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REVISTA INGENIO

Valorization of Sugarcane Bagasse into Nano-Silica: Optimized Route for Enhancing Strength and Sustainability in Cement Mortars

Valorización del Bagazo de Caña de Azúcar en Nanosílice: Ruta Optimizada para Mejorar la Resistencia y la Sostenibilidad en Morteros de Cemento

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Nanomateriales, Partículas de nano-sílice, Morteros de cemento, Propiedades mecánicas, Hidrofobicidad del mortero

RESUMEN
La presente investigación tiene como objetivo evaluar la influencia de la adición de nanopartículas de sílice derivadas del bagazo de caña de azúcar, sintetizadas en el laboratorio mediante el método sol-gel, sobre las propiedades del mortero de cemento en estado fresco y endurecido. En el estado fresco, se analizan la trabajabilidad, la consistencia y la fluidez, mientras que en el estado endurecido se evalúan la resistencia a la compresión y la hidrofobicidad. Se prepararon especímenes utilizando cementos Tipo N y Tipo HS, con la adición de nanopartículas de sílice en proporciones de 0,25 %, 0,50 %, 0,75 %, 1,00 % y 1,50 % en peso, como reemplazo del cemento en el mortero de control. Los resultados revelaron que el 0,25 % de nano-sílice fue el porcentaje de adición óptimo para ambos tipos de mortero. Además, se observó que tanto las propiedades en estado fresco como en estado endurecido se vieron afectadas negativamente a medida que aumentaba el porcentaje de nano-sílice. La resistencia a la compresión aumentó en un 9 % a los 28 días y en un 12 % a los 56 días para el mortero con cemento Tipo N; mientras que, para el mortero con cemento Tipo HS, el incremento fue del 9 % a los 28 días, 10 % a los 56 días y 5 % a los 90 días. La prueba del ángulo de contacto indicó que las nanopartículas de sílice reducen la permeabilidad de los morteros, siendo los especímenes elaborados con cemento Tipo N los que presentaron una mayor impermeabilidad en comparación con aquellos elaborados con cemento Tipo HS.

KEY WORDS
Nanomaterials, Nano-silica particles, Cement mortars, Mechanical properties, Mortar hydrophobicity

ABSTRACT
The present research aims to evaluate the influence of adding silica nanoparticles derived from sugarcane bagasse, synthesized in the laboratory using the sol-gel method, on the properties of cement mortar in both its fresh and hardened states. In the fresh state, the study examines workability, consistency, and flow, while in the hardened state, it evaluates compressive strength and hydrophobicity.
Specimens were prepared using Type N and Type HS cement, with the addition of nano-silica particles at 0.25%, 0.50%, 0.75%, 1.00%, and 1.50% by weight as a replacement for cement in the control mortar. The results revealed that 0.25% nano-silica was the optimal addition percentage for both mortars. Moreover, it was found that both fresh and hardened properties were negatively affected as the percentage of nano-silica increased. The compressive strength increased by 9% at 28 days and 12% at 56 days for the Type N cement mortar, while for the Type HS cement mortar, the strength increase was 9% at 28 days, 10% at 56 days, and 5% at 90 days. The contact angle test indicated that nano-silica particles reduce the permeability of the mortars, with specimens made with Type N cement exhibiting greater impermeability compared to those made with Type HS cement.

1. Introduction

Construction is one of the industries that generates the most carbon footprint in the world, as the materials it relies on are mainly cement-based, which requires a considerable amount of energy and the exploitation of non-renewable resources for its production [1]. According to the UN, the construction sector is responsible for more than 34% of energy demand and around 37% of CO2 emissions during 2021 [2].

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In recent years, there has been a proposal to reduce cement use in construction by replacing it with industrial, agricultural, and other byproducts, one of which is sugarcane bagasse.

Sugarcane is produced worldwide, and according to the FAO, global sugar consumption is expected to increase over the next 10 years, with sugarcane continuing to account for more than 85% of sugar crop production [3]. Once the raw material is extracted from this plant, a residue is generated, the sugarcane bagasse [4].

Nanotechnology has been researched and introduced in the construction industry, specifically in the production of concrete and mortars, aiming to improve the physical and mechanical performance of cements added with certain types of nanometric particles such as silica dioxide, graphene, iron oxide, zinc oxide, carbon nanotubes, titanium dioxide, among others [5], [6], [7], [8], [9]. These have resulted in higher strength, greater durability, and more contributions to sustainable construction [10].

