Advancing Sustainable Construction: Analysis and Evaluation of Replacing Aerosil 200 Nano-Silica with Rice Husk Ash Nano-Silica in High-Strength Concrete

Fomento de la construcción sostenible: análisis y evaluación de la sustitución de nanosílice Aerosil 200 por nanosílice de ceniza de cáscara de arroz en hormigón de alta resistencia

EÍDOS Nº 26
Scientific Journal of Architecture and Urbanism
ISSN: 1390-5007
revistas.ute.edu.ec/index.php/eidos

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***Abstract**:*

The search for alternative material sources to innovate in the construction industry is continuously evolving. This drives the exploration of synthesis methods that facilitate local production of sustainable materials, while maintaining high quality standards and reducing costs. This study focuses on identifying a new source of silica nanoparticles capable of maintaining or improving the physicochemical properties of high-

Received: 28, 04, 2025 - Accepted: 28, 05, 2025 - Published: 01, 07, 2025

strength concrete, in comparison with the commercial product Aerosil 200, which has limited availability. The nano silica used in this research is obtained from rice husk ash sourced from the rice milling plant in Mocache, Los Ríos, Ecuador, through a chemical synthesis process that produces silicon dioxide nanoparticles. These nanoparticles replace 1.5% of the total cement weight in optimized high-strength concrete mixes. The results of this partial replacement have proven to be economically viable, with significant improvements in mechanical properties, particularly at early ages, including compressive strength, indirect tensile strength, elastic modulus, and Poisson's ratio. Additionally, these improvements have been confirmed through advanced analyses using scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray diffraction (XRD), which allow for a detailed study of the material's chemical composition, particle size, and microstructure.

***Keywords**: Rice husk ash, nanometric silicon dioxide, optimized mix design, enhancement of physicochemical properties in concrete.*

Resumen:

La búsqueda de fuentes alternativas de materiales para innovar en la industria de la construcción está en constante evolución. Esto impulsa la exploración de métodos de síntesis que faciliten la producción local de materiales sostenibles, manteniendo altos estándares de calidad y reduciendo costos. Este estudio se centra en la identificación de una nueva fuente de nanopartículas de sílice capaz de mantener o mejorar las propiedades fisicoquímicas del hormigón de alta resistencia, en comparación con el producto comercial Aerosil 200, cuya disponibilidad es limitada. La nanosílice utilizada en esta investigación se obtiene de ceniza de cascarilla de arroz proveniente de la planta de molienda de arroz de Mocache, Los Ríos, Ecuador, mediante un proceso de síntesis química que produce nanopartículas de dióxido de silicio (SiO_2). Estas nanopartículas reemplazan el 1.5 % del peso total del cemento en mezclas optimizadas de hormigón de alta resistencia. Los resultados de esta sustitución parcial han demostrado ser económicamente viables, con mejoras significativas en las propiedades mecánicas, especialmente en etapas tempranas, incluyendo la resistencia a la compresión, la resistencia a la tracción indirecta, el módulo elástico y el coeficiente de Poisson. Además, estas mejoras se han confirmado mediante análisis avanzados mediante microscopía electrónica de barrido (MEB), microscopía electrónica de transmisión (MET) y difracción de rayos X (DRX), que permiten un estudio detallado de la composición química, el tamaño de partícula y la microestructura del material.

Palabras claves: Ceniza de cascarilla de arroz, dióxido de silicio nanométrico, diseño optimizado de mezclas, mejora de las propiedades fisicoquímicas del hormigón.

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1. INTRODUCTION

The growing demand for more sustainable and efficient construction materials has driven research into the use of nanometric additives to enhance the properties of concrete. The various applications of nanoparticles not only reveal the potential of the nanomaterials considered in this review but also indicate the way forward for improving concrete through nanotechnology (Zhang, 2018).

One of the most representative agro-industries in Ecuador is rice processing, which generates waste such as straw, husks, ash, bran, and broken rice (Alvansazyazdi et al, 2024a). In this context, rice husk ash (RHA) emerges as a promising alternative. RHA is an abundant byproduct of the agricultural industry that, when properly processed, can produce nano silica with properties comparable to Aerosil 200 (Zambrano, 2021).

According to the National Institute of Statistics and Censuses (INEC) in conjunction with the Ministry of Agriculture and Livestock, rice cultivation ranks fourth in production in Ecuador, with approximately 1.6 million tons harvested annually, generating about 20% husk (Pode, 2016).

The disposal of this waste represents a pollution challenge that all countries must face, as rice is one of the most widely cultivated plants in the world, with a production of around 1.2 billion tons per year. Consequently, tons of waste are generated daily, much of which lacks an adequate final disposal (Avila et al., 2019).

The development of advanced construction materials not only depends on material science innovations but also on the systematic management of experimental data and project progress, where tools such as web-based executive dashboards have proven to be critical (Alvansazyazdi et al., 2019a). Moreover, the ability of organizations to rapidly adapt and integrate emerging technologies, such as nano-silica derived from agricultural waste, is fundamental for innovation and competitiveness in sustainable construction (Alvansazyazdi et al., 2019b).

Concrete is one of the most widely used materials in civil engineering projects, composed of a combination of several components, including cement, water, coarse aggregates, and fine aggregates (Alvansazyazdi et al., 2024a). Additionally, an additive known as an admixture can be incorporated into the mixture. The inclusion of nanomaterials in the concrete matrix is believed to enhance its physic mechanical properties due to their fineness and cementitious properties, allowing for the production of denser concrete with greater durability and strength (Alvan et al., 2022a). The inclusion of nanoparticles as partial cement substitutes has significantly improved the mechanical properties of concrete, notably increasing compressive and tensile strength (Morales et al., 2020). Silica dioxide nanoparticles (SiO_2) react with calcium hydroxide in the cement matrix to form calcium silicate hydrate (C-S-H), resulting in a denser and stronger structure. The inclusion of 1.5% nano silica was found to be the optimal percentage for weight replacement of cement, achieving better mechanical properties in high-performance concrete. This approach not only has the potential to reduce costs and environmental impact but also promotes the valorization of agricultural waste, contributing to the sustainability of the construction industry (Janampa, 2021).

However, the production and use of nano silica from alternative sources such as RHA require more efficient and sustainable synthesis methods (Nakamura, 2019). Implementing such additives helps reduce dependence on conventional materials and minimizes the negative environmental impact of concrete production. Furthermore, utilizing agricultural byproducts like rice husk ash fosters a circular economy by transforming waste into valuable resources for construction (Costa & Paranhos, 2018).

This study focuses on the synthesis of nano silica from rice husk ash carried out in the Colloid Chemistry Laboratory of the Faculty of Chemical Sciences at the Central University of Ecuador (Lascano Robalino & Soledispa Pereira, 2025). During the synthesis process, 30 grams of nano silica were obtained from 2 kg of rice husk, which presents a challenge in terms of yield, but two crystalline forms of silica, rich in silicon content, were successfully produced.

Preliminary results indicate significant improvements in compressive strength and durability of the concrete. Pozzolanic activity increases as the age of the concrete progresses, plateauing around 90 days, indicating that mechanical strength continues to rise during this period (Alvansazyazdi et al., 2023). This suggests that RHA nano silica is a viable alternative to Aerosil 200. Regarding mechanical properties, nano silica contributes to increased strength at early ages due to its ability to accelerate the hydration process of the cement (Alvansazyazdi et al., 2022a).

Scanning Electron Microscopy (SEM) is an advanced tool used to analyze the microstructure of concrete at the microscopic level (Williams y Carter, 2009). This is essential for optimizing concrete formulation and understanding how certain factors, such as water/cement ratio and the use of additives, affect its performance (Bentley, 2012).

Transmission Electron Microscopy (TEM) is an advanced analytical tool used to examine the microstructure of materials at the nanometric scale. This technique is crucial for researching additives such as nano silica, as it allows observation of their dispersion and interaction with the cement matrix (Williams y Carter, 2009).

X-Ray Diffraction (XRD) is an analytical technique used to identify the crystalline phases present in a concrete sample. This method is particularly useful in the study of cementitious materials, as it allows evaluation of the mineralogical composition of cement and its hydration products, which directly influences the mechanical and durability properties of the concrete. Each mineral produces a unique diffraction pattern, facilitating its identification (Cullity y Stock, 2001).

2. EXPERIMENTAL PLAN

2.1 Synthesis of Nano Silica from Rice Husk Ash

To obtain nano silica, several steps must be followed to remove components and contaminants found in the rice husk ash. Table 1 and Figure 1 illustrate the sequence of the chemical process that leads to the production of this nanometric substance, which enhances concrete mixtures.

