

Article

Corn Leaf-Derived Nanosilica as a Sustainable Supplementary Material for Mortars: Synthesis, Characterization, and Mechanical Performance

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Abstract

Cement has long been considered the essential binder of modern construction, but the price paid for this convenience is high. The production of ordinary Portland cement is estimated to contribute close to 8% of global CO₂ emissions, which explains why so many research efforts now concentrate on reducing its impact. One of the directions that has attracted interest is the use of agricultural residues, materials that are usually discarded after harvest and that, in many cases, contain significant amounts of amorphous silica. In the present study, corn leaves (*Zea mays L.*) were selected as a raw material. This residue is abundant in Ecuador, yet it rarely finds any technical application. The leaves were first calcined at 600 °C, then washed with acid and ground until nanosilica with particle sizes in the nanometer range was obtained. Mortars were prepared in which cement was partially replaced by 0.25%, 0.5%, and 1.0% of this nanosilica. After testing compressive strength at 1, 3, 7, 28, and 90 days, the trend became clear: the lowest content, 0.25%, delivered the best performance, reaching an increase of 27% at 90 days compared with the control mix. Higher contents did not lead to further improvement, which may be related to particle agglomeration and limited dispersion. Microstructural analyses (SEM, TEM, XRD, EDS) confirmed the presence of a denser and more homogeneous matrix and contact angle measurements suggested reduced water uptake. These results show that nanosilica obtained from corn leaves can work as a sustainable additive for mortars, while also providing a way to give value to an abundant agricultural residue.

Keywords: *cement; nanosilica; corn leaves; agricultural residues; compressive strength; sustainable mortars.*

Artículo original

Nanosílice derivada de hojas de maíz como material suplementario sostenible para morteros: síntesis, caracterización y rendimiento mecánico

Resumen

El cemento se ha considerado durante mucho tiempo el aglutinante esencial de la construcción moderna, pero el precio que se paga por esta comodidad es alto. Se estima que la producción de cemento Portland ordinario contribuye cerca del 8% de las emisiones globales de CO₂, lo que explica por qué tantos esfuerzos de investigación ahora se concentran en reducir su impacto. Una de las direcciones que ha atraído interés es el uso de residuos agrícolas, materiales que generalmente se desechan después de la cosecha y que, en muchos casos, contienen cantidades significativas de sílice amorfica. En el presente estudio, se seleccionaron hojas de maíz (*Zea mays L.*) como materia prima. Este residuo es abundante en Ecuador, pero rara vez encuentra alguna aplicación técnica. Las hojas se calcinaron primero a 600 °C, luego se lavaron con ácido y se molieron hasta obtener nanosílice con tamaños de partícula en el rango nanométrico. Se prepararon morteros en los que el cemento se reemplazó parcialmente por 0,25%, 0,5% y 1,0% de esta nanosílice. Tras probar la resistencia a la compresión a los 1, 3, 7, 28 y 90 días, la tendencia se hizo evidente: el contenido más bajo, 0,25 %, presentó el mejor rendimiento, alcanzando un aumento del 27 % a los 90 días en comparación con la mezcla de control. Los contenidos más altos no produjeron una mejora adicional, lo que podría estar relacionado con la aglomeración de partículas y la dispersión limitada. Los análisis microestructurales (SEM, TEM, XRD, EDS) confirmaron la presencia de una matriz más densa y homogénea, y las mediciones del ángulo de contacto sugirieron una menor absorción de agua. Estos resultados demuestran que la nanosílice obtenida de las hojas de maíz puede funcionar como un aditivo sostenible para morteros, a la vez que proporciona una forma de valorizar un residuo agrícola abundante.

Palabras Clave: *cemento; nanosílice; hojas de maíz; residuos agrícolas; resistencia a la compresión; morteros sostenibles*

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1. Introduction

Portland cement is part of almost every building we see. It is mixed into mortars for houses, poured into bridges, and spread across pavements. People in construction like it because it is affordable and easy to work with. Still, there is a serious downside. Making cement produces a lot of pollution. Current estimates suggest that the cement industry alone is behind nearly 7% of global CO₂ emissions (Andrew, 2018; Scrivener et al., 2018).

The reason is simple. When limestone is burned to form clinker, it releases CO₂. At the same time, kilns must be kept extremely hot—above 1400 °C—and that burns a lot of fuel (Miller et al., 2016; Habert & Ouellet-Plamondon, 2016). Taken together, these steps explain why cement is one of the most carbon-intensive materials in modern life.

Because of this, scientists and engineers are under pressure to find eco-efficient alternatives. The idea is not to stop using cement completely—society depends on it—but to cut its environmental cost (Imbabi et al., 2012; Du et al., 2019). Some options involve replacing part of the clinker with other materials, often called supplementary cementitious materials (SCMs). Others test ways to recycle waste from industry or agriculture. In recent years, nanomaterials have been added to the list of possible solutions (AlTawaiha et al., 2023; Reddy Babu et al., 2019).