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 [10].

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 [11].

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 [12].

A sustainable material with a wide range of applications is silica dioxide ($SiO2$), which occurs naturally as quartz and can be extracted from sand and other minerals. Another source of $SiO2$ is plants, and therefore, this compound can be extracted from agro-industrial waste, such as sugarcane bagasse [13]. According to the Ibero-American Agency for the Diffusion of Science and Technology (DICYT), the chemical composition of sugarcane bagasse ash is dominated by silica oxide, with alumina and iron oxide content, which can react with calcium hydroxide in cement hydration and produce materials that improve the mechanical and durability properties of concrete [14].

Nanosilica consists of nanometric-sized particles of $SiO2$, which possess pozzolanic properties that react with portlandite and improve its properties [15]. Due to its size, it can group together in the small pores of the cement paste, closing them or reducing their size, decreasing permeability by making the mortar denser and enhancing its mechanical properties [17]. The incorporation of this addition results in a reduction in cement requirements, while increasing the demand for mixing water due to the high specific surface area of the nanomaterial [16].

Studies on the incorporation of nanosilica in construction materials support the aforementioned benefits of this material on the properties of concrete [17], [18], [19], [20], [21], [22], paving stones [23], and mortars [24], [25], [26]. This improvement in properties positions nanosilica as an advanced solution for more durable and sustainable construction [29].

Given the above, this study aims to analyze the modification of the properties of mortars in both the fresh and hardened states, with additions of $SiO2$ nanoparticles derived from sugarcane bagasse in percentages of: 0.25%, 0.50%, 0.75%, 1.00%, and 1.50% by weight as a partial replacement for cement, as a positive factor in reducing the environmental impact generated by agro-industrial waste and the construction industry [30].

2. Materials y Methods

2.1 MATERIALS

2.1.1. Cement

Two types of cement were used: Maestro by Holcim (Type N cement) and Campeón by Selvalegre (Type HS cement).

2.1.2. Fine Aggregate

The fine aggregate was sourced from Santo Domingo, with a density of 2.69 g/cm³ and an absorption capacity of 1.7%. Its granulometry meets the grading limits for natural sand established in INEN 2536 [27].

Additionally, the organic impurities in the fine aggregate were assessed, revealing a low impurity content. The quantity of lightweight and friable particles was within the limits set by NTE INEN 699 and 698, respectively.

2.1.3. Nanosílice derived from sugarcane bagasse

The silica nanoparticles were obtained in the laboratory from sugarcane bagasse waste through a multi-step extraction process that includes: 1. Conditioning of the material: drying and grinding of sugarcane bagasse, 2. Calcination of the material: pyrolysis using a Bunsen burner and transformation into ashes using a muffle furnace, 3. Acid treatment of the ashes: reflux in HCl and HNO_3 acids, 4. Purification of the ashes: reflux in NaOH, and 5. Precipitation of the product: application of the sol-gel method to obtain silica particles [13], [28]. Figure 1 indicates the process carried out to obtain silica nanoparticles from sugarcane bagasse.

The mentioned procedure was performed four times (four cycles) to obtain the desired amount.

Table 1 shows the quantities of each material used per cycle, as well as the final amounts.

Table 1

Quantities of each material used per cycle

Cycle	*Bagasse (kg)*	*Ashes (g)*	*HCl (ml)*	*HNO_3 (ml)*	*NaOH (ml)*
1	0.33	14.9	59.8	50.0	50.0
2	0.65	10.1	60.5	50.0	100.0
3	34.97	427.6	855.1	420.0	696.6
4	44.56	203.8	507.6	210.0	600.0
Total	80.51	656.4	1483.0	730.0	1446.6

Table 2 presents the quantities of nano-silica obtained from sugarcane bagasse in each cycle, as well as the yield per cycle and the overall yield of this material.

Table 2

Quantities of nanosilica obtained from sugarcane bagasse per cycle and its yield

Cycle	*Nanosilica obtained (g)*	*Yield (%)*
1	1.8	0.542
2	3.6	0.556
3	208.6	0.596
4	254.4	0.571
Total	468.4	0.566

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 algorithms [29]. The characterization of the nano-silica particles obtained from sugarcane bagasse was performed using the following laboratory techniques:

2.1.3.1. Scanning Electron Microscopy (SEM)

Figure 2 shows a surface with a rough and heterogeneous texture, composed of particles varying in size and shape. The distribution of shades suggests the presence of elevated areas (brighter) and deeper regions (darker). The highly agglomerated structure indicates a material with a high specific surface area, which is characteristic of nanoparticles.

