furthermore, the drop test serves as a valuable tool for predicting the behavior of cementitious materials when exposed to liquid penetration, including water or aggressive agents, making it essential for evaluating the durability of mortar and concrete structures (Marketing, 2024).

The contact angle quantifies the interaction between a mortar's solid surface and the liquid it encounters, where a smaller contact angle signifies improved wettability, which correlates with increased permeability of the material. This is because a low contact angle signifies that the liquid, in this case water, will wet the surface of the mortar well, allowing good penetration into the material (Klein *et al.*, 2012). Therefore, this contact angle is crucial for determining whether the surface is hydrophobic or hydrophilic, which is fundamental for evaluating the effectiveness of the modifications made to the nanosilica particles (Alvansazyazdi *et al.*, 2023).

RESULTS AND DISCUSSION

Uniaxial compressive strength

This is one of the most common tests, typically performed using mortar cubes (usually 50 mm) to determine the mortar's ability to withstand compressive loads. The INEN 488 standard is commonly employed for this test (INEN, 2009).

The results presented in Table 6 summarize the compressive strengths obtained at different ages for the various types of mixes prepared.

TABLE 6

Summary of the compressive strength of mortars

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+$Q_{0.75\%}$	1.23	3.04	4.36	6.71	8.03	9.18
C	4.12	12.41	15.90	23.67	28.36	31.63
C+$Q_{0.75\%}$	4.62	12.78	15.27	25.05	30.75	33.56

The table 6 presents the compressive strength evolution of mortar mixes incorporating nanochitin ($Q_{0.75}$%), compared to standard mixes (M and C) over different curing ages (24 hours to 90 days).

The C+$Q_{0.75}$% mix exhibited the highest compressive strength across all ages, reaching 33.56 MPa at 90 days, a 6% increase compared to the unmodified C mix (31.63 MPa). In Type N cement, the mix with nanochitin (M+$Q_{0.75}$%) demonstrated improved long-term performance, achieving 9.18 MPa at 90 days, an increase of 16.5% compared to the reference mix (M: 7.88 MPa). The early-age strength gain (24 hours to 7 days) in the C+$Q_{0.75}$% mix indicates a positive interaction between nanochitin and hydration products, enhancing the densification and cohesion of the cementitious matrix.

These results confirm that nanochitin improves compressive strength and long-term durability, making it an effective nanomaterial for enhancing mechanical performance in cement-based mortars.

Uniaxial compressive strength vs. Time

Figure 12 illustrates the compressive strength evolution of mortars prepared with Maestro Cement, comparing the reference mix (M) and the mix modified with 0.75% nanochitin (M+$Q_{0.75}$%) over different curing ages (24 hours to 90 days).

At early ages (24 hours to 7 days), both mixes exhibit similar performance, indicating that nanochitin does not significantly affect initial hydration.

From 28 days onwards, the M+$Q_{0.75}$% mix surpasses the reference mix in compressive strength, reaching 9.18 MPa at 90 days, an increase of 16.5% compared to the M mix (7.88 MPa). The enhanced performance at later ages suggests that nanochitin improves the cementitious matrix's cohesion and densification, leading to better long-term durability.

These findings confirm that nanochitin enhances compressive strength development over time, making it an effective nanomaterial for improving mechanical properties in cement-based mortars.

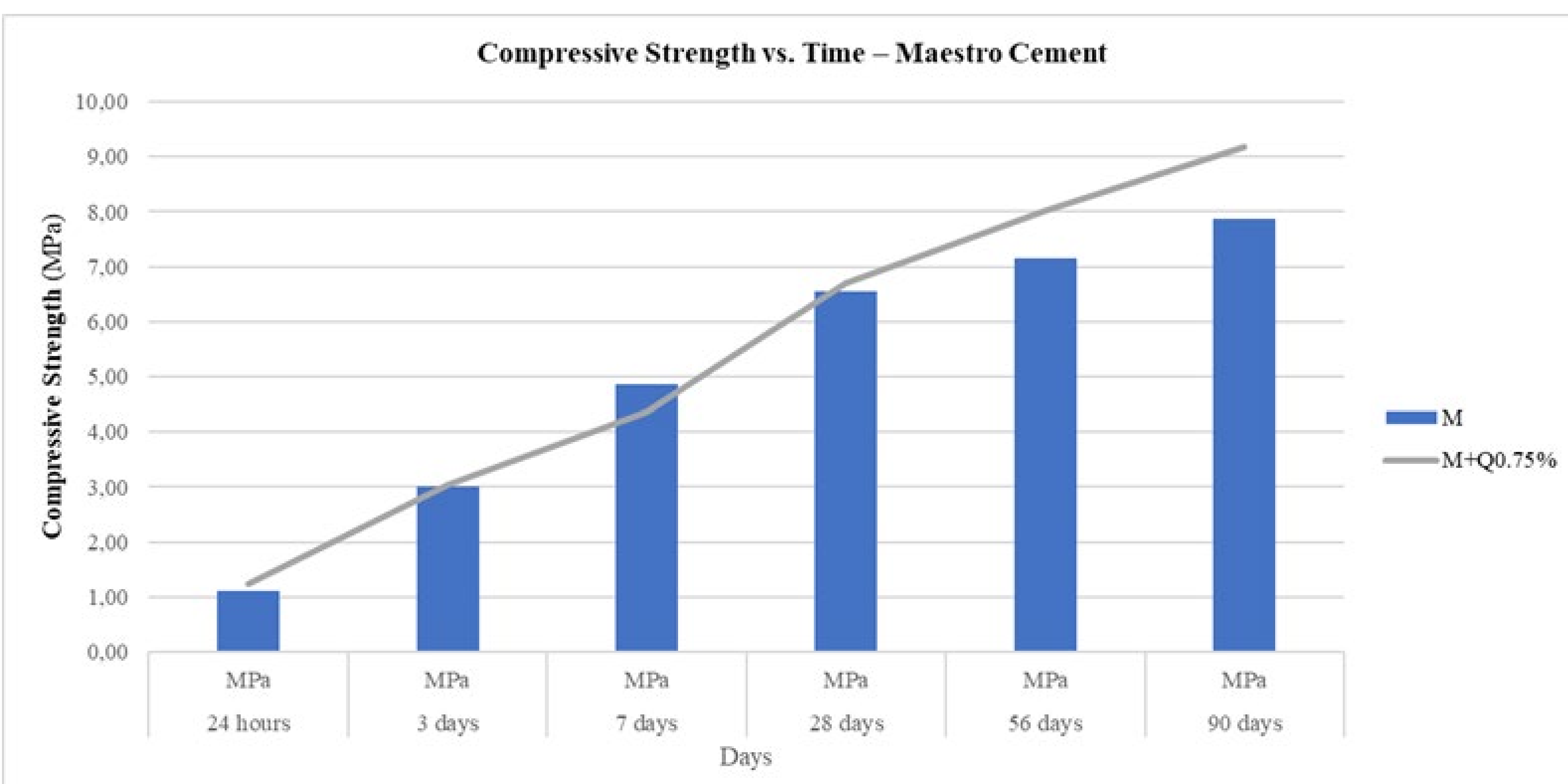


FIGURE 12

Simple Compressive Strength vs Time – Maestro Cement

Figure 13 presents the compressive strength evolution of mortars prepared with Campeón Cement, comparing the reference mix (C) with the mix modified with 0.75% nanochitin (C+$Q_{0.75}$%) over different curing ages (24 hours to 90 days).