Among them, nanosilica (SiO₂) has become a favorite. Its particles are very small, usually under 100 nm, and amorphous in structure. They also have a very large surface area, which makes them highly reactive (Ren et al., 2020; Geng et al., 2025). Inside a mortar mix, nanosilica acts like a trigger. It speeds up hydration, fills pores, and creates a denser structure (Ranjan et al., 2024; Kaura et al., 2014). The outcome is easy to measure: mortars and concretes get stronger and last longer. Dozens of papers have reported these gains (Althoeey et al., 2023; Venkata et al., 2024).

Some side effects are interesting too. Several studies noticed that nanosilica changes how water interacts with mortar surfaces, making them less absorbent (Liu et al., 2021; Zhang et al., 2021). Others found that it helps resist chemical attack and freeze-thaw cycles (Huang et al., 2024; Liu et al., 2022). When combined with certain photocatalytic oxides, it has even been linked to self-cleaning properties (Wang et al., 2022).

There is, however, a problem. Producing nanosilica through commercial methods is expensive. Sol-gel, flame pyrolysis, and sodium silicate precipitation give excellent results but consume a lot of energy and costly chemicals (Tessema, 2023; Saha et al., 2024). For use in large-scale construction, these processes are not practical.

That is why researchers are now turning to biogenic sources of nanosilica. Agricultural residues are generated in huge amounts and often contain high levels of amorphous silica. Most of the time, this biomass is wasted or openly burned. Transforming it into nanosilica not only reduces waste but also creates a cheaper additive (Yaqueen et al., 2025; Prabha et al., 2021).

Rice husk ash is the classic example. Torres-Carrasco et al. (2019) showed that nanosilica from rice husks improved mortar strength by around 40% at 28 days. Tran et al. (2021) confirmed similar results and also found lower permeability. Maheswaran et al. (2023) demonstrated improvements in geopolymeric binders as well.

Sugarcane bagasse ash has been equally promising. Basnet et al. (2022) reported 10–20% strength gains in concrete with nanosilica from bagasse. Yarra et al. (2025) added that it also reduces water absorption, improving its permeability. Bamboo leaves are another case. Lwin et al. (2021) showed that nanosilica obtained from bamboo can enhance both strength and durability.

Compared to those residues, corn is less studied, even though it is one of the most important crops in Latin America. Ecuador alone produces more than 1.3 million tonnes each year (FAOSTAT, 2025). Leaves, husks, and cobs pile up after harvest, and most of them are either wasted or burned, creating pollution. Chemical analyses show that corn leaves can contain over 50% amorphous silica (Sulaiman et al., 2023). Cobs, by contrast, contain much less and often carry impurities (Dahliyanti et al., 2022).

So far, only a few studies have looked at nanosilica from corn. Dahliyanti et al. (2022) tested cobs and found low reactivity. Even so, research is scarce and there is no agreement on optimal dosages or durability.

In our own work, we began with cobs. The results were poor—little silica and a dark, impure ash. In hindsight, this failure was useful. It matched what earlier authors had already suggested (Sulaiman et al., 2023; Dahliyanti et al., 2022) and pushed us to turn our attention to corn leaves. The difference was obvious: higher yields and nanosilica of better quality, white and uniform.

The goal of this study was simple: to synthesize nanosilica from corn leaves and test its effect on standardized mortars. Compressive strength was the main property we measured, since it is central to any cementitious system. To add context, we also examined microstructure (SEM, TEM, XRD, EDS) and ran contact angle tests (Wang et al., 2022).

Finally, it is worth noting what this study does not cover. We did not carry out detailed physicochemical analyses such as FTIR, BET, or TGA (Saha et al., 2024). Others have done that before. Our focus was more practical: to see if nanosilica

from corn leaves could improve mortar performance. This narrower scope gave us a clear research question. More advanced characterization and long-term durability tests will be needed in future work.

2. Materials and Methods

2.1 Cement, aggregates, and water

The mortars in this study were prepared with Ordinary Portland cement, type GU, according to ASTM C150 (American Society for Testing and Materials [ASTM], 2021). This type of cement is widely available in Ecuador and serves as a fair baseline for comparing results.

The sand came from a nearby river. It was washed to remove dirt and organics, then dried in open air. Sieve analysis, following ASTM C136 (ASTM, 2021), confirmed a fineness modulus of about 2.65, which falls within the acceptable range for standardized mortars. The mixing water was drinking water, tested to ensure it complied with ASTM C1602 (ASTM, 2021). This way, we avoided unwanted reactions that might come from salts or impurities.

2.2. Collection and pretreatment of corn residues

Corn is abundant in the inter-Andean valleys of Ecuador. For this work, we collected both leaves and cobs after harvest. Prior studies had already hinted that the silica content varies depending on which part of the plant is used (Sulaiman et al., 2023).

- Once collected, the residues went through three simple steps:
- Washing with tap water to remove soil and dust.
- Drying in an oven at 105 °C for 24 hours.
- Grinding in a small mill, reducing particle size to less than 5 mm.
- In the early stage of the project, we tried cobs as the raw material. The outcome was poor: yields below 0.3% and a dark ash with visible carbon. That failure turned out to be useful, though—it directed our efforts toward corn leaves, which promised better results.