Figure 2

SEM test conducted by the Microscopy Laboratory, ESPE, on the nanosilica sample obtained with a 1µm scale.

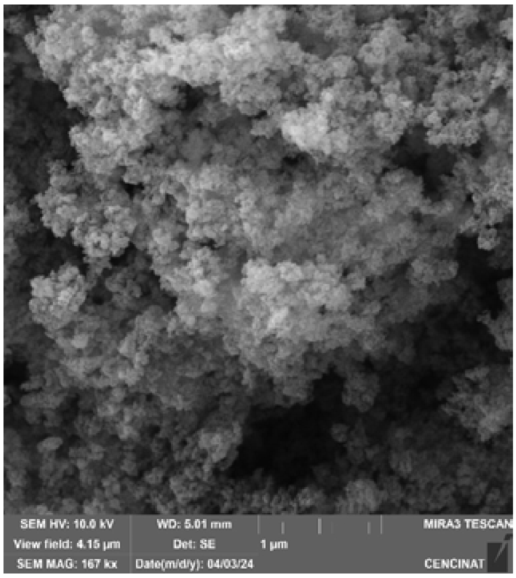
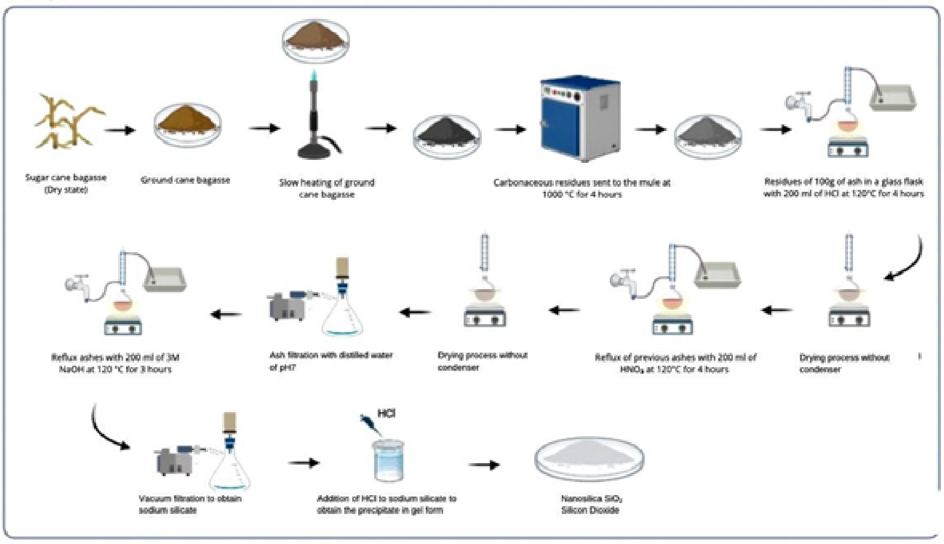


Figure 1

Laboratory process for obtaining nanosilica derived from sugarcane bagasse

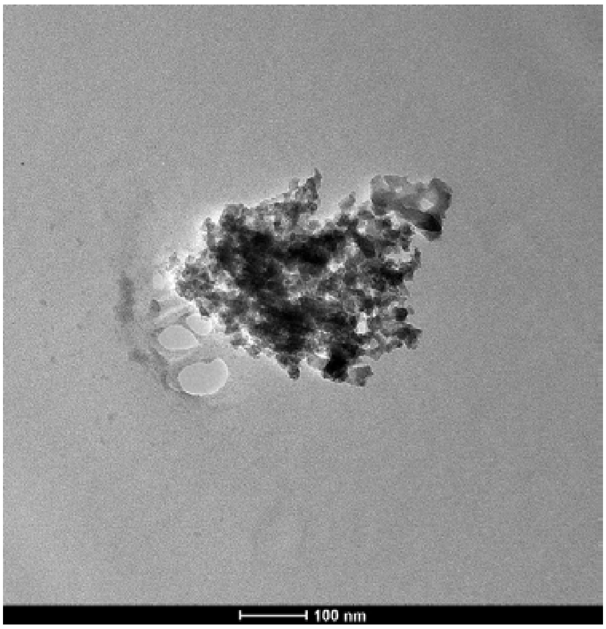


2.1.3.2. Transmission Electron Microscopy (TEM)

Figure 3 shows that the SiO_2 nanoparticles tend to agglomerate, possibly due to attractions generated by the differences in electronegativity between O and Si atoms. The 100 nm scale allows visualization of particles smaller than 100 nm, confirming that this material qualifies as a nanomaterial.