Table 1. Chemical Process for Nano Silica Production

Sequence	Chemical Process
A	Pyrolysis of the Original Sample
B	Ash Formation
B	Ash Reflux with Mineral Acids
C	Ash Washing
D	Reflux with Sodium Hydroxide
E	Precipitation and Sol-Gel Formation

The optimization of the synthesis parameters, including acid concentration and reflux cycles, plays a crucial role in maximizing the quality and yield of nano-silica. Similar optimization strategies have been successfully applied in surface engineering through the use of NSGA-II algorithmsThe

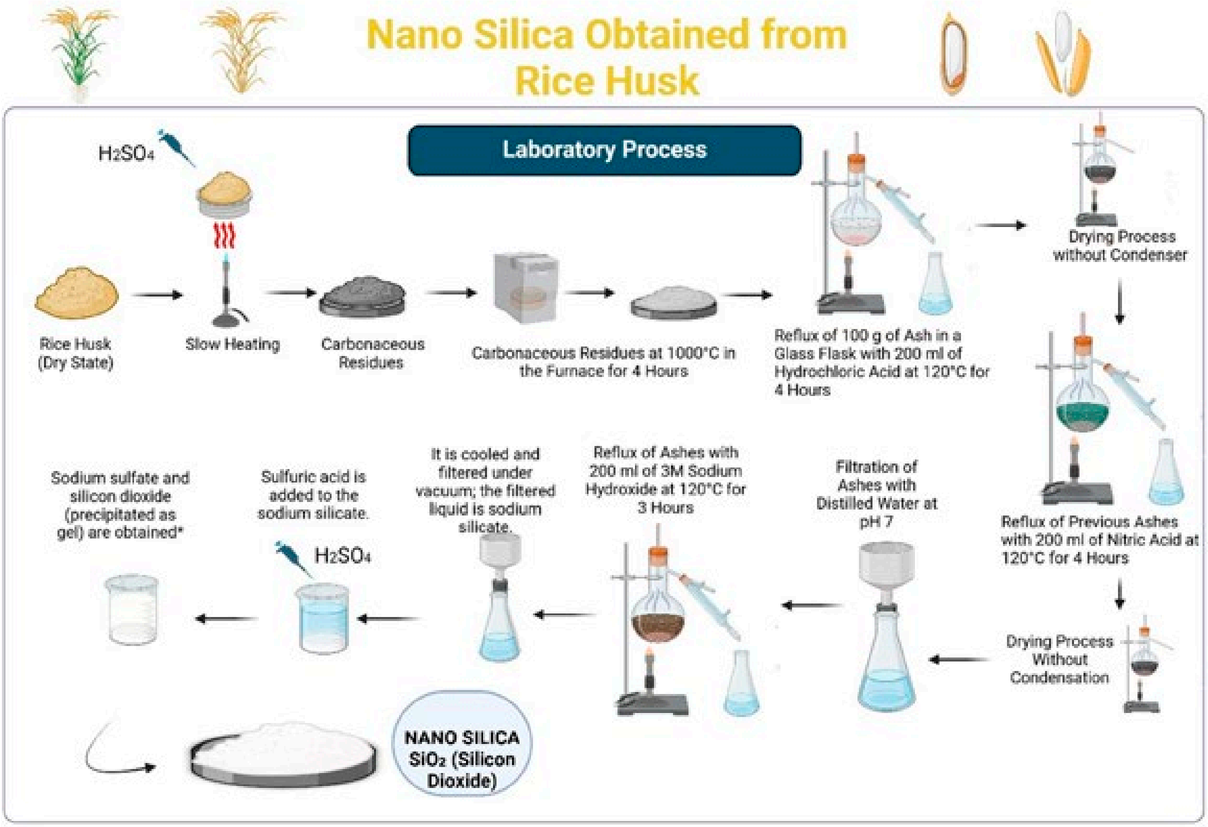

Figure 1. Chemical Process for Obtaining Nano Silica from Rice Husk Ash

optimization of the synthesis parameters, including acid concentration and reflux cycles, plays a crucial role in maximizing the quality and yield of nano-silica. Similar optimization strategies have been successfully applied in surface engineering through the use of NSGA-II algorithms (Golsha et al., 2012).

2.1.1 Results Obtained from Rice Husk

Figures 2 and 3 illustrate the two phases of nano silica obtained through the synthesis of rice husk: amorphous silica (SiO_2) and cristobalite. The amorphous form of silica stands out due to its high reactivity, promoting greater compaction and densification of the concrete microstructure, which translates into significant improvements in its mechanical performance. In contrast, cristobalite, a crystalline phase of silica, although less reactive, provides key advantages in terms of thermal resistance and structural stability, optimizing the performance of concrete under high temperatures and in aggressive environments (Wu et al., Compressive Strength Prediction of High-Strength Concrete Using Long Short-Term Memory and Machine Learning Algorithms, 2022).

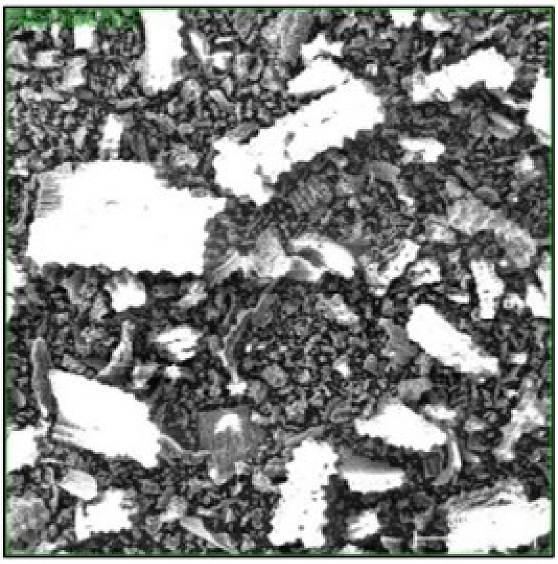

Figure 2. Cristobalite Obtained from Rice Husk Synthesis

Figure 2 (Cristobalite): The particles in this figure are predominantly laminar or plate-like, with angular edges and relatively flat surfaces. The image reveals a highly granulated structure with particles distributed relatively uniformly. This pattern is characteristic of crystalline materials, where cristobalite forms well-defined crystals. The lighter grains appear to be larger cristobalite aggregates, while the darker areas may represent less dense regions or impurities.

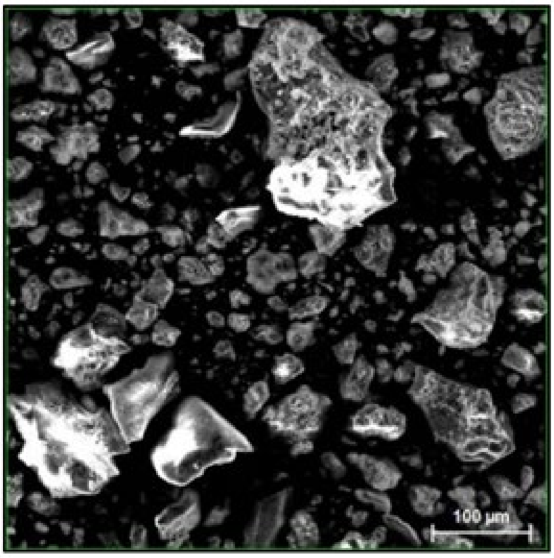

Figure 3. SiO_2 Obtained from Rice Husk Synthesis

Figure 3 (SiO_2): The particles in this figure are angular and irregular with sharp edges and rough surfaces. The silicon dioxide shown in this image appears to exhibit a bimodal particle size distribution, with a mixture of fine particles and larger, rougher ones. This distribution is ideal for enhancing density and strength in applications such as high-performance concrete, as the fine particles can act as an effective filler, while the larger particles improve the stiffness and mechanical strength of the material. Furthermore, the roughness of the larger particles suggests optimal interaction with the cement matrix, resulting in greater bonding capacity and possibly an increase in the durability of the final material.

2.2 Concrete Mix Design

The concrete mix design was carried out using the ACI 211.4 code, which is specific for high-strength concretes. Initially, a General Use (GU) type cement was used, but the results were unsatisfactory. Therefore, the mix was adjusted to use a High Early Strength (HE) type cement, which is better suited for achieving the desired strength in a shorter time frame. For the concrete tests analyzed, there are certain regulatory requirements that justify the sampling and testing procedures for each experiment. These guidelines are followed to ensure a systematic approach, leading to more reliable and consistent results.