2.3. Synthesis of nanosilica

The method we used was based on approaches already tested with rice husks and sugarcane bagasse (Prabha et al., 2021).

It was a thermo-chemical route with five main stages.

Calcination: dried corn leaves were placed in a muffle furnace at 700, 800, and 900 °C for 4 hours. Quercia and Brouwers (2020) had pointed out that below 600 °C, carbon residue remains, while above 950 °C, crystalline phases like cristobalite may appear, reducing reactivity.

Acid treatment: the ash was washed with hydrochloric acid (37%) for 3 hours at room temperature. This step removed metals like Ca, Fe, and Mg, which could interfere with purity.

Alkaline digestion: the treated ash was dissolved in sodium hydroxide solution (3M) at 110 °C for 3 hours, producing sodium silicate. This process mirrors methods used successfully with rice husk ash and sugarcane bagasse (Prabha et al., 2021).

Neutralization and precipitation: the sodium silicate solution was titrated with hydrochloric acid until reaching neutral pH. A white gel formed during this stage.

Drying: the gel was repeatedly washed with distilled water, filtered, and dried at 110 °C for 24 hours, producing a fine nanosilica powder.

The yields were clear: 1.7% in the laboratory muffle and up to 4.2% in a semi-industrial furnace. These values were in line with results from rice husk nanosilica and bagasse nanosilica (Yaqueen et al., 2025; Basnet et al., 2022).

2.4. Characterization of nanosilica

We did not attempt a full physicochemical characterization. Instead, we focused on basic imaging and diffraction tests to confirm that the product was indeed amorphous nanosilica.

TEM (transmission electron microscopy) showed spherical particles between 20–60 nm, with some clustering.

SEM (scanning electron microscopy) revealed agglomerates of fine particles, consistent with what others have reported about the high surface energy of nanosilica (Liu et al., 2021). XRD (X-ray diffraction) patterns displayed the typical amorphous halo at 20–30° 2θ, along with reduced portlandite peaks in mortars with nanosilica.

EDS (energy-dispersive spectroscopy) confirmed the presence of mainly Si and O (>95%), plus traces of Na, K, and Al.

More advanced techniques such as FTIR, BET, and TGA could have provided additional insights (Saha et al., 2024). But since our aim was practical application in mortars, we kept the characterization limited.

2.5. Mortar mix design

The mixes followed ASTM C270 [49]. The cement-to-sand ratio was 1:2.75 by weight, with a water-to-cement ratio of 0.64. Four series of mortars were prepared (**Table 1**):



Table 1: Quantity of materials and number of specimens per experimental group

No.	Description	Cement (g)	Sand (g)	Water (g)	Nanosilica (g)	No. of Specimens
1	Control mortar (MP)	740.00	2035	473.6	—	32
2	Mortar + 0.25% NS	738.15	2035	473.6	1.85	12
3	Mortar + 0.50% NS	736.30	2035	473.6	3.70	12
4	Mortar + 0.75% NS	734.45	2035	473.6	5.55	12
5	Mortar + 1.00% NS	732.60	2035	473.6	7.40	12

Nanosilica was first dispersed in the mixing water before being added, a method shown to reduce clustering. Mixing was performed in a vertical-shaft mixer following ASTM C305 (ASTM, 2021). Mortar cubes of 50 mm were cast according to ASTM C109 (ASTM, 2021) and cured in water at 23 ± 2 °C until testing ages (1, 3, 7, 28, and 90 days).

2.6. Mechanical and functional tests

Compressive strength was measured at the specified ages using a hydraulic testing machine, following ASTM C109 (ASTM, 2021). Three cubes per mix were tested, and the mean value with standard deviation was reported.

Contact angle measurements were made with the sessile drop method. Tiny drops of distilled water were placed on polished cube surfaces, and the angle was recorded after 5 seconds. Angles above 90° were taken as preliminary evidence of hydrophobic behavior (Wang et al., 2022).

2.7. Statistical analysis

Data were analyzed using one-way ANOVA. Whenever significant differences were found ($p < 0.05$), Tukey's post hoc test was applied to identify which mixes differed. Similar approaches have been recommended in nanosilica research (Yaqueen et al., 2025).

3. Results

3.1. Yield of nanosilica from corn residues

The first step was to check whether cobs or leaves were more suitable for producing nanosilica. The figure 1 highlights the sharp contrast in total silica content obtained from two maize by-products. Corn husk reached only 7.8%, while corn leaves yielded a markedly higher value of 70.4%. This difference clearly demonstrates the superior potential of leaves as a raw material for nanosilica production, compared to the limited contribution of husk.

The difference was obvious from the start. When we processed cobs, the outcome was poor: yields below 0.3% of the dry weight, along with a dark, carbon-rich ash. Leaves,

on the other hand, told a different story. In the laboratory muffle furnace, yields reached around 1.7%. In the semi-industrial furnace, we saw even higher values, close to 4.2%. The product was a white, homogeneous powder, visually like nanosilica obtained from rice husks and sugarcane bagasse (Tessema, 2023; Prabha et al., 2021). Figure 2 illustrates this contrast: cobs generated little and impure silica, while leaves consistently produced more and cleaner nanosilica.