At early ages (24 hours to 7 days), both mixes exhibit similar strength development, indicating that nanochitin does not significantly alter the early hydration process. From 28 days onwards, the C+$Q_{0.75}$% mix shows a progressive increase in compressive strength, outperforming the reference mix, reaching 33.56 MPa at 90 days, a 6% improvement compared to the unmodified mix (31.63 MPa). This behavior suggests that nanochitin enhances the microstructural integrity of the cementitious matrix, contributing to greater durability and long-term mechanical performance.

These results confirm that nanochitin effectively improves compressive strength and densification, making it a viable additive for high-performance cementitious materials.

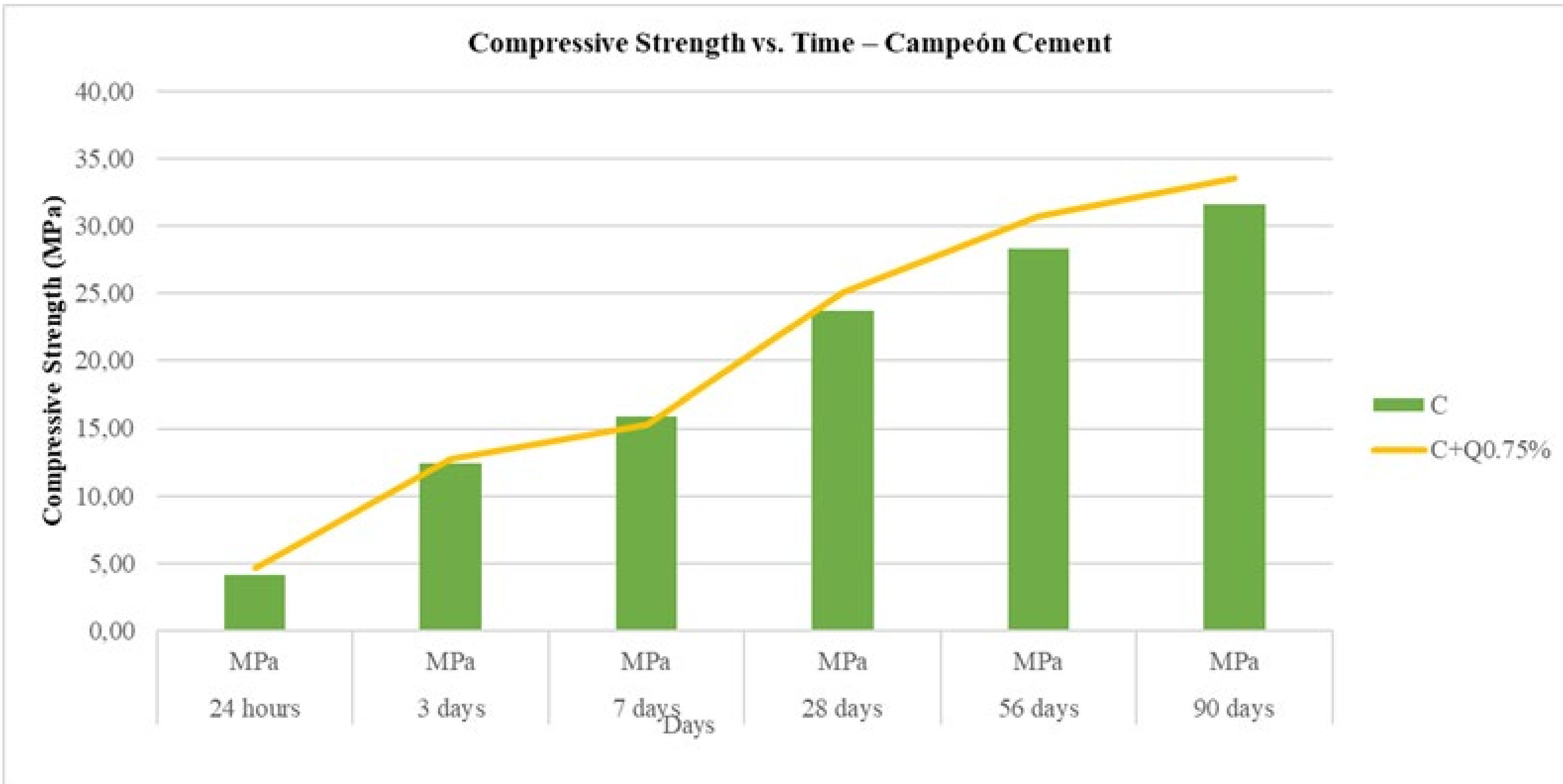


FIGURE 13

Simple Compressive Strength vs Time – Campeón Cement

Figure 14 illustrates the evolution of compressive strength in mortars prepared with Maestro Cement, comparing the reference mix (M) and the mix modified with 0.75% nanochitin (M+$Q_{0.75}$%) over different curing periods (24 hours to 90 days).

At early ages (24 hours to 7 days), both mixes exhibit similar trends, with minor variations, indicating that nanochitin does not significantly affect early strength development. From 28 days onwards, the M+$Q_{0.75}$% mix shows a progressive increase in compressive strength, reaching 9.18 MPa at 90 days, a 16.5% improvement compared to the reference mix (7.88 MPa).

The enhanced strength at later curing ages suggests that nanochitin contributes to improved cement hydration and matrix densification, resulting in higher durability and long-term mechanical stability.

These findings confirm that nanochitin enhances the long-term compressive strength of mortar, making it a promising additive for optimizing cementitious materials in sustainable construction applications.