Figure 3

TEM test conducted by the Microscopy Laboratory, ESPE, on the nanosilica sample obtained with a 100 µm scale.

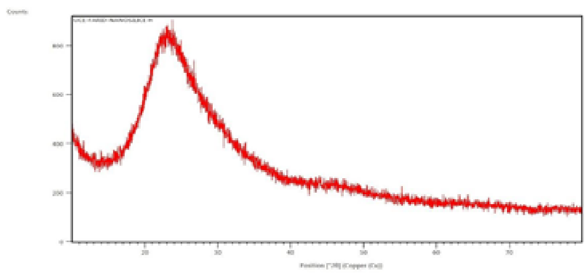


2.1.3.3. X-ray Diffraction (XRD)

Narrower peaks indicate larger crystals, while wider peaks may indicate smaller crystal sizes. However, it maintains the characteristic pattern of nano-silica proposed by Puerto Suárez. [17]

Figure 4

XRD analysis performed on the nanosilica sample.



2.1.3.4. Energy Dispersive Spectroscopy (EDS)

The elements Si and O from SiO2 were identified in the sample. The percentages obtained for each element are close to the ratio that represents SiO2, with 2 parts of O for every part of Si (66.10% for O and 25.37% for Si).

Tabla 3

Components and percentages present in the sample.

Element	*Amount present in the sample.*
Oxygen (O)	66.10 %
Sodium (Na)	6.42 %
Aluminium (Al)	1.24 %
Silicon (Si)	25.37 %
Iron (Fe)	0.88 %
TOTAL	100 %

2.2 METHODS

2.2.1. Mixture Proportioning

The proportioning used was selected based on NTE INEN 1806 [30], which specifies the commonly used cement: sand ratio in construction of 1:3. The amount of water was selected based on mortar flow tests.

Two types of mortar were prepared: with type N cement and type HS cement. The proportions are shown in Table 4.

Table 4

Quantities for 9 specimens of standard mortar.

Material	Weight (g)	Ratio
Type N Cement		
Cement	740.0	1
Sand	2220.0	3
Water	437.3	0.54
Type HS Cement		
Cement	740.0	1
Sand	2220.0	3
Water	429.9	0.53

Once the weight quantities of each material for the reference mortar are determined, the weight of the cement is replaced with each percentage of nanosilica that is to be added to the mixture (see Table 5).

Table 5

Quantities for 9 specimens of mortar with the addition of nanosilica particles.

Material	Quantity in weight of the materials (g)				
	0.25%	0.50%	0.75%	1.00%	1.50%
Type N Cement					
Cement	738.1	736.3	734.4	732.6	728.9
Sand	2220	2220	2220	2220	2220
Water	437.3	437.3	437.3	437.3	437.3
Nanosilica	1.85	3.70	5.55	7.40	11.10
Type HS Cement					
Cement	738.1	736.3	734.4	732.6	728.9
Sand	2220	2220	2220	2220	2220
Water	437.3	437.3	437.3	437.3	437.3
Nanosilica	1.85	3.70	5.55	7.40	11.10

2.2.2. Mixing procedure

The nanosilica particles must undergo a predispersal to obtain stable suspensions before being integrated into the mortar mixture, as their high specific surface area can cause particle agglomeration that hinders the proper combination of the mortar materials [18]. To prepare the nanosilica suspension, a high-shear mixer was required. A portion of the mixing water, along with the nanosilica, was mixed for 120 seconds at 1000 rpm.

To prepare the mortar mixtures, the water and the nanosilica suspension were placed in the mixer, the cement was added and mixing continued for 3 minutes at low speed. The fine aggregate in a dry condition was then added to the mixer, mixing for 30 seconds at low speed, then for another 30 seconds at medium speed. The process was paused, and the mixture was allowed to rest for 90 seconds. After this time, the mixture was mixed again for an additional 60 seconds.

2.2.3. Optimal Nanosilica Addition Percentage

Immediately after mixing, the flowability of the fresh mixtures was measured according to NTE INEN 2502 [31], which specifies a flow of 110 ± 5 in 25 drops on the flow table.