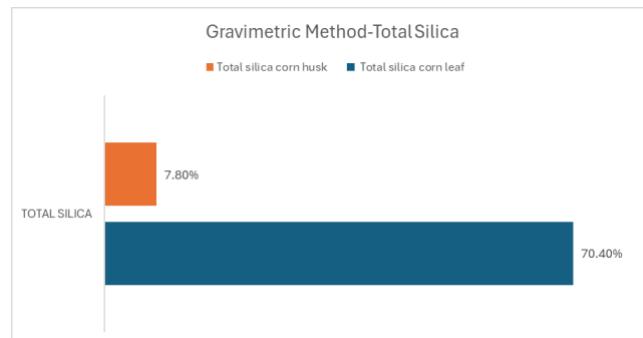


Figure 1: Silicon dioxide content.



Figure 2: Nanosilica produced from corn cob.

In practical terms, these findings confirmed that corn leaves are the better raw material. The negative result with cobs was

not wasted effort—it provided a baseline and reinforced the decision to focus exclusively on leaves.

3.2. Microstructural features of the nanosilica

Transmission electron microscopy (TEM) gave us the first clue about particle size. **Figure 3a** shows that most particles were spherical, between 20 and 60 nm. Some clustering was visible, which is common in nanosilica due to its high surface energy. These values align with what Larkunthod et al. (2022) reported for rice husk nanosilica and Gupta et al. (2021) found with bamboo nanosilica.

Scanning electron microscopy (SEM) (**Figure 3b**) confirmed the tendency of particles to form agglomerates.

This behavior has been described in other studies as well (Sulaiman et al., 2023; Zhang et al., 2021).

X-ray diffraction (XRD) patterns (**Figure 3c**) displayed the typical amorphous halo between 20° and 30° 2 θ . More importantly, mortars containing nanosilica showed a reduction in portlandite peaks, suggesting that the pozzolanic reaction was consuming $\text{Ca}(\text{OH})_2$. Similar evidence was presented in studies on nanosilica from bagasse (Basnet et al., 2022) and rice husks (Ren et al., 2020).

Energy-dispersive spectroscopy (EDS) (**Figure 3d**) confirmed that the nanosilica consisted mainly of Si and O (over 95%), with traces of sodium, potassium, and aluminum. These results fit well with prior reports on silica-rich agricultural ashes (Prabha et al., 2021).