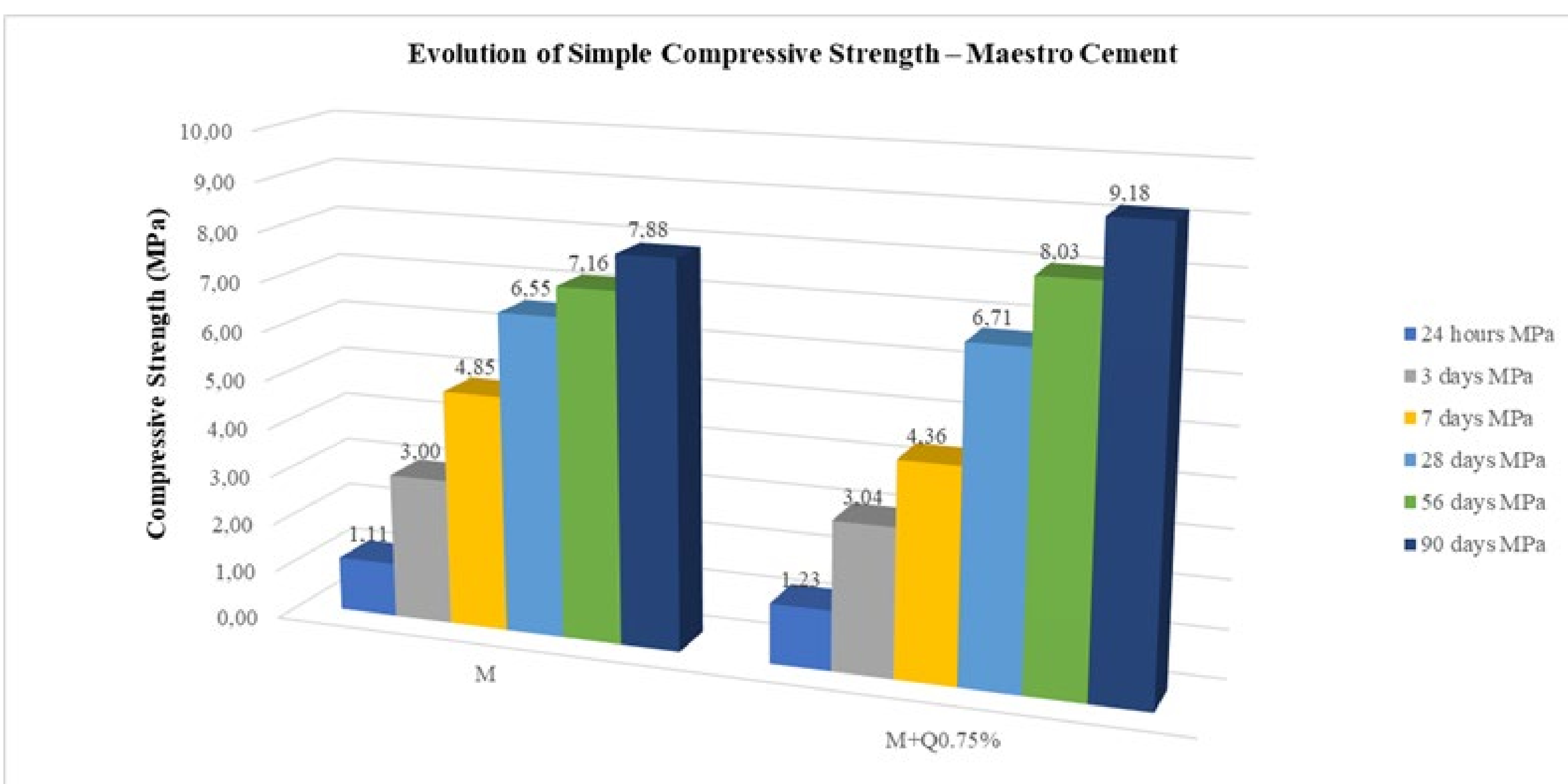


FIGURE 14

Evolution of simple compressive strength – Maestro Cement

Figure 15 illustrates the evolution of compressive strength in mortars prepared with Campeón Cement, comparing the reference mix (C) and the mix modified with 0.75% nanochitin (C+$Q_{0.75}$%) over different curing periods (24 hours to 90 days).

At early ages (24 hours to 7 days), both mixes exhibit similar trends, with minor variations, indicating that nanochitin does not significantly alter the early hydration process. From 28 days onwards, the C+$Q_{0.75}$% mix shows a progressive increase in compressive strength, reaching 33.56 MPa at 90 days, a 6% improvement compared to the unmodified mix (31.63 MPa).

The enhanced mechanical performance at later curing ages suggests that nanochitin enhances hydration reactions, matrix densification, and particle cohesion, resulting in greater durability and long-term strength.

These findings confirm that nanochitin improves the long-term compressive strength and durability of cementitious materials, making it a viable additive for high-performance construction applications.