2.2.1 Aggregate

For the aggregates, several tests based on the INEN NTE 872 standard must be applied to determine the suitability of the ag-

Table 2. Aggregate Tests for High-Strength Concrete

Items	Description
Characterization of the Aggregates	This test is carried out in accordance with INEN 872: Aggregates for Concrete - Requirements. It included tests for gradation, absorption, density, and moisture content to ensure compliance with quality standards.
Test for Loose Bulk Density and Compacted Bulk Density of Aggregates	This test is performed to determine the bulk density and the void percentage of fine and coarse aggregates, in accordance with the NTE INEN 858 standard (NTE INEN 858, 2010).
Specific Gravity and Absorption Capacity Test	The tests to determine the specific gravity and absorption capacity of fine and coarse aggregates are carried out in accordance with NTE INEN 856 and 857 standards, which establish the test methods for measuring the relative density and absorption capacity of fine and coarse aggregates, respectively (NTE INEN 856, 2010).
Moisture Content Test	This test is conducted based on the INEN NTE 862 standard, which outlines the procedure for determining the moisture content in coarse and fine aggregates (NTE INEN 862, 2011).
Aggregate Gradation Test	The test for determining the particle size distribution of fine and coarse aggregates is carried out in accordance with the INEN NTE 696 standard. This standard establishes the procedure for determining the gradation of aggregates, ensuring their suitability for use in concrete mixes (NTE INEN 696, 2011).
Coarse Aggregate Abrasion Test	The coarse aggregate abrasion test follows the procedure and requirements based on the NTE INEN 860 standard, which aims to determine the degradation of coarse aggregate smaller than 37.5 mm due to wear and impact in the Los Angeles machine. This test serves as an indicator of the aggregate's quality (NTE INEN 860, 2011).
Colorimetry Test	The determination of the colorimetry test for fine aggregates is based on the NTE INEN 855 standard, which checks whether the sample contains high amounts of organic impurities in the fine aggregate for mortar and concrete mixes (NTE INEN 855, 2010).

Table 3. Aggregate data for mix design of the control sample and Nano Silica Sample

Aggregate Data from Copeto Quarry in Santo Domingo de los Tsáchilas					
Fine Aggregate			Coarse Aggregate		
Dry Loose Unit Weight	1648.33	kg/m^3	Dry Loose Unit Weight	1705.78	kg/m^3
Dry Compacted Unit Weight	1835.00	kg/m^3	Dry Compacted Unit Weight	1753.91	kg/m^3
Specific Gravity of Fine Aggregate	2742.78	kg/m^3	Apparent Specific Gravity of Coarse Aggregate	2744.54	kg/m^3
Absorption Percentage	1.13	%	Absorption Percentage	1.06	%
Moisture Content	4.58	%	Moisture Content	1.64	%
Fineness Modulus	3.3	-	Fineness Modulus	6.34	-

gregate for use in concrete mixes. Table 2 lists the tested items for this article along with their respective descriptions, and Table 3 provides the results of these tests.

2.2.2 Cement

The determination of the absolute density test for cement is based on the NTE INEN 156 standard, which is performed using the Le Chatelier volumetric flask method (NTE INEN 156, 2009). The results for the density of Selvalegre Type HE cement are shown in Table 4. The rapid strength gain of Type HE cement makes it an ideal choice for structures requiring quick progress in construction and commissioning, enhancing durability and reducing the porosity of the concrete (YURA S.A., 2024).

Table 4. Density of Selvalegre Type HE Cement

Cement Density	
Cement:	Selvalegre
Type	HE
Test	Cement Density (g/cm^3)
1	2.896
2	2.882
3	2.916
Average	2.898

2.2.3 Superplasticizer

Fresh concrete must allow for adequate workability to ensure proper handling and placement on-site, as it remains a heterogeneous material (Alvansazyazdi et al., 2022b). Superplasticizers increase the fluidity of concrete without the need for additional water, enhancing its strength and durability (Torres, 2021). Table 5 presents the results for the amount of superplasticizer used. For the control sample, 0.25% of the total cement weight was chosen, while for the nano silica sample, 0.35% of the total cement weight was selected to improve the workability and performance of the concrete mix containing nano silica derived from rice husk.

Table 5. Amount of Superplasticizer for the Control Sample and Nano Silica Sample

Amount of Superplasticizer	
Superplasticizer: VISCOCRETE 4100	
Description	Weight of Superplasticizer (g)
Control Concrete Sample	142.55
Concrete Sample with 1.5% of the Total Cement Weight Replaced by Nano Silica Obtained from Rice Husk	196.58

2.2.4 Nano silica

In the research conducted by (Alvan et al., 2022a), the objective is to test dosages of 0.75%, 1.5%, and 3% as replacement percentages of cement with nano silica from the product Aerosil 200, where the best result in the experiment was achieved with a 1.5% replacement. Therefore, this study is based on the optimal percentage of 1.5% replacement of the total cement weight with nano silica from rice husk (Janampa, 2021). Table 6 presents the results of the two types of products, noting the data for the Aerosil 200 product.

The use of nano silica in concrete not only improves mechanical properties but also significantly enhances its durability and resistance against aggressive agents (Al-Tawaiha et al., 2023). Nano silica obtained from organic materials, such as rice husk, has emerged as an eco-friendly and efficient solution for enhancing the properties of concrete. This type of nano silica exhibits high pozzolanic activity, enabling it to react with the calcium hydroxide released during cement hydration to form calcium silicate hydrate (C-S-H) gel, thereby improving compressive strength, impermeability, and durability of the concrete. The incorporation of nano silica derived from organic materials, such as rice husk, significantly enhances the mechanical properties of concrete, while also contributing to sustainability in its production (Alqamish y Al-Tamimi, 2021).

Table 6. Comparison of Properties: Aerosil 200 vs Rice Husk Nano Silica

Nano Silica (Aerosil 200 vs Rice Husk)			
Description	Unit	Aerosil 200	Nano Silica from Rice Husk
Compacted Density	g/l	50	60
Silicon Content	%	99.8	80
Particle Size	nm-um	3-100 nm	10 - 300 um
pH Value	pH	3.7-4.5	7.63

2.2.5 Water

Water is an essential component in the concrete mix, as it not only facilitates the workability of the mixture in its fresh state but also plays a crucial role in the hydration of cement and the development of strength. The quantity and quality of water are determinants for the durability, strength, and workability of concrete. According to studies, the water must be free from impurities such as oils, acids, or salts that could compromise the quality of the material. Ideally, the water used should be potable, with a pH close to neutral to avoid adverse effects on the hydration reaction of the cement, thereby improving mechanical properties and reducing risks associated with impurities (Shafaq Bazaz, 2023) (Kumar, 2021).

Table 7 details the results of the water used for the mix of the control concrete sample and the concrete sample with 1.5% of the total cement weight replaced by nano silica obtained from rice husk. This indicates a low water-cement ratio (w/c) of 0.29, considering that the amount of cement is high for the design of such concrete mixes.

Table 7. Water-Cement Ratio for High-Strength Concrete Design with f'c = 65.5 MPa

f'cr (psi)	**Ratio a/c**
10000	0.26
9316.29	0.29
9000	0.30

2.2.6 Dosage of Control Concrete and Concrete Sample with 1.5% Nano Silica Obtained from Rice Husk

Based on all the information presented, the design of high-strength concrete can be developed. Table 8 provides a summary of the dosages for the control sample and the sample with a 1.5% replacement of nano silica from rice husk.

Table 8. Dosage of the Control Sample and Concrete Sample with 1.5% Nano Silica Obtained from Rice Husk

Summary of Dosages for 1 m³ of Concrete						
Dosages	Cement	Water	Gravel	Sand	Nano silica	Superplasticizer
Unidad	(kg)	(kg)	(kg)	(kg)	(kg)	(kg)
Control Sample	636.86	162.02	1146.65	430.88	0	1.59
Sample NS-1.5% Rice Husk	627.30	162.02	1146.65	430.88	9.41	2.20

3. RESULTS OF HARDENED CONCRETE

3.1 Compressive Strength

Compressive strength is one of the fundamental properties of concrete, determining its ability to withstand applied loads without fracturing (Alvansazyazdi et al., 2024b). This parameter is commonly measured at 28 days through destructive tests on cured concrete cylinders. Key factors such as the water-cement ratio, the quality of the aggregates, and the presence of additives directly influence the strength of the concrete (Wu et al., 2022) (Lin y Wu, 2021). In high-strength concrete (HSC), this property exceeds 40 MPa, making it ideal for applications requiring durability and low permeability, such as large infrastructures and skyscrapers (Sihag, 2021).