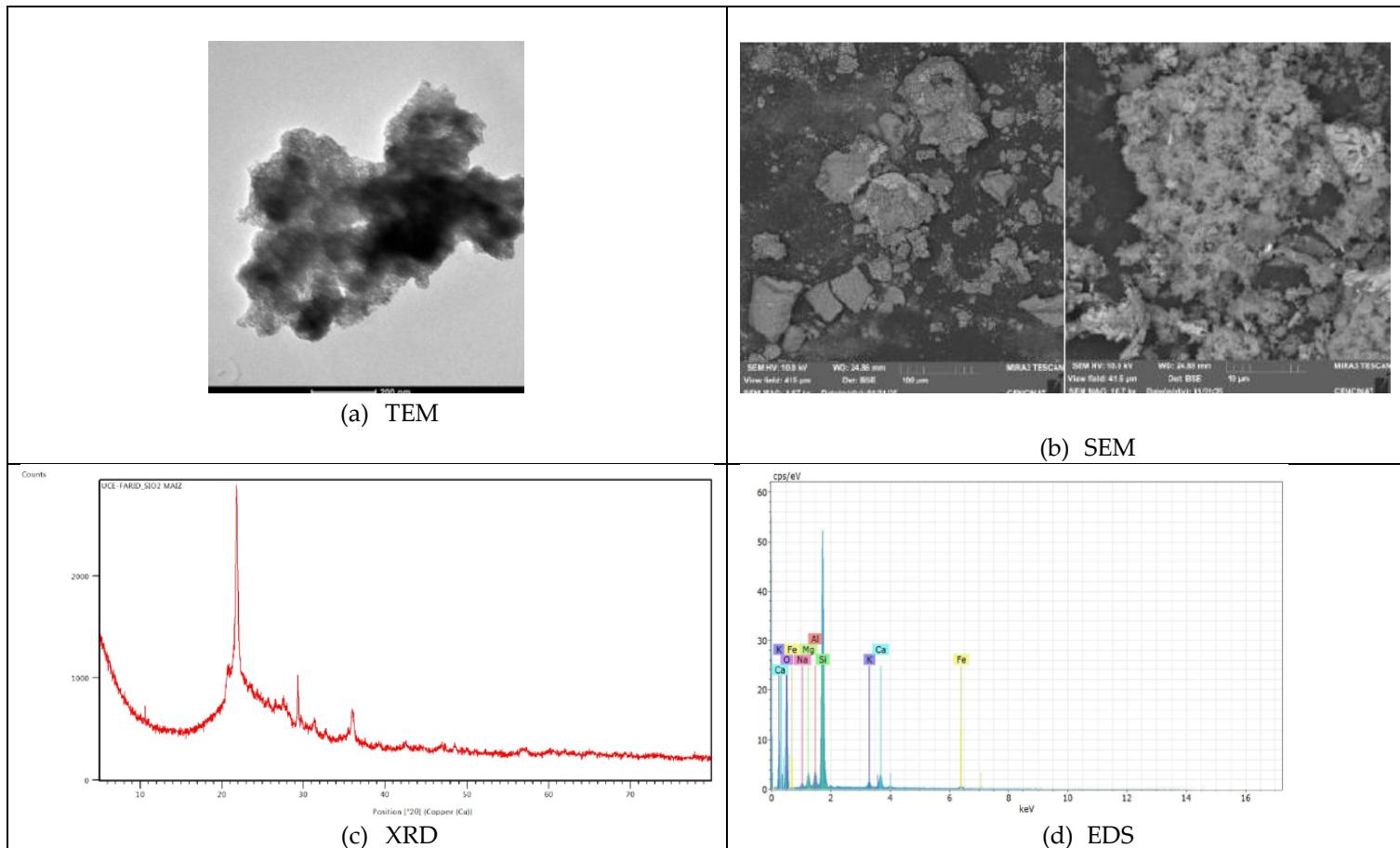


Figure 3: Microstructural characterization nanosilica.

3.3. Compressive strength performance

At 28 days, the figure tells a clear story. The control mortar (MP) behaved just as expected, reaching around 25–26 MPa—nothing surprising there. But the moment a tiny dose of 0.25% nanosilica was introduced, the curve lifted noticeably, climbing close to 29 MPa. It was a modest

change in proportion, yet it produced a striking improvement. On the other hand, when the dosage was pushed further—to 0.50%, 0.75%, or even 1.0%—the results lost momentum (**Figure 4**). Strength values flattened, circling back to the control level, and in some cases even slipped slightly. The figure reminds us that with nanosilica, more is not always better: its real power lies in small, well-

balanced additions that unlock its reactivity. ANOVA results indicated significant differences ($p < 0.05$), and Tukey's test showed that M1 was distinct from all other groups (**Table 2**).

The lesson is clear: a small dose, 0.25%, made a big difference. Larger amounts did not help and sometimes hurt. This reflects what Montgomery et al. (2016) reported: nanosilica works best in small, well-dispersed doses.

The compressive strength tests were central to this work. **Figure 5** presents the results across curing ages of 1, 3, 7, 28, and 90 days.

The control mortar (M0) reached about 31 MPa at 90 days, which is typical for standardized mortars (ASTM, 2021). The mortar with 0.25% nanosilica (M1) stood out. Its strength grew steadily and reached almost 40 MPa at 90 days—roughly 52% higher than the control.