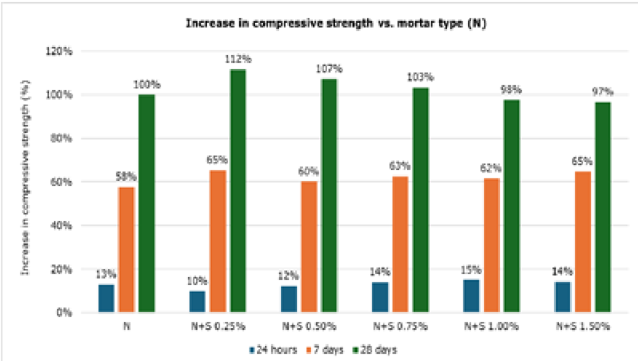
The results of the fresh-state mortar tests, Table 4, show that with the incorporation of nanosilica particles into the mortar, there is a loss of workability as the percentage of nanosilica added increases. In other words, the higher the amount of nanosilica, the lower the flowability of the mixture. The mixtures with 0.25% and 0.50% nanosilica with type N and type HS cement exhibited medium workability and were found to be easy to handle and apply. However, starting from 0.75% addition, it was observed that the consistency of the mixtures shifted from plastic to dry over time. As the percentage of addition increased, this decreased the workability of the mixtures, making them more difficult to handle.

The analysis of the fresh-state properties indicates that the mixtures with both types of cement are negatively affected by the increase in nanosilica quantity added. However, the mixtures with 0.25% nanosilica addition presented the best results in terms of workability, consistency, and flowability.

Subsequently, a compressive strength test was performed according to NTE INEN 488 [32] on the hardened mortar specimens. Nine specimens were made for both the reference mixture and the mixtures with nanosilica addition using the two types of cement (Total 108 specimens). The tests were carried out at 1, 3, and 28 days for type N cement and 1, 3, and 7 days for type HS cement (The difference in testing ages is due to the strength each type of cement can achieve).

Graph 1

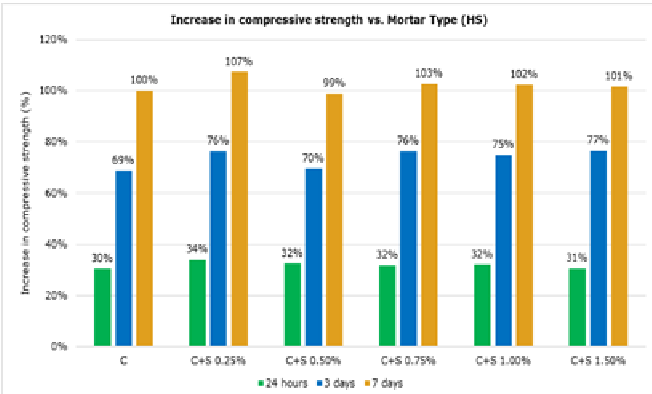
Incremento de la resistencia a la compresión (%) vs Tipo de mortero, cemento tipo N



Graph 1 shows the compressive strength as a percentage for the different mixtures, taking the compressive strength of the reference mortar at 28 days as 100%. It can be observed that the greatest increase in strength for the mortar with type N cement is achieved with a 0.25% addition of nanosilica, reaching 12% more strength compared to the reference mixture.

Graph 2

Incremento de la resistencia a la compresión (%) vs Tipo de mortero, cemento tipo HS



Graph 2 shows the compressive strength as a percentage for the different mixtures with type HS cement, taking the compressive strength of the reference mortar at 7 days as 100%. It can be observed that the greatest increase in strength for the mortar is achieved with a 0.25% addition of nanosilica, reaching 7% more strength compared to the reference mixture.

The analysis of the hardened-state specimens with the two types of cement used indicates that the addition of nanosilica improves compressive strength. However, it is observed that high percentages do not seem to generate significant improvements in strength and may even negatively affect it, suggesting that there is a limit to the amount of nanosilica that can be added. It is observed in both types of cement that the 0.25% nanosilica addition percentage is the one that allows for the highest strength in mortars.

Therefore, a 0.25% addition of nanosilica is established as the optimal percentage, and the preparation of the final specimens is carried out to obtain better statistics and confirm the results for comparison with the reference sample.

3. Results

3.1 MECHANICAL PROPERTIES

A total of 10 mortar specimens were prepared for each mixture type, i.e., reference mixture and mixture with 0.25% nanosilica addition for each testing age: 1, 3, 7, 28, 56, and 90 days, and each type of cement used (Total 240 specimens). Table 5 presents the arithmetic mean value of the compressive strength results for the reference mixture specimens and the mixture with 0.25% nanosilica addition from sugarcane bagasse.