Previous studies have demonstrated that the incorporation of nano-silica into cementitious matrices leads to significant improvements in mechanical performance and microstructural densification, promoting greater durability and strength (Alvansazyazdi et al., 2024c).

In the compression tests, three specimens were tested at various ages (1, 3, 7, 28, and 56 days). It is important to note that compressive strength is an indicator test of durability, allowing us to address various stresses that may occur in any infrastructure. Table 9 presents the values obtained from this test for the control sample and the sample with 1.5% rice husk nano silica. The most significant impact is observed at 7 days, with a 20.25% improvement. However, as time progresses, the additional benefit of nano silica diminishes, showing smaller improvements at 28 and 56 days.

In Figure 4, the compression strength curves over time are presented. The sample with nano silica (blue curve) shows a slight advantage over the control sample (grçeen curve) from the first day. This suggests that the addition of nano silica enhances the early strength gain, possibly due to improved hydration and a denser structure.

Table 9. Results of Compressive Strength for the Control Sample and Sample with 1.5% Rice Husk Nano Silica

Compression Test (MPa)			
Age	Control Sample	Sample with 1.5% Nano Silica	% Improvement with Nano Silica
	Average	Average	
1 day	45.10	50.47	10.65%
3 days	51.07	56.0967	8.96%
7 days	53.12	66.6067	20.25%
28 days	67.72	71.44	5.21%
56 days	68.52	71.44	4.09%

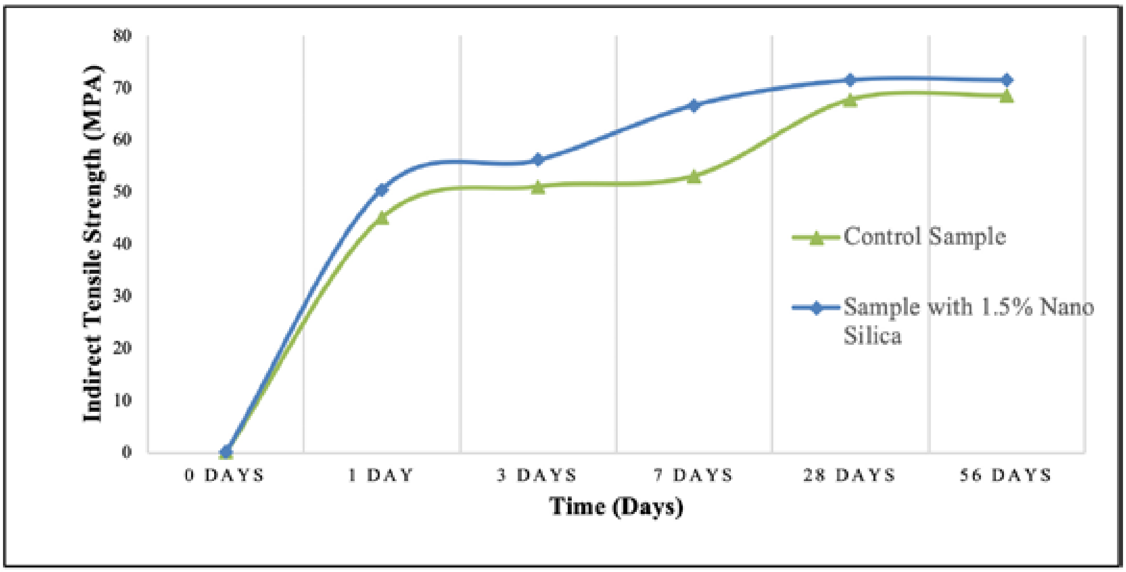

Figure 4. Compressive Strength Over Time for the Control Sample and Sample with 1.5% Rice Husk Nano Silica

3.2. Tensile Strength

The tensile strength of concrete is considerably lower than its compressive strength. It is essential for assessing its behavior under stress and preventing cracks that could compromise its durability. Tensile strength is primarily measured using direct tensile tests, beam bending tests, and the Brazilian test, with the latter being the most common (Ge, W. et al., 2022).

Direct tensile tests provide a more accurate measure of concrete's resistance under stress, but their complexity limits their use. The Brazilian test is the most commonly employed method, applying a load across the diameter of a cylinder, which generates a tensile failure in the cross-section, yielding more consistent results than beam bending tests (Wu et al., 2021).

Recent studies have evaluated the relationship between compressive strength and tensile strength of concrete, noting that an improvement in compressive strength (e.g., through the inclusion of steel fibers) also increases tensile strength. However, tensile behavior is more sensitive to the presence of defects and cracks in the material (Jiang et al., 2022).

Table 10 and Figure 5 present a summary of the tensile strength results for the ages of 1, 3, 7, 28, and 56 days. The incorporation of rice husk nano silica significantly enhances the indirect tensile strength on the first, third, and seventh days, with increases of 23.36%, 16.13%, and 20.82%, respectively. This indicates that nano silica provides better initial cohesion among the particles in the concrete.

Table 10. Results of Indirect Tensile Strength for the Control Sample and Sample with 1.5% Rice Husk Nano Silica

Indirect Tensile Test (MPa)			
Age	Control Sample	Sample with 1.5% Nano Silica	% Improvement with Nano Sica
	Average	Average	
1 día	2.95	3.85	23.36
3 día	3.20	3.82	16.13
7 día	3.66	4.63	20.82
28 días	4.62	4.64	0.43
56 días	4.67	4.75	1.68

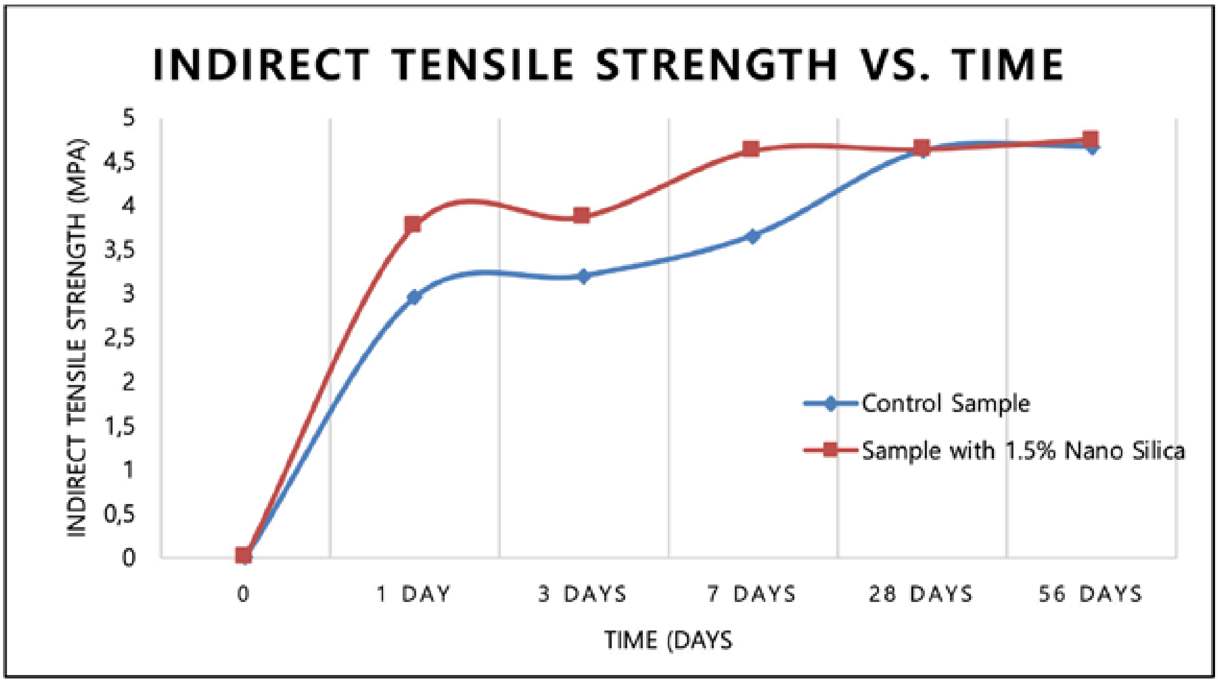

Figure 5. Graph of Indirect Tensile Strength vs. Time

3.3.Flexural Strength

Flexural tests are generally performed on beams, and the results are crucial for designing elements such as slabs and pavements. It has been shown that the addition of reinforcement fibers, such as polyethylene and polypropylene, significantly increases flexural strength, while the use of fly ash also contributes to improving both the strength and durability of concrete in aggressive environments (Wu et al., 2021) (Supit y Shaikh, 2015) (Ahmed et al., 2016).