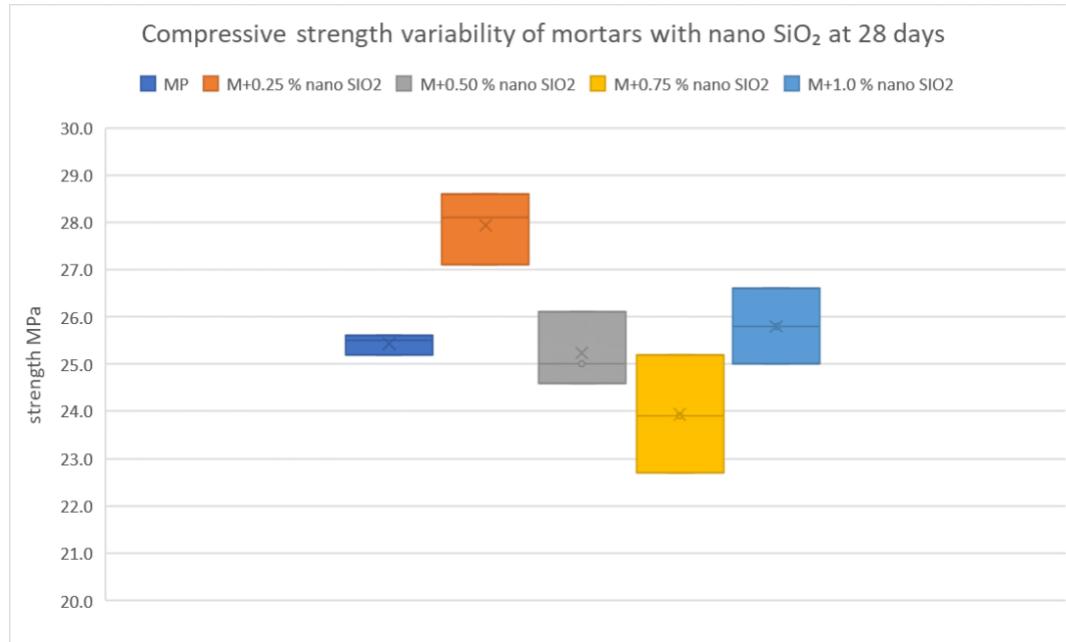


Figure 4: Compressive strength with the different nanosilica combinations

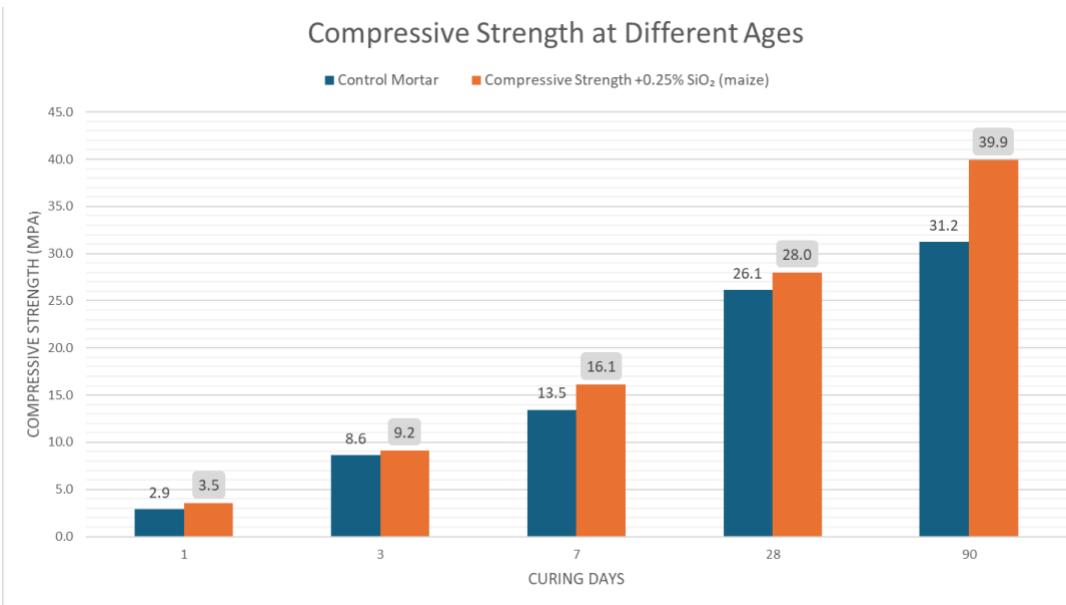


Figure 5: Compressive strength- curing time curve.

Table 2: Table. Multiple comparisons between nanosilica dosages and the control mortar at 28 days (ANOVA + Tukey HSD).

Comparison with Control (0%)	Mean Difference (MPa)	p-value	Significant (p<0.05)
0% vs 0.25%	-2.5	0.027	Yes
0% vs 0.50%	0.2	0.998	No
0% vs 0.75%	1.67	0.361	No
0% vs 1.0%	-0.37	0.981	No

3.4. Surface behavior: contact angle

The control mortar had an angle of about 25°, typical for hydrophilic surfaces. With 0.25% nanosilica, the angle rose to around 70°, suggesting a modest reduction in water absorption. At 1.0%, the angle exceeded 101°, crossing into hydrophobic territory.

This shift is promising. It matches observations by Ren et al. (2020), who saw nanosilica increase water repellency in mortars. But it is also important to be cautious. As Cheng et al. (2024) noted, contact angle is only a preliminary indicator. Tests like capillary absorption or sorptivity would be needed to confirm the hydrophobic effect. For now, these results show a tendency: nanosilica appears to make mortars less wettable, especially at higher contents (Figure 6)..