Type N Cement

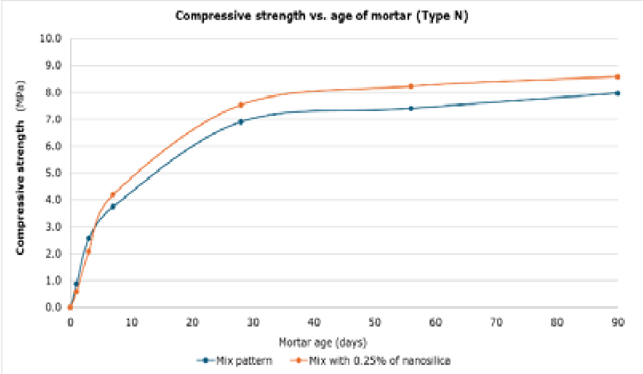
Table 7 shows that, at 24 hours, the mixture with 0.25% nanosilica exhibits a decrease of 0.6 MPa in strength compared to the reference mixture, which had a strength of 0.9 MPa. The same behavior was observed up to 3 days, with the reference mortar reaching 2.6 MPa and the mortar with 0.25% nanosilica reaching 2.1 MPa. This suggests that the initial decrease in the mortar's strength is attributed to the addition of nanosilica.

Starting from 7 days, the mortar with nanosilica addition shows higher strength than the reference mortar. The reference mortar achieved 3.7 MPa, while the mortar with nanosilica reached 4.2 MPa. At 28, 56, and 90 days, the trend of increased strength continued for the mixture with nanosilica addition, indicating that, in the long term, the addition of nanosilica provides positive benefits in terms of strength.

This behavior can be observed in Graph 3, which shows that the highest strengths over time were achieved by the mortar with 0.25% nanosilica.

Graph 3

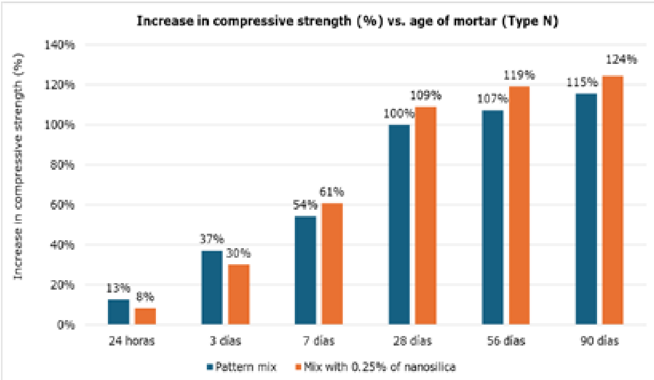
Compressive strength vs. age of mortar (Type N)



In Graph 4, the percentage increase in compressive strength between the reference mortar and the mortar with 0.25% nanosilica addition is shown. A strength increase of 9% was achieved at 28 days, 12% at 56 days, and 9% at 90 days.

Graph 4

Increase in compressive strength (%) vs. age of mortar (Type N)



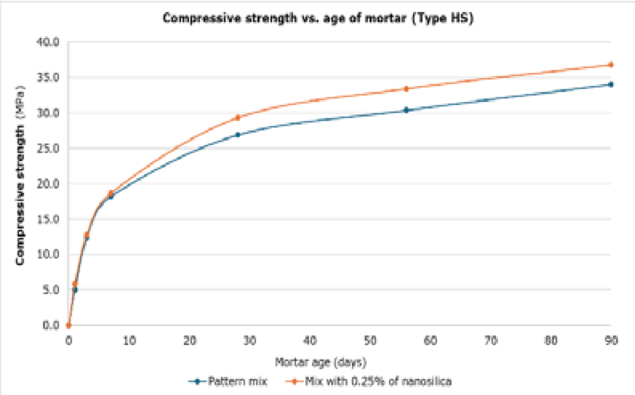
Type HS Cement

Graph 5 shows the increase in compressive strength of the mortar with nanosilica addition starting from the first day, where a strength of 5.9 MPa is observed compared to the 5.0 MPa reached by the reference mortar. This growth trend continues until the 90-day age, where the mortar with 0.25% nanosilica reaches a strength of 36.8 MPa, compared to the 34.0 MPa of the reference mortar.