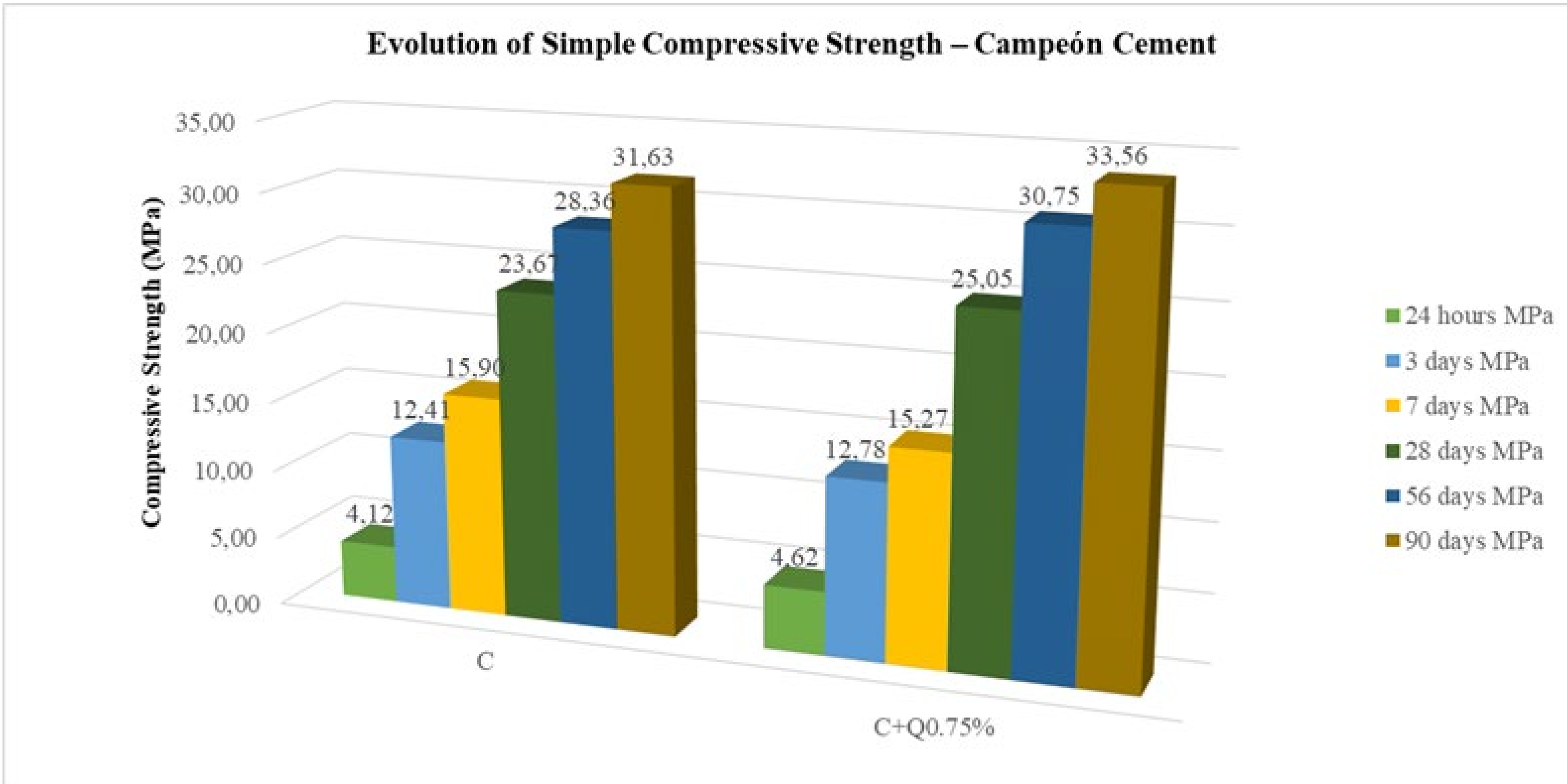


FIGURE 15

Evolution of simple compressive strength – Campeón Cement

Scanning Electron Microscopy (SEM)

SEM images reveal a granular microstructure with particles of various sizes homogeneously distributed, suggesting good compaction and low porosity—critical factors for enhancing the material's strength and durability. In particular, the M+Q0.75% (Figure 16) sample shows well-distributed nanochitin, which contributes to reduced porosity and structural reinforcement. A similar analysis was performed on mixtures incorporating Campeón cement (Figure 17), where the addition of nanomaterials was found to improve densification and decrease porosity. The control sample (C) displays a heterogeneous structure with unevenly sized particles, typical of cementitious mixtures, whereas the C+Q0.75% sample exhibits a more uniform distribution of nanochitin, resulting in lower porosity and improved mechanical performance and durability.