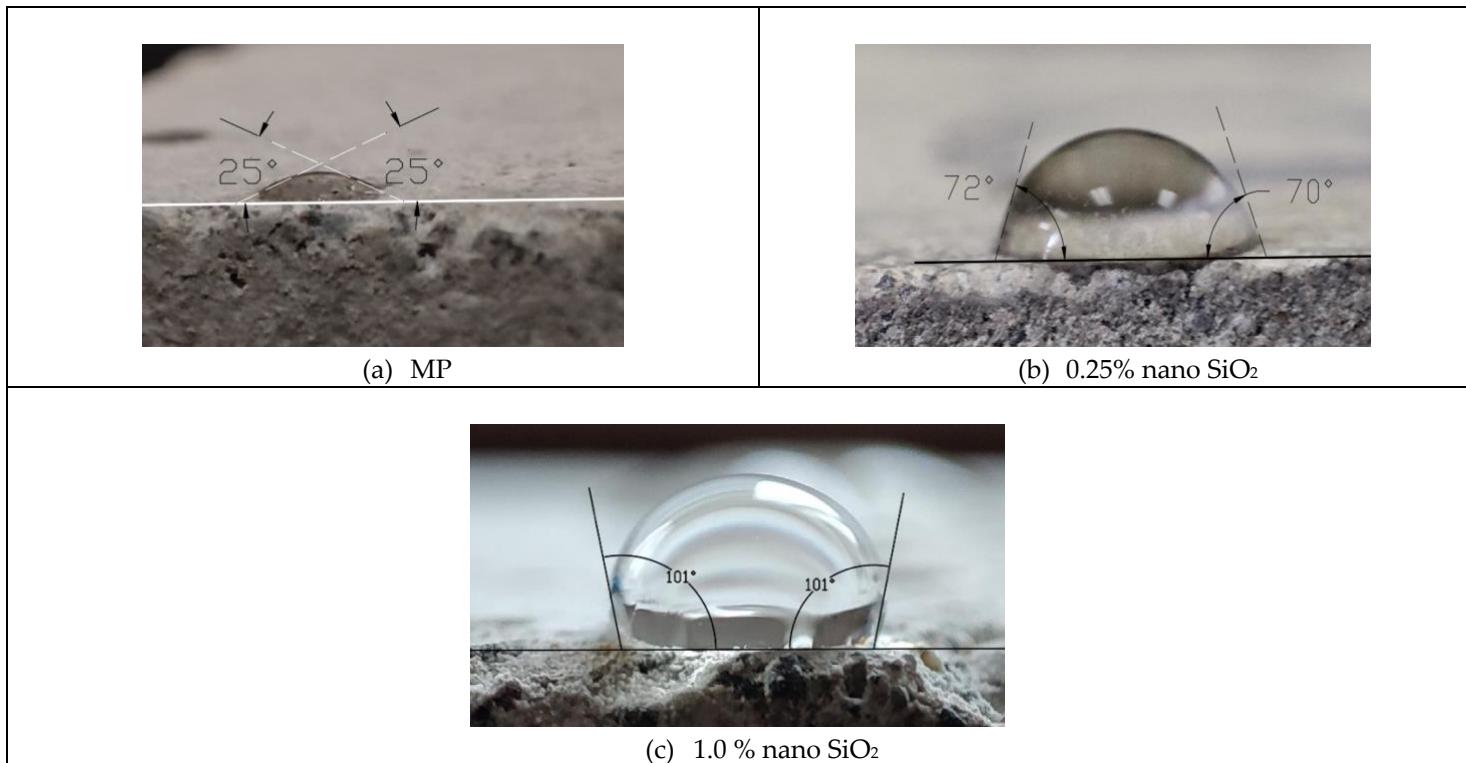


Figure 6: Contact angles with different nanosilica additions.

3.5. Mortar microstructure

The SEM images of mortars (Figure 7) told the story in visual form. In the control sample, we observed open, interconnected pores, which explain its lower strength and higher permeability.

With 0.25% nanosilica, the matrix looked denser and more compact. Voids were reduced, and hydration products appeared to be better integrated.

In the 1.0% mix, clusters of nanosilica were visible. These clusters disrupted the matrix, acting as weak points and explaining why the strength did not increase further.

These observations match what Li et al. (2021) reported: too much nanosilica leads to agglomeration, which offsets its potential benefits.