Table 11 presents the results obtained from the flexural test at 28 days, with two specimens for each sample. The incorporation of 1.5% nano silica into the mix improved the flexural strength of the material by 3.27% after 28 days. Figure 6 shows the comparison of the flexural strength test results, where the increase in flexural strength is approximately 0.32 MPa with the incorporation of 1.5% nano silica, representing an improvement in the mechanical properties of the material.

Table 11. Comparison of Flexural Strength between Control Concrete and Concrete with 1.5% of Cement Weight Replaced by Nano Silica Obtained from Rice Husk

Flexural Test (MPa)			
Age	Control Sample	Sample with 1.5% Nano Silica	% Improvement with Nano Sica
	Average	Average	
28 días	9.39	9.71	3.27%

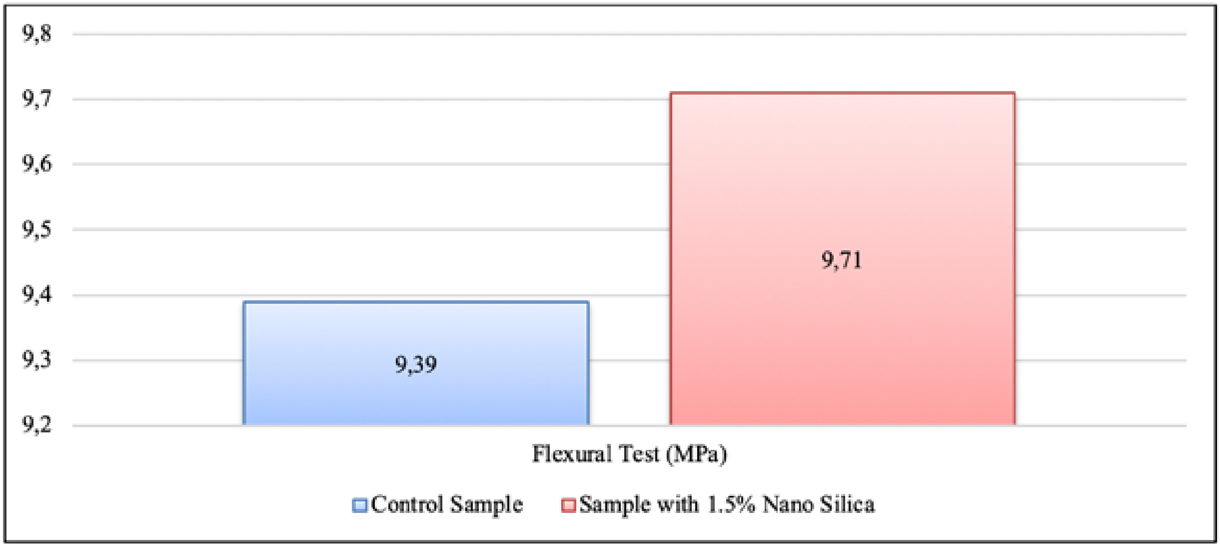

Figure 6. Summary of Flexural Strength Test of Control Sample vs. Sample with 1.5% Nano Silica

3.4. Results of X-Ray Diffraction (XRD) Test

The X-ray diffraction (XRD) test is an analytical technique used to identify and quantify the crystalline phases present in materials such as concrete. By diffracting an X-ray beam over the sample, unique patterns are obtained that allow for the determination of the mineralogical composition and the phases present in the material. This technique is particularly useful in the analysis of cement, as it facilitates the identification of compounds such as calcium silicate hydrate (C-S-H), portlandite, and other hydration products (Handoo et al., 2020).

In concrete research, XRD is applied to study the evolution of phases over time, the impact of additives, and the effects of external conditions, such as exposure to aggressive environments. It has been particularly useful in evaluating concrete mixes that contain industrial or agricultural waste, such as the incorporation of fly ash or concrete floor polishing residues, which directly influence the mechanical and chemical properties of the material (Silva, L. A. et al., 2020). Moreover, the use of techniques such as the Rietveld method, in conjunction with XRD, allows for a quantitative analysis of the crystalline phases in concrete, which is key to improving the durability and strength of the material in various applications (Wu et al., 2021).

This test enables the determination of the chemical composition of concrete samples, identifying the crystalline phases present in a sample and determining the structure of the material for both the control sample and the sample with 1.5% rice husk nano silica at 28 days.

In Figure 7, the peaks in the graph of the control concrete sample can be observed, showing several intensities, particularly the peak at 2θ = 28°, which is characteristic of calcite ($CaCO_3$). This indicates a significant carbonation process in the control concrete sample. This intense peak serves as an indicator of the presence of this mineral in the control concrete sample and suggests that carbonation has occurred to a certain extent.

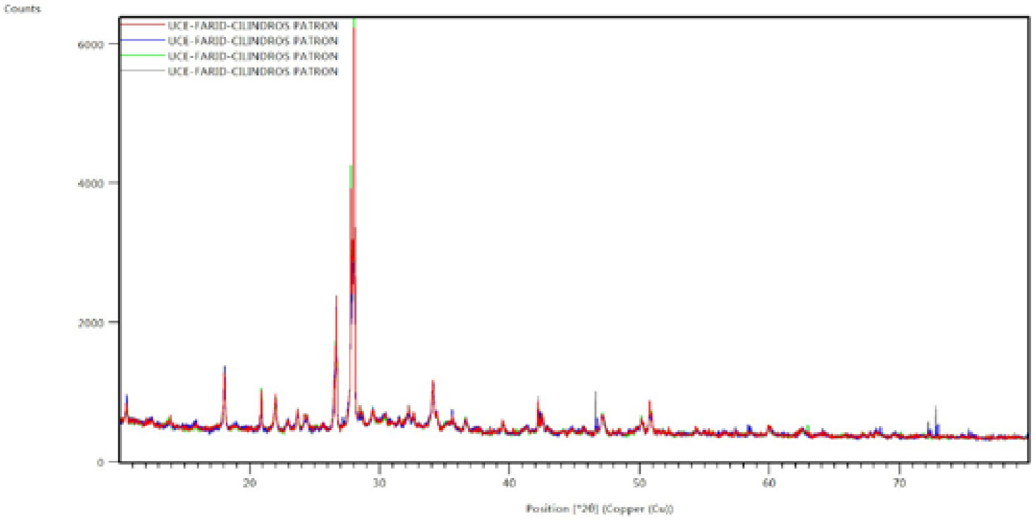

Figure 7. XRD Test of the Control Sample

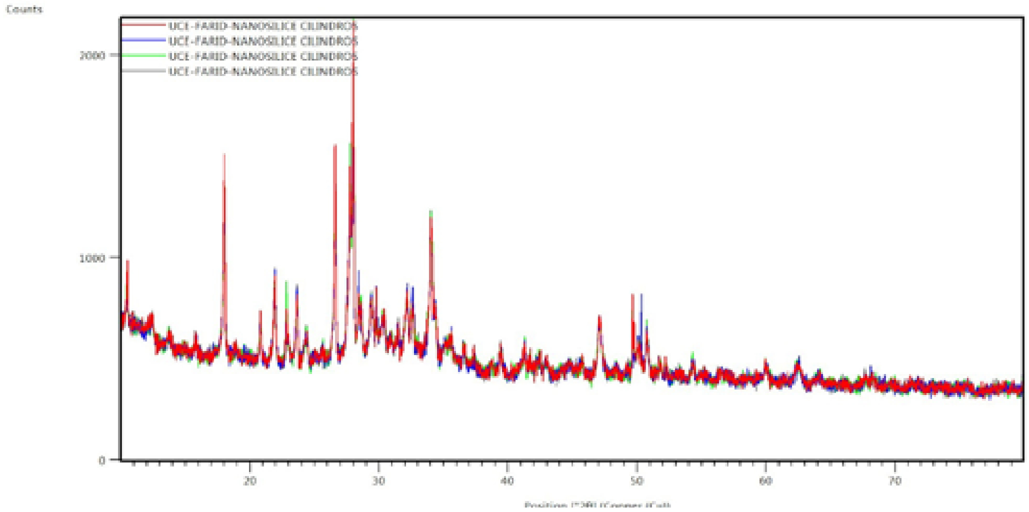

Figure 8. XRD Test of the Sample with 1.5% Rice Husk Nano Silica

In Figure 8, it can be observed that the peaks have lower intensity compared to the control concrete, which may indicate a reduction in the amount of portlandite and calcite crystals due to the interaction with the nano silica. This could also suggest that the presence of nano silica promotes a greater formation of amorphous or semi-crystalline products, such as C-S-H gel (calcium silicate hydrate).