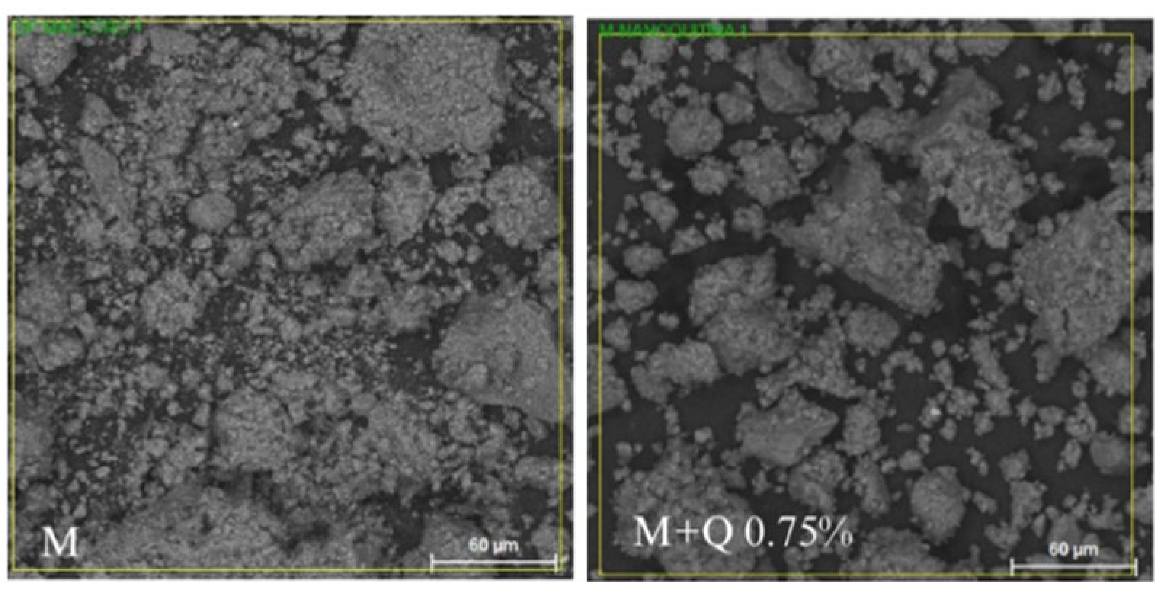


FIGURE 16

M+Q0.75%

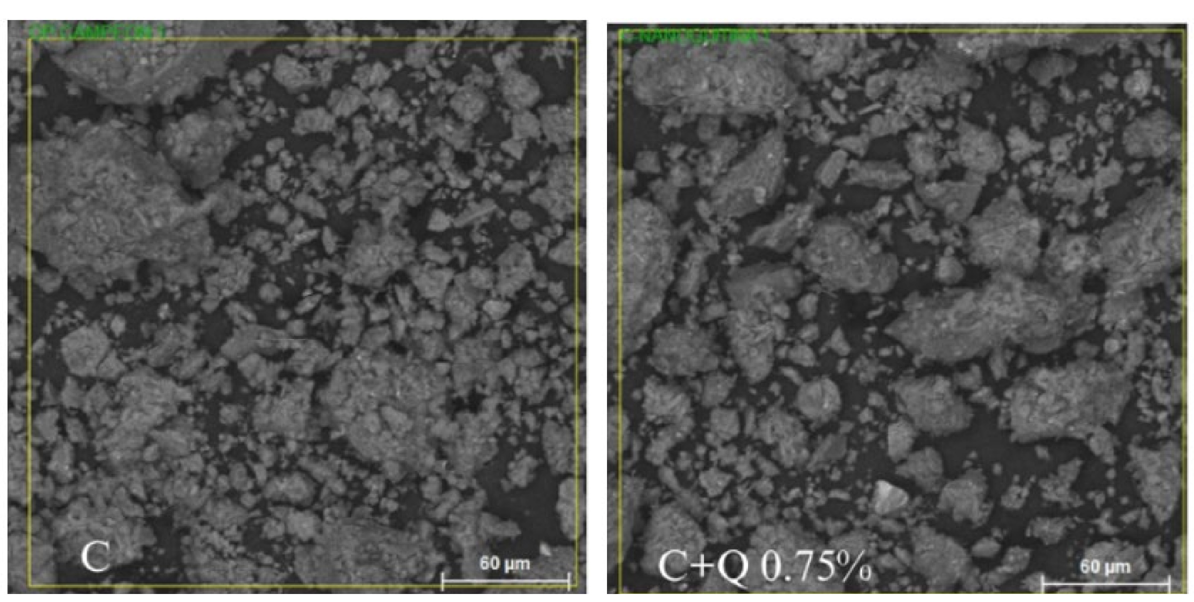
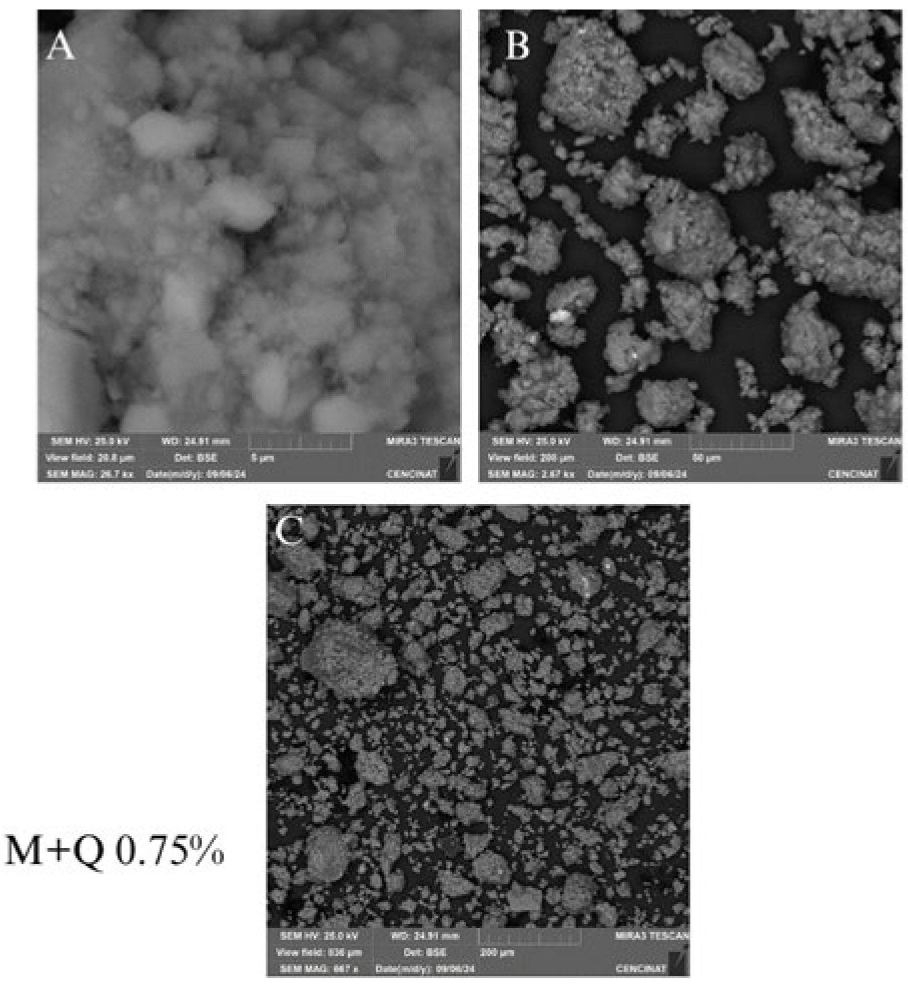


FIGURE 16

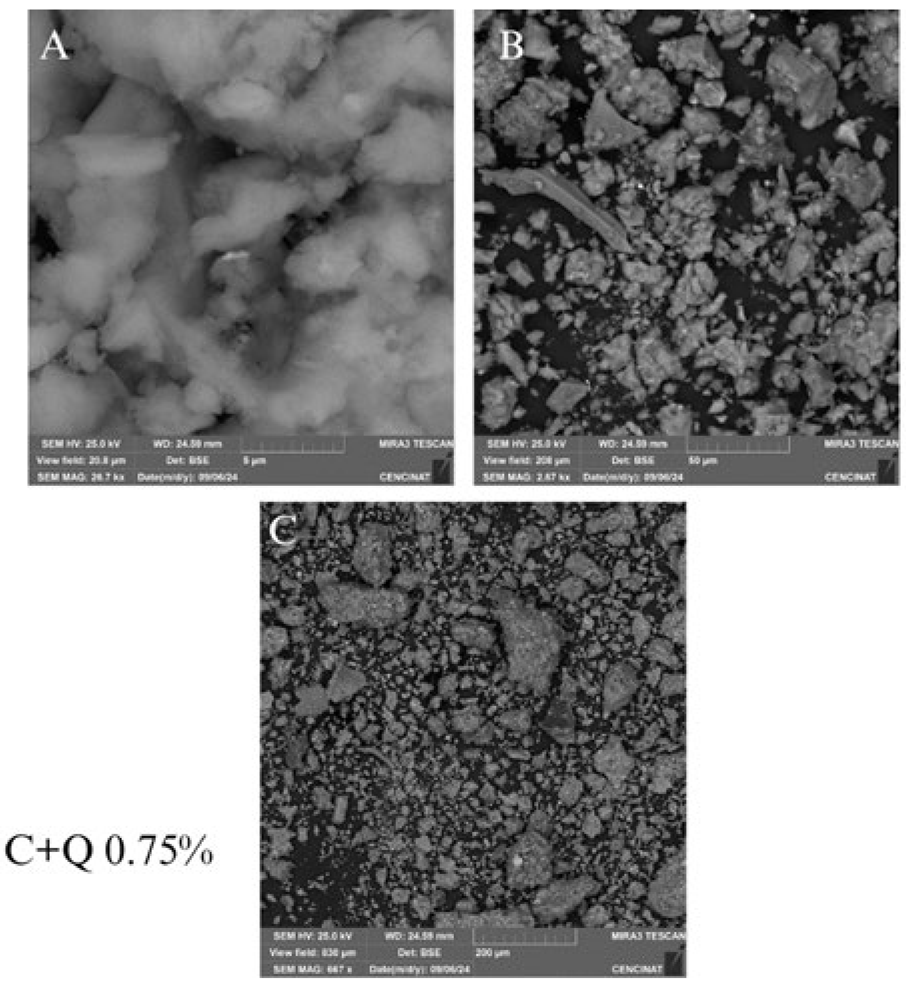
C+Q0.75%

Transmission Electron Microscopy (TEM)

TEM images provide detailed insight into the atomic distribution of nanomaterials, revealing particle sizes ranging from 5 µm to 200 µm. These images show a more uniform particle distribution and a significant reduction in porosity, indicating a denser and more compact structure. Such characteristics suggest enhanced durability and improved long-term resistance to both mechanical stress and environmental conditions. In contrast, the base samples M and C exhibit a more dispersed and porous microstructure, which implies lower cohesion and reduced mechanical strength. Conversely, the modified samples M+Q0.75% and C+Q0.75% display a fibrous structure that contributes to increased toughness and improved load absorption capacity.