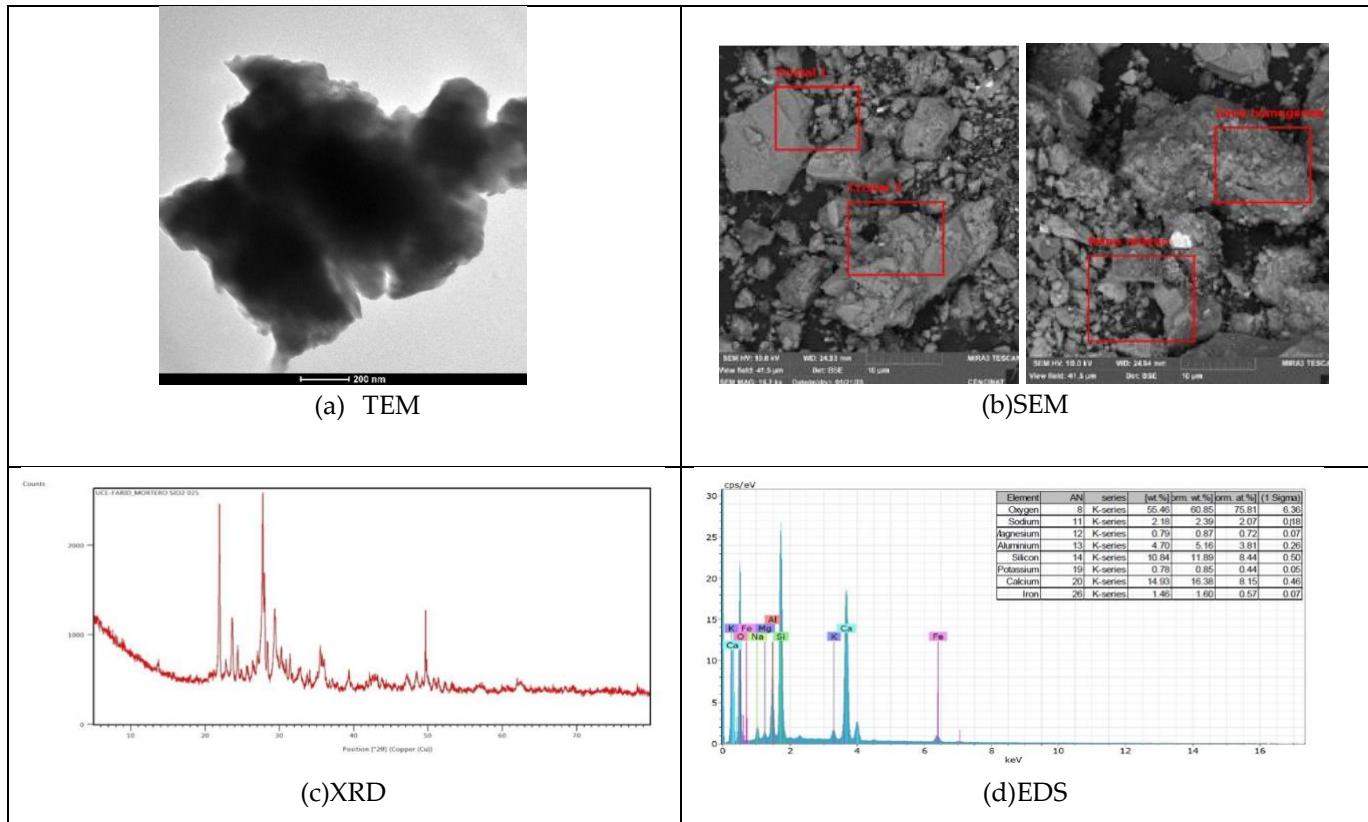


Figure 7: Microstructural characterization mortars.

3.6. Key findings

From all the data collected, three main points emerge:

- Leaves vs. cobs: corn leaves clearly outperformed cobs as a source of nanosilica, both in yield and in purity.
- Optimal dosage: 0.25% nanosilica was the sweet spot. It boosted compressive strength significantly, while higher dosages offered no added benefit.
- Functional effects: contact angle and microstructure suggest nanosilica may also improve durability, though further tests are required.

4. Discussion

The strength tests gave a clear message. The control mortar developed as expected, reaching roughly 31 MPa at 90 days. Nothing surprising here, which is consistent with typical standardized mortars (ASTM, 2021). But when a very small amount of nanosilica from corn leaves was added—only 0.25%—the response was different. The strength rose close to 40 MPa, more than 50% higher than the control. For such a minor change in composition, the outcome feels disproportionate. It suggests that the leaves carry a strong potential as a raw source of silica, perhaps underestimated until now.

Statistical analysis reinforced this trend. ANOVA confirmed significant differences ($p < 0.05$), and Tukey's test showed that the 0.25% mix stood apart from the others. Higher dosages, however, did not provide the same benefit. At 0.50%, 0.75%, and 1.0%, strength values stayed close to the control or even dropped a little. This pattern has been noted before: nanosilica works best in small, well-dispersed amounts. Beyond that point, clustering starts to dominate and the expected improvement simply vanishes (Li et al., 2021).

The microstructural observations help make sense of this. SEM images of the control revealed open, connected pores. With 0.25%, the mortar looked denser, hydration products filling gaps more thoroughly. At 1.0%, the picture changed: clusters appeared, breaking the continuity of the matrix. XRD data supported this view, with a reduction in portlandite peaks that indicates consumption of $\text{Ca}(\text{OH})_2$ through the pozzolanic reaction. EDS confirmed the high Si–O content, very much in line with reports from other agroresidues (Prabha et al., 2021). Together, the evidence is coherent: nanosilica strengthens the microstructure at low levels but loses efficiency when overdosed.

Surface behavior added another dimension. The contact angle of the control was around 25° , which means a hydrophilic surface. With 0.25% nanosilica, it increased to 70° , and with 1.0% it went beyond 100° . On its own, this is

not enough to claim hydrophobicity—surface roughness matters too, but the tendency is interesting. If confirmed by absorption or sorptivity tests, it could mean improved durability in humid or aggressive conditions (Ren et al., 2020). Beyond the laboratory, there is also the practical view: in Ecuador and across Latin America, maize is produced in large volumes, and leaves are mostly discarded. Turning this residue into nanosilica creates both a technical advantage and an environmental benefit, linking local agriculture with sustainable construction.

5. Conclusion

The study began with a simple idea: use corn leaves, a common agricultural residue, to produce nanosilica and test it in mortars. The results were encouraging.

The synthesis showed clear differences among residues. Leaves processed in a semi-industrial furnace gave yields up to 4.2%. Cobs, by contrast, stayed below 0.3% and produced impure ash. This confirmed that not all corn waste works the same way; leaves are the most suitable precursor.

When nanosilica was added to mortars, the effect was evident. With only 0.25% by cement weight, compressive strength rose by more than 50% at 90 days compared with the control. This trend was consistent at different curing ages. Larger dosages (0.50–1.0%) did not bring further gains and sometimes reduced strength, likely due to particle agglomeration.

Surface behavior also shifted. The contact angle increased from 58° in the reference mortar to more than 100° with 1.0% nanosilica. This points to a move toward hydrophobicity, which may help resist water ingress.

SEM images supported these findings. Low dosages produced a denser, less porous matrix, while high contents created weak areas from clustering.

Beyond the lab, the approach offers environmental benefits. It reuses agricultural waste, avoids open burning, and provides a sustainable additive. At the same time, partial cement replacement can reduce CO₂ emissions.

In short, nanosilica from corn leaves proved to be a low-dosage, high-impact material. Future research should focus on structural concrete, durability, and life cycle assessments to confirm its practical potential.

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Data Availability

The data supporting the findings of this study are available upon reasonable request to the corresponding author.

Conflict of interest

The authors have declared that there is no conflict of interest in this publication.

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