3.5. Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a key tool in the analysis of the microstructure of concrete. This device allows for high-resolution images of the surfaces of materials, facilitating the identification and characterization of the phases present in the cement paste, aggregates, and hydration products. Additionally, the SEM is useful for studying the morphology and distribution of pores, the adhesion between the cement paste and aggregates, as well as structural defects such as microcracks that may compromise the durability of concrete (Stutzman, 2000).

The SEM is crucial for analyzing the impact of additives such as micro silica or nano silica, as it enables visualization of how these additives improve compaction and reduce the porosity of concrete. Through detailed images, differences between conventional and modified cement matrices can be observed, thereby optimizing high-strength mixes. Moreover, when combined with techniques such as Energy Dispersive Spectroscopy (EDS), it complements the chemical analysis of the hydrated phases (Balendran et al., 1998) (Zhao et al., 2021).

In Figure 9 (a-b), the results obtained from the scanning electron microscope (SEM) test are shown, which determines the particle sizes present in the control concrete sample. The scale value is visible for reference regarding the particles in the sample. The sizes range from 5 µm to 200 µm, concluding that there are both large and small particles, with black spaces between the particles corresponding to pores in the material's microstructure.

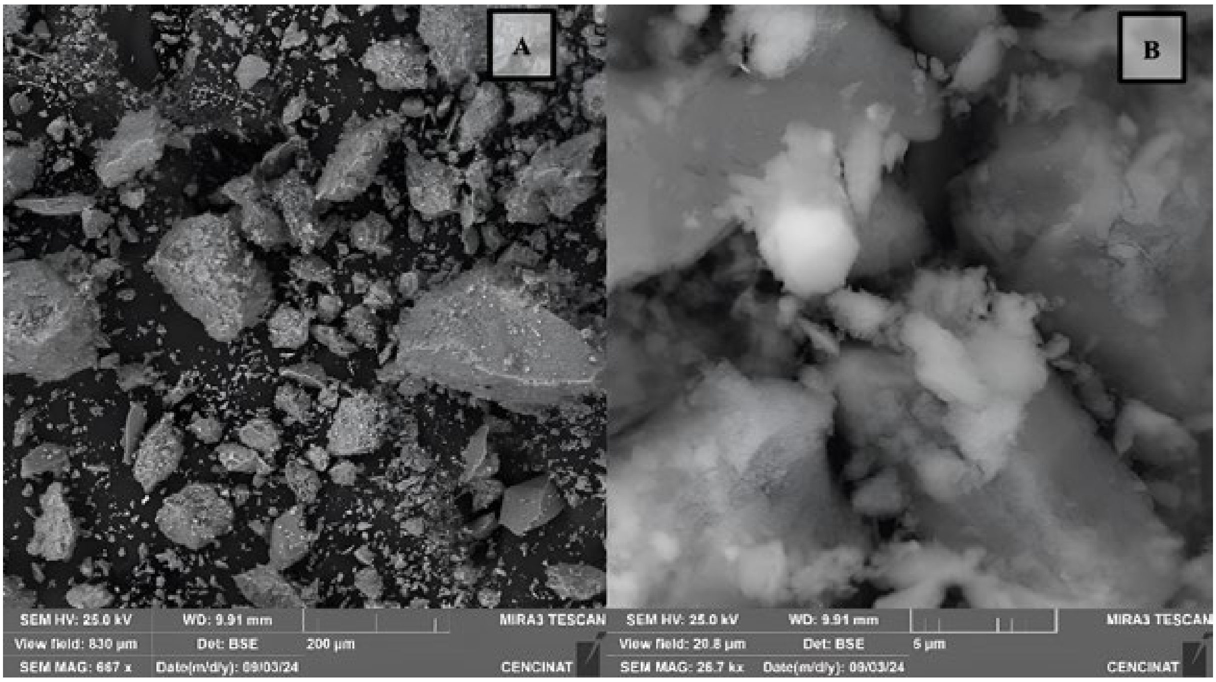

Figure 9. XRD Test of the Control Sample

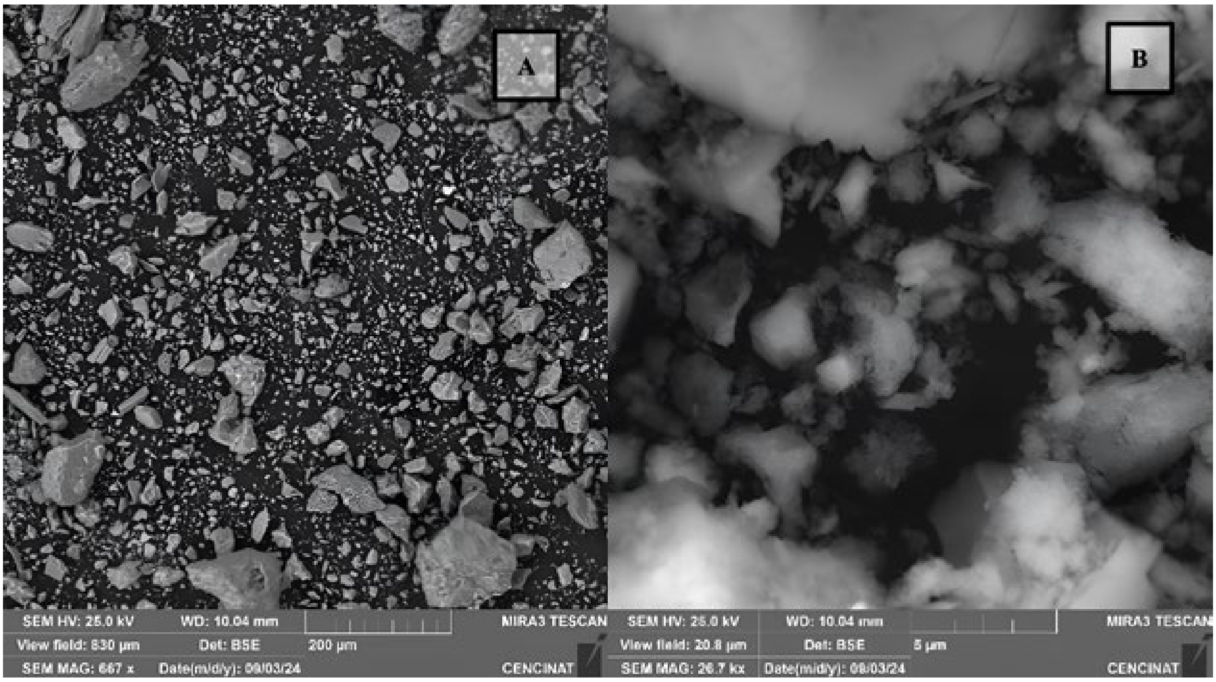

Figure 10. XRD Test of the Sample with 1.5% Rice Husk Nano Silica

In Figure 10 (a-b), it is evident that the particles are smaller compared to the control sample. This is due to the nano silica, which refines the microstructure of the mix by facilitating more effective hydration and the generation of C-S-H gel, resulting in a finer and more uniform particle distribution. In this case, the porosity is lower due to the smaller size and more interlocked particles. The nano silica has improved the quality of the mix by refining the particle size and reducing void spaces, resulting in stronger and more durable concrete.

3.6. Energy Dispersive X-ray Spectroscopy (EDS) Spectrum

The Energy Dispersive X-ray Spectroscopy (EDS) test allows for the identification and quantification of the elements present in concrete by analyzing its chemical composition at the microscopic level. This technique, often used in conjunction with Scanning Electron Microscopy (SEM), provides elemental mapping of the sample, which is essential for studying the distribution of additives and hydration products in the cement matrix. It is particularly useful for analyzing nano additives, such as nano silica, and evaluating the impact of recycled materials on concrete (Energy-Dispersive X-ray Spectroscopy (EDS), 2021) (Goldstein et al., 2021) (Zhao et al., 2021).

In Figure 11, the EDS spectrum is presented, showing the key components expected in Portland cement-based concrete: calcium, silicon, oxygen, iron, aluminum, magnesium, and sulfur, along with nickel. These elements correspond to the hydration products (C-S-H, portlandite, ettringite) as well as the original components of the clinker and gypsum. It is noted that the majority of the components correspond to calcium.