This behavior can be observed in Graph 5, which shows that the highest strengths over time were achieved by the mortar with 0.25% nanosilica.

Graph 5

Compressive strength vs. age of mortar (Type HS)



In Graph 6, the percentage increase in compressive strength between the reference mortar and the mortar with 0.25% nanosilica addition is shown. A strength increase of 9% was achieved at 28 days, 10% at 56 days, and 10% at 90 days.

The results presented demonstrate the benefits of nanosilica addition in this study, with 0.25% sugarcane bagasse nanosilica, in increasing the compressive strength of mortars with type N cement and mortars with type HS cement.

After performing the compressive strength tests, a contact angle test was conducted to determine the hydrophobicity of the specimens. It was decided that the test would be carried out at 28, 56, and 90 days for both the reference mortar specimens and those with sugarcane bagasse nanosilica addition, as the mortar reaches its maximum strength at this age.

To perform the contact angle test, the surface of the specimens was cleaned to remove any dust, impurities, or detachment. A drop of water was then placed on the smoothest and least porous surface of the specimen using a micro needle, which allowed a small drop to be placed. Using a laboratory microscope, the behavior of the water drop on the mortar surface was observed (whether it is absorbed or not). Images were captured with a smartphone camera, and through the ImageJ software, the angle formed between the water drop and the mortar surface was measured.

Angles less than 90° indicate that the material is hydrophilic (more permeable/more absorbent), and angles greater than 90° indicate that the material is hydrophobic (less permeable/less absorbent).

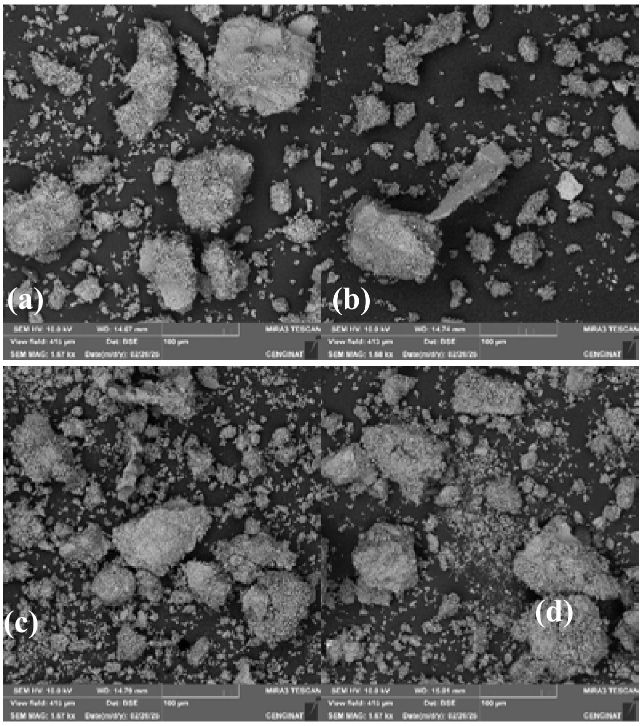
It can be seen in Table 8 that the mixtures with nanosilica addition for both types of cement used have higher values compared to the reference mixtures. This means that the addition of nanosilica reduces the mortar's absorption capacity, making it a more hydrophobic or less permeable material. This contributes to the durability of the mortar, as it helps prevent or reduce damage that may occur due to freeze-thaw cycles, as well as issues like efflorescence, internal moisture, presence of microorganisms, etc.

3.2 MICROSTRUCTURE OF MIXTURES WITH NANOSILICA

The SEM images of mortars with both Type N and Type HS cement and 0.25% nanosilica reveal a more uniform and dense structure, with smaller and better-distributed particles. At 56 and 90 days, a decrease in porosity is observed, indicating greater compaction of the mortar matrix.

Figure 5

SEM analysis of mortar mix with type N cement (a) and 0.25% nanosilica, at 28(b), 56(c) and 90 days(d).



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 [26].

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 [33].

The smaller pore size and the increase in hydration products, such as C-S-H (calcium silicate hydrate), contribute to improving the durability and strength of the mortar.

The incorporation of nanosilica significantly enhances the microstructure, reducing porosity and promoting the formation of cementitious products, which translates into higher mechanical strength and durability.