M+Q 0.75%



C+Q 0.75%

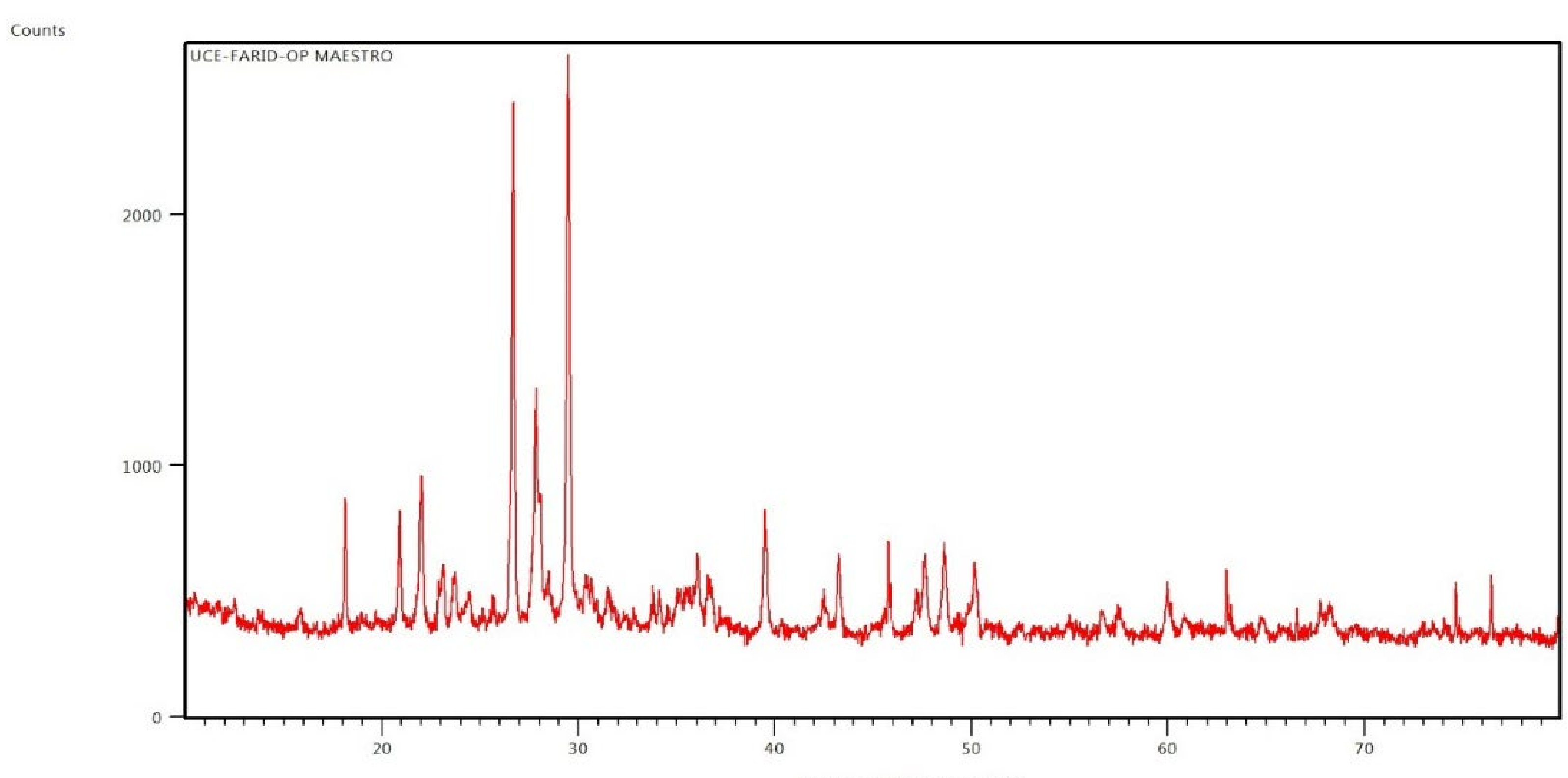
FIGURE 18

M+Q0.75% and C+Q0.75%

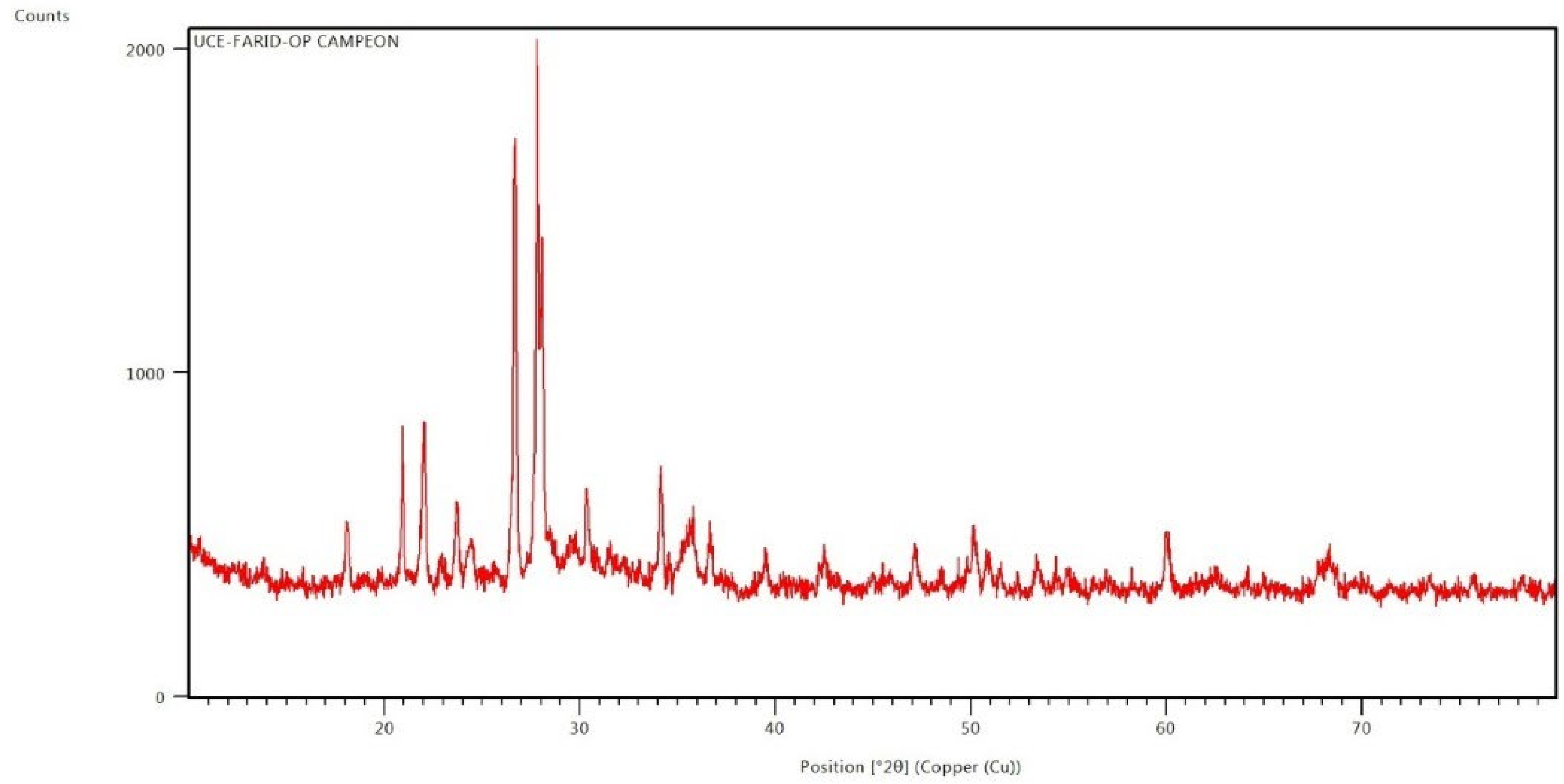
Fibrous structure, improving toughness and load absorption