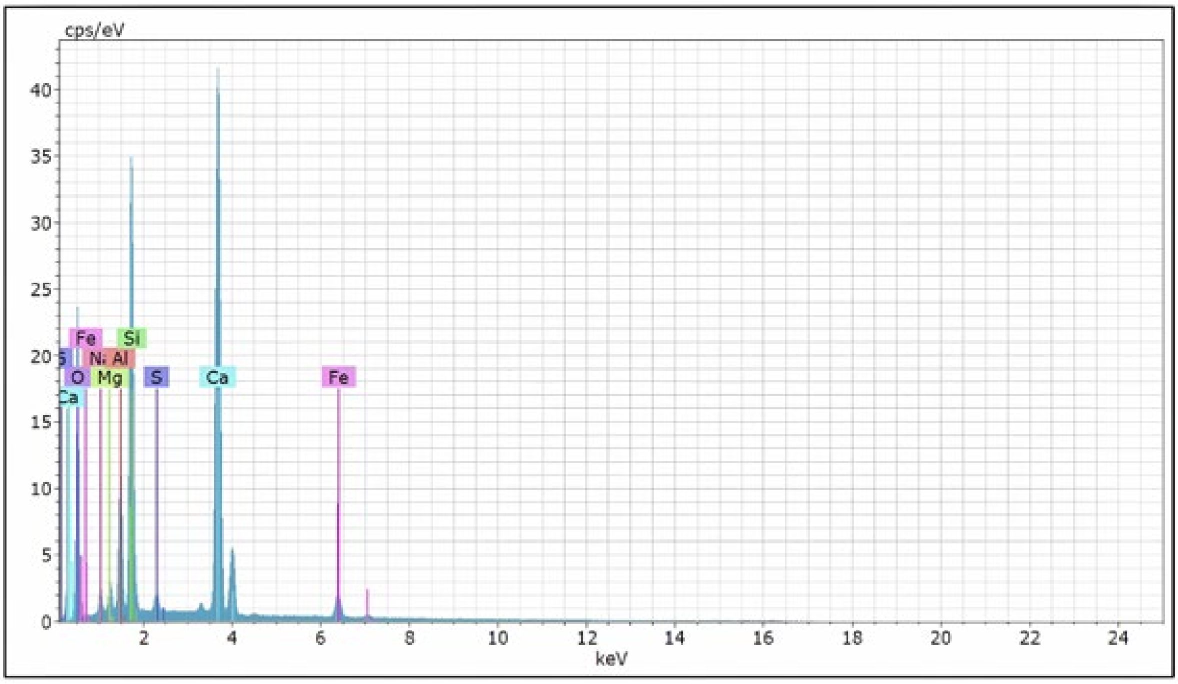

Figure 11. EDS Test of the Control Sample

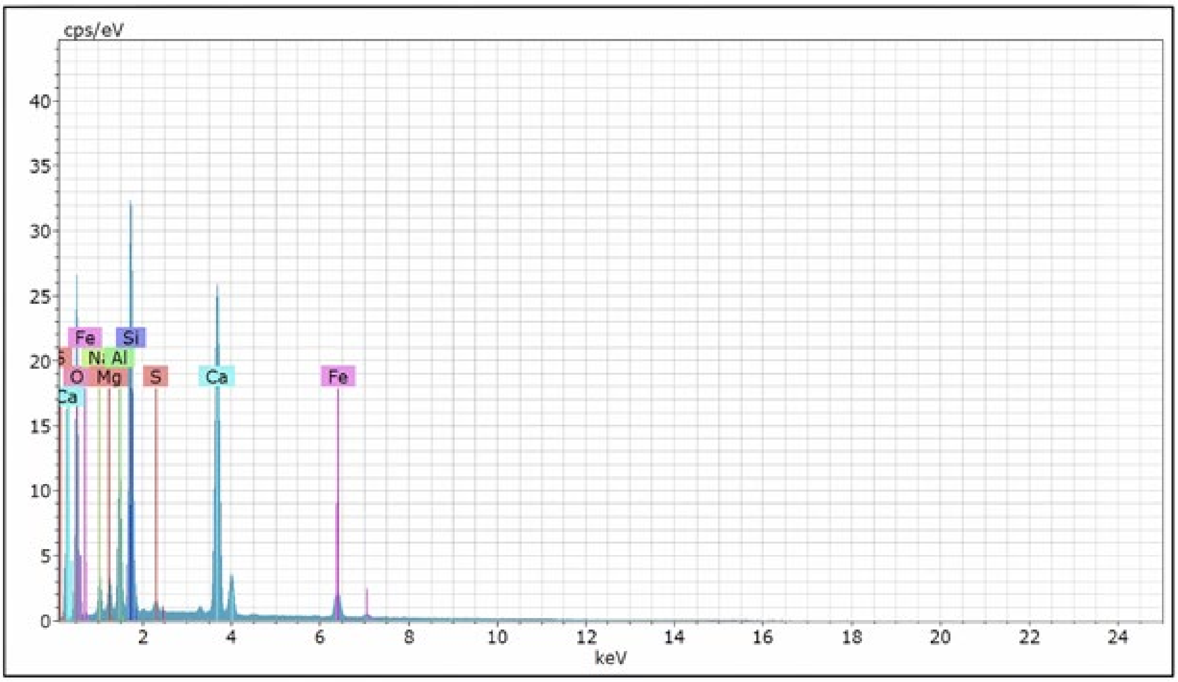

Figure 12. EDS Test of the Sample with 1.5% Rice Husk Nano Silica

In Figure 12, the EDS analysis results of the concrete sample with 1.5% rice husk nano silica are displayed. There is a clear and pronounced increase in the silicon content in the concrete, which ensures better formation of C-S-H gel, enhancing its mechanical properties through the interlocking of particles. The overall composition remains dominated by calcium, but the rice husk nano silica improves the microstructure of the material.

Figure 13 presents a summary of the tests conducted for hardened concrete:

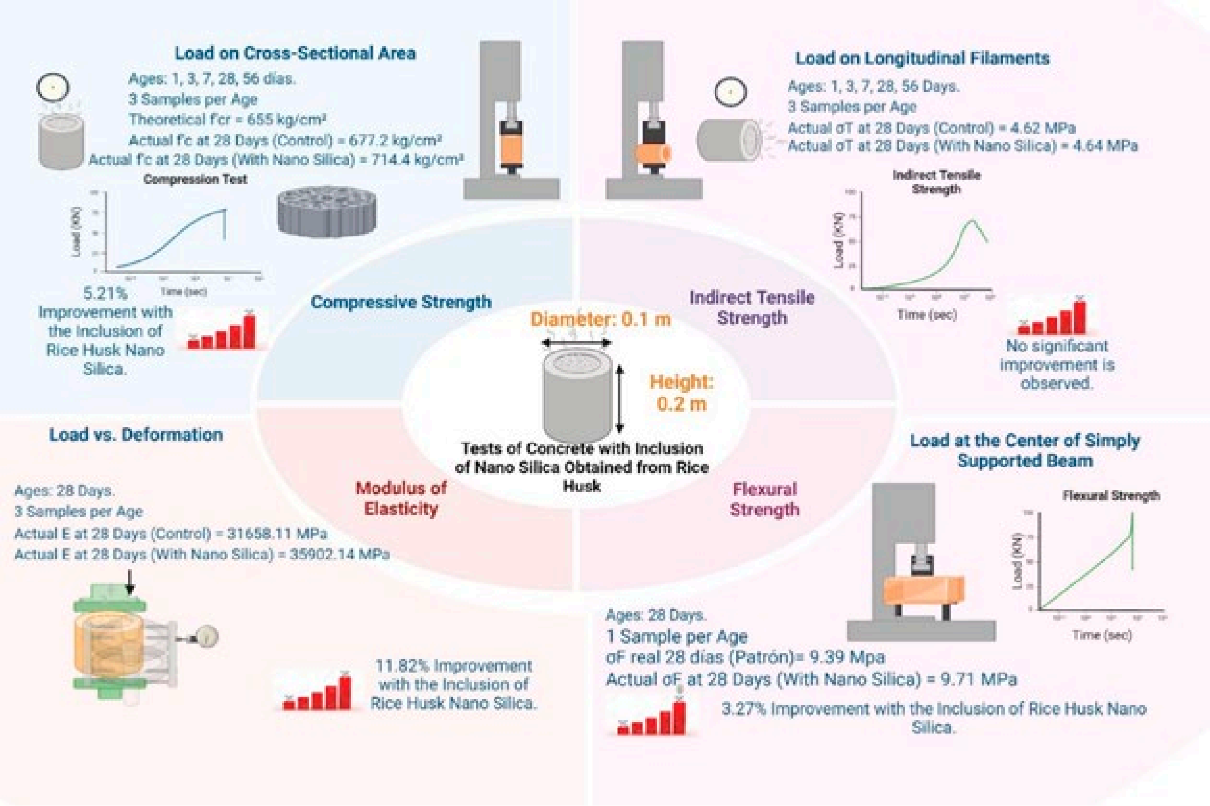

Figure 13. Results and Testing Process of Control Concrete Cylinders vs. Concrete with Inclusion of 1.5% Rice Husk Nano Silica

In addition to mechanical improvements, the introduction of functionalized nano-silica particles enhances the hydrophobic behavior of mortar, thus improving its resistance to water ingress and extending its service life (Alvansazyazdi et al., 2023).