4. Conclusions

- The extraction of silica nanoparticles using the sol-gel method was carried out in four cycles, yielding a total production of 468.4 grams of nanosilica from 80.5 kg of sugarcane bagasse waste sourced from the Puyo canton, demonstrating a yield of 0.566%.
- The characterization of the nanosilica obtained from sugarcane bagasse reveals several structural and chemical properties that confirm it is a material with typical SiO_2 nanoparticle characteristics. The SEM test revealed a rough and heterogeneous surface with an agglomerated structure, indicating a high specific surface area, which is characteristic of nanoparticles. Meanwhile, the TEM test showed that the particle size is below 100 nm, confirming it as a nanomaterial. The XRD image indicated that the diffraction pattern matches the nanosilica structure reported in the literature, suggesting a crystalline phase consistent with the characteristics of a nanostructured material. Finally, the EDS analysis confirmed the presence of Si and O in proportions close to the stoichiometry of SiO_2, with 66.10% oxygen and 25.37% silicon.
- The fresh and hardened state properties of the reference mortar and mortar with nanosilica addition at different percentages, using two types of cement, determined that the optimal nanosilica addition percentage is 0.25%. This percentage does not affect the fluidity, workability, or consistency of the fresh-state mortar, allowing for easy handling and placement. In the hardened state, this percentage achieved the highest compressive strength compared to the reference mortar.
- It was observed that high nanosilica percentages can negatively affect fresh-state mortar mixtures with both Type N and Type HS cement, causing the mixtures to transition from a plastic state to a dry state (lower workability, dry consistency, and difficult handling).
- The contact angle test showed that the tested specimens, including conventional mortar with Type N and HS cement, exhibited a hydrophilic surface, except for the mortar made with Type N cement and 0.25% nanosilica addition, which presented a hydrophobic surface at 90 days of age. Additionally, the incorporation of nanosilica increased the contact angle in these specimens, confirming that nanoparticles reduce water absorption by filling the voids in the mortar.
- The incorporation of nanosilica synthesized from sugarcane bagasse in mortars not only contributes to the sustainable management of agricultural waste but also enhances construction efficiency and sustainability. By reducing cement dependency, CO_2 emissions are decreased, helping to mitigate the environmental impact of the construction industry. Moreover, the improvement in mechanical properties optimizes material usage, leading to lower resource consumption.

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Annexes

Table 6

Properties of mortars in fresh state

Mix Type	Fluidity	
	Type N Cement	Type HS Cement
M control	113	112
M with 0.25% nanosilica	111	109
M with 0.50% nanosilica	110	105
M with 0.75% nanosilica	110	99
M with 1.00% nanosilica	100	96
M with 1.50% nanosilica	97	92

Table 7

Strength values of mortars at different ages

Mix	Compressive strength (MPa)					
	1 day	3 days	7 days	28 days	56 days	90 days
Type N Cement						
M control	0.9	2.6	3.7	6.9	7.4	8.0
M with 0.25% nanosilica	0.6	2.1	4.2	7.5	8.2	8.6
Type HS Cement						
M control	5.0	12.4	18.2	26.9	30.4	34.0
M with 0.25% nanosilica	5.9	12.8	18.7	29.3	33.4	36.8

Table 8

Hydrophobicity of mortars, contact angle test

Mortar	*Age (days)*		
	28	*56*	*90*
Type N Cement			
M control	62.8°	79.1°	84.2°
M with 0.25% nanosilica	63.4°	80.1°	98.2°
Type HS Cement			
M control	43.3°	47.6°	59.9°
M with 0.25% nanosilica	48.1°	51.2°	78.4°

Graph 6

Increase in compressive strength (%) vs. age of mortar (Type HS)

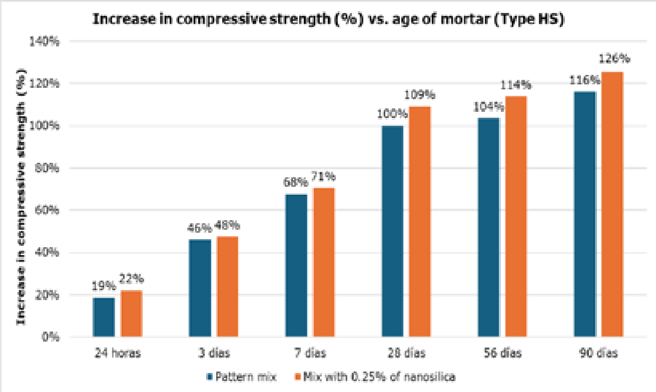

Figure 6

SEM analysis of mortar mix with HS type cement (a) and 0.25% nanosilica, at 28(b), 56(c) and 90 days(d).