X-Ray Diffraction (XRD)

XRD analysis allows for the identification and confirmation of crystalline phases within the mixtures. In the case of nanochitin, the presence of crystalline phases contributes to improved cohesion and durability. The M+Q0.75% sample exhibits well-defined peaks, indicating a predominant crystalline phase that is likely associated with the integration of nanochitin, which may also promote the formation of additional crystalline compounds that enhance structural stability. Similarly, the C+Q0.75% sample shows peaks at positions consistent with previous samples, suggesting effective incorporation of nanochitin and its positive contribution to the material's structural cohesion (Figure 19).



M+Q0.75%



C+Q0.75%

FIGURE 19

X-Ray Diffraction

Permeability

Figure 20 shows contact angle measurements using the drop method on different mortar samples modified with nanochitin and compared to a reference cement sample. The contact angle is a key parameter in evaluating the surface hydrophobicity of a material, indicating its ability to repel water. A technical interpretation of the results is provided below.

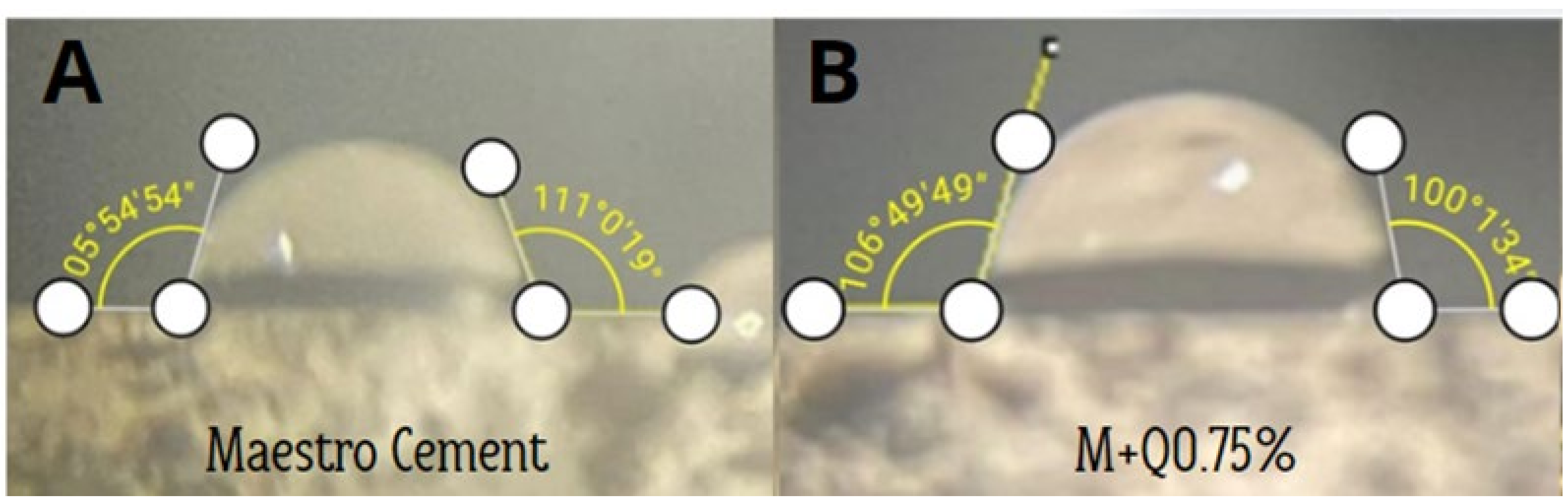


FIGURE 20

Permeability by Drop Method Test – Maestro Cement

Presents the contact angle analysis for Maestro Cement (A) and Maestro Cement modified with 0.75% nanochitin (M+$Q_{0.75\%}$) (B). A notable increase in the contact angle is observed in the sample modified with nanochitin, indicating a higher hydrophobicity and a lower water absorption capacity compared to the reference cement. This behavior suggests that the incorporation of nanochitin enhances the water-repellent properties of the mortar, potentially reducing permeability and increasing resistance to moisture-related degradation.

These results confirm that nanochitin acts as a hydrophobic agent, reducing water interaction with the cementitious matrix. This can lead to improved durability by minimizing moisture infiltration, which is particularly beneficial for structures exposed to aggressive environmental conditions. Additionally, the presence of nanochitin may contribute to better adhesion and cohesion of the mortar components, further optimizing its mechanical performance over time.

Figure 21 presents the contact angle analysis of mortars made with Campeón Cement (A) and those incorporating 0.75% nanochitin (C+Q0.75%) (B).

The modified mixture exhibits an increase in the contact angle, indicating a reduction in wettability and lower water absorption. This behavior suggests an enhancement in the hydrophobicity of the cementitious matrix, which may result in decreased porosity and greater resistance to the penetration of aggressive agents.

These findings confirm that the incorporation of nanochitin contributes to reducing capillary absorption, potentially improving the durability and resistance of the mortar against degradation, particularly in environments exposed to adverse conditions. Additionally, the hydrophobic nature of nanochitin may help mitigate the risk of moisture-induced damage, thereby enhancing the long-term performance and structural integrity of the material.