The utilization of agro-industrial waste, such as sugarcane bagasse, for nano-silica production contributes significantly to environmental protection strategies by reducing waste and lowering the carbon footprint of the construction industry (Morales et al., 2020).

The integration of innovative methodologies, as seen with the application of information and communication technologies (ICTs) in educational settings, parallels the transformative impact of nanomaterials in enhancing the sustainability and efficiency of construction practices (Salgado et al., 2019).

3.7. Comparison of Unit Prices for Control Concrete, Concrete with 1.5% Rice Husk Nano Silica, and Concrete with Aerosil 200 Nano Silica

With the analyses established in this thesis, a comparison of unit costs is made for the following: control concrete, concrete with 1.5% Aerosil 200 nano silica, and concrete with 1.5% rice husk nano silica.

Figure 14 illustrates that incorporating nano-silica into concrete mixes increases the overall cost compared to unmodified control concrete. However, nano-silica derived from rice husk offers a more cost-effective alternative to the commercial product "Aerosil 200," making it a viable option from a cost-benefit perspective. While concrete incorporating rice husk nano-silica remains more expensive than standard mixes, it provides a balanced solution—offering enhanced material properties at a moderate cost increase, thus maintaining economic competitiveness relative to commercial nano-silica.

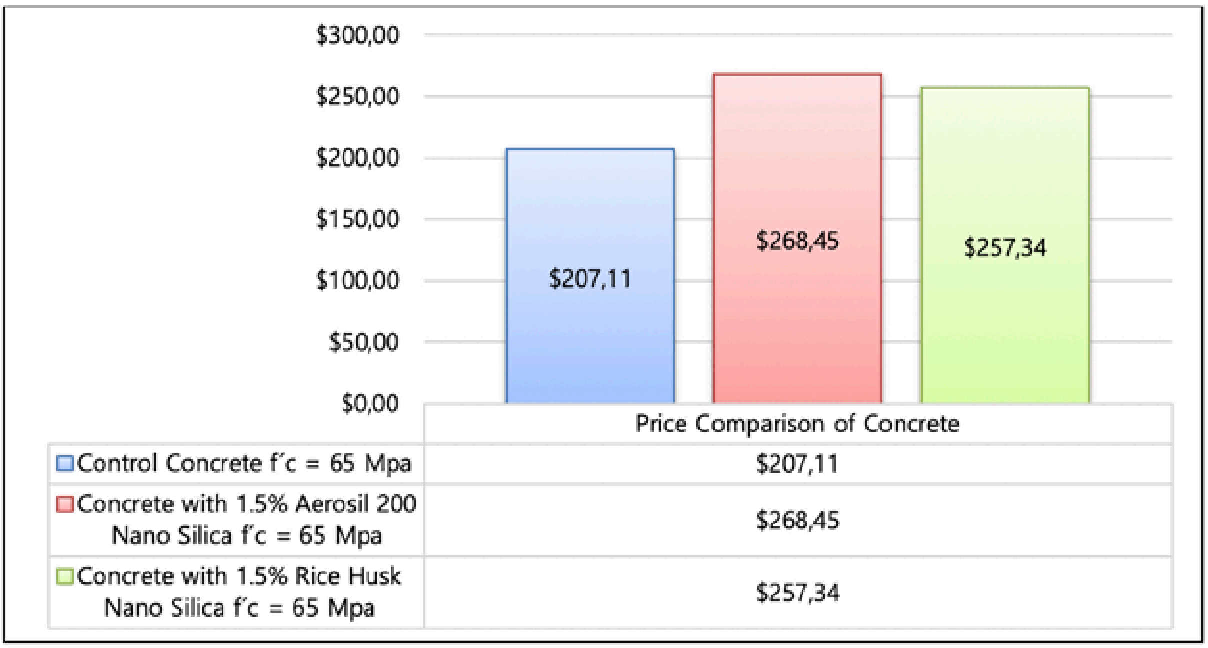

Figure 14. Comparative Analysis of Unit Prices per m³ of Concrete

5. CONCLUSIONS

The chemical process used to extract nano-silica from rice husk resulted in the formation of two primary compounds: SiO_2 and cristobalite. These compounds exhibit distinct morphological characteristics, indicating the presence of different phases or structural forms of silica. Cristobalite, typically formed at elevated temperatures, appears as large, laminar particles, whereas the SiO_2 particles are smaller, angular, and exhibit a rough surface texture —suggesting a less crystalline or more amorphous form of silica. The consistency observed in the density and absorption values of the aggregates reflects uniform material properties, which are critical for ensuring the quality, strength, and long-term durability of the concrete. Moreover, the use of Selvalegre Type HE cement —characterized by its high early strength— has proven effective for structural applications that demand rapid strength gain. Its density plays a key role in the mix design, directly influencing the water-cement ratio and contributing to the achievement of the desired final compressive strength.The absolute volume values play a crucial role in the concrete mix design, ensuring that the desired properties are attained in terms of both strength and workability. In this context, to determine the optimal dosage of the control concrete, several mixes were conducted, ultimately selecting the superplasticizer Viscocrete 4100. This additive met the established requirements, allowing for a homogeneous mix without segregation and adequate workability, as well as achieving the required compressive strength of 55 MPa at 28 days.

On the other hand, although Aerosil 200 nano silica showed lower initial strength compared to other additives, its long-term strength increase is considerable, suggesting its suitability for applications where resistance at later ages is essential.

X-ray Diffraction (XRD) analysis confirmed the positive impact of incorporating 1.5% nano-silica derived from rice husk, revealing notable improvements in the concrete's microstructure. Specifically, an increase in the formation of calcium silicate hydrate (C-S-H) gel and a reduction in porosity were observed. These microstructural enhancements contribute significantly to the improvement of both the mechanical performance and durability of the concrete.The scanning electron microscopy (SEM) tests revealed that the incorporation of 1.5% rice husk nano silica significantly improves the microstructure compared to the control concrete, showing greater density, reduced porosity, and a more homogeneous particle distribution. Additionally, the analysis using energy dispersive spectroscopy (EDS) indicated a significant increase in silicon content, suggesting greater formation of hydration products, such as C-S-H gel, thereby contributing to the enhancement of the mechanical properties and durability of the concrete.

This set of findings suggests that the inclusion of rice husk nano silica can be a promising strategy for improving the quality and sustainability of cementitious materials.

ACKNOWLEDGMENTS

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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7. APPENDICES

Ministerio
del Interior

MINISTERIO DEL INTERIOR

DIRECCIÓN DE CONTROL DE SUSTANCIAS CATALOGADAS SUJETAS A FISCALIZACIÓN

Permiso No. **P17-006850-1**
Fecha: 30/05/2023
Recibo No. 4384
Form. No. **2**

La Dirección de Control de Sustancias Catalogadas autoriza a:

GUALAN MILLINGALLE ANGEL STALIN

Con dirección : CALLE EL RANCHO, CALLE 11, LOTE 935
Con Céd / RUC: 1717376824
La compra de:

Sustancia	Cantidad	Unid.	Uso
ACIDO CLORHIDRICO	10.00	kg	LAVADO DE CENIZAS
ACIDO SULFURICO	10.00	kg	LAVADO DE CENIZAS

A la empresa: QUIMICOS EXPORTACIONES E IMPORTACIONES RELUBQUIM CIA. LTDA.
Calificación: 17-0474-I **Código:** 1074

comprometiéndose legalmente a controlar, mediante un registro de ingresos y egresos, la utilización de estas sustancias para los fines constantes en la solicitud y autorización.

Autorizado por

FANNY LUCIA
GALLEGOS FIERRO

Dra. Fanny Gallegos
Delegada de Control y Administración de S.C.S.F.- Coordinació

NOTA: Este documento caducará en el término de 8 días después de la fecha de emisión, el mismo que le facilitará adquirir por UNA SOLA VEZ y cuyo original retendrá la empresa comercializadora como documento de descargo.

Appendix 1. Certificate of Use of Regulated Substances Used in the Synthesis of Rice Husk Nano Silica