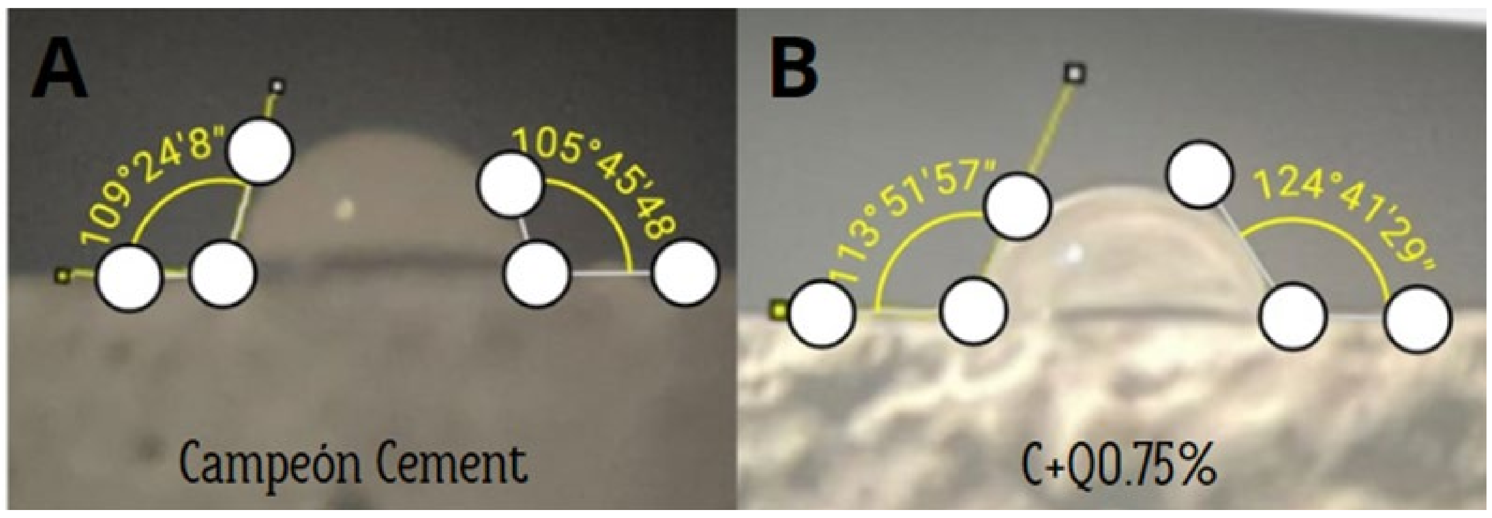


FIGURE 21

Permeability through the Drop Method test – Campeón Cement

Table 7 presents the results of the permeability test using the drop method for the 4 mortar samples, highlighting the effect of the different nanoparticles on the behavior and performance of the material.

TABLE 7

Drop Test (Contact Angle Measurement)

Mix Type	Contact Angle	Surface Type
M	108.3°	Hydrophobic > 90°
M+$Q_{0.75\%}$	103.4°	Hydrophobic > 90°
C	107.5°	Hydrophobic > 90°
C+$Q_{0.75\%}$	118.6°	Hydrophobic > 90°

Table 7 presents the contact angle values for different mortar mixtures, categorizing their behavior as hydrophobic (>90°).

The results indicate that all mixtures exhibit a hydrophobic surface, reflecting low water absorption. However, the mixture containing Campeón Cement and 0.75% nanochitin (C+$Q_{0.75}$%) demonstrates the highest contact angle (118.6°), suggesting increased resistance to water penetration and improved compaction of the cementitious matrix.

These findings confirm that the incorporation of nanochitin contributes to reducing porosity and enhancing the durability of the mortar, which is advantageous for improving its performance in challenging environmental conditions. Furthermore, the elevated contact angle observed in the C+$Q_{0.75}$% mixture indicates greater hydrophobicity, which may help minimize capillary absorption and improve the material's long-term resistance.

Sustainability Analysis

The reduction of CO_2 emissions resulting from the use of nanoparticles obtained from crab waste was assessed. In Ecuador, 8.0 tons of waste are generated annually, leading to an estimated emission of 3,856 tons of CO_2.

CO_2 emissions from the production of mortars incorporating nanoparticles derived from crab waste were analyzed in Table 8.

TABLE 8

CO_2 Emissions from 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	%
Nanochitin	212.7	167	349	900	1416	3856	37%

Environmental Impact of Nanomaterials

Nanochitin contributes to a 37% reduction in CO_2 emissions from crab waste decomposition.
The data highlights that the use of nanochitin in cementitious materials helps mitigate greenhouse gas emissions, reducing the environmental footprint of construction processes.

These findings support the implementation of circular economy strategies, as they promote the recycling of crab waste into value-added nanomaterials for sustainable applications in the construction industry.

CONCLUSIONS

The incorporation of nanochitin in cement mortars significantly improves mechanical properties, with 0.75% nanochitin formulations demonstrating competitive compressive. Nanochitin enhances internal cohesion and adhesion, leading to a denser cementitious matrix, which contributes to greater durability and structural stability over time.

The use of nanochitin derived from crab shell waste supports sustainable construction practices, promoting waste valorization and aligning with circular economy principles by repurposing food industry byproducts.

Water absorption tests confirm that nanochitin-modified mortars exhibit hydrophobic behavior, with contact angles exceeding 90°, reducing moisture absorption and permeability, thus enhancing resistance to chemical attacks and corrosion in aggressive environments.

The low porosity and improved compaction of nanochitin-enhanced mortars contribute to higher resistance against environmental degradation, making them suitable for long-term structural applications.

The findings comply with NTE INEN 2518 standards, validating the technical feasibility of incorporating nanochitin into high-performance mortar formulations for structural and industrial applications.

Exploring other sources of biological waste for nanoparticle extraction could further enhance the sustainability and efficiency of cementitious materials, reduce environmental impact and promote greener construction solutions.

The ability of nanochitin to improve mortar workability while maintaining structural integrity suggests its potential as an alternative to traditional additives in cement-based materials.

The combination of nanochitin with other nanomaterials, such as nanosilica or lime nanoparticles, may lead to synergistic effects, further optimizing mechanical properties, water resistance, and durability for advanced cementitious applications.

Authors' contributions

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Hugo Alexander Cadena-Perugachi: Investigation

Natali Elizabeth Lascano-Robalino: Validation

Jorge Alexander Bucheli-García: Conceptualization, Software

Edwin Iván Soledispa-Pereira: Validation, Writing – Review & Editing

Jorge Oswaldo Crespo-Bravo: Conceptualization, Investigation, Methodology, Resources

Pablo Mauricio Bonilla-Valladares: Investigation, Software, Writing – Original Draft

Alexis Patrice Martial-Debut: Validation, Writing – Review & Editing

Ángel Mauricio Espinoza-Cotera: Conceptualization

Byron Giovanoli Heredia-Ayala: Software

Jhon Fabricio Tapia-Vargas: Software

Assignment of rights and declaration of conflict of interests

 The authors declare that they have respected the ethical principles of research and that they are free of any conflicts of interest.

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