

Research Article

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Impact of nanoparticles on the performance of metakaolin-based geopolymer composites

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Abstract: Despite its growing recognition as a sustainable alternative to cement composites, geopolymer mortar faces performance limitations that hinder its practical application, prompting researchers to explore novel enhancement strategies, including incorporating nanomaterials, to improve its properties. Investigating the effects of various weight percentages (1, 2, 3, and 4%) of nano-silica and nano-silicon carbide on the density, microstructure, compressive and flexural strength, and abrasion resistance of metakaolin-based geopolymer mortar composites is the aim of this work. The experimental results carried out in this study demonstrate that nanoparticles can significantly change the properties of the geopolymer mortar. These changes caused increasing density, compressive, flexural, and tensile strengths, as well as wear resistance with improvement rates of 5.5, 44, 43, 52, and 41%, respectively, due to the changes in the microstructure of the geopolymer matrix, resulting from its incorporation. The present study showed that the addition of nanomaterials enhanced durability and resistance to environmental degradation, which means that structures will require less maintenance and fewer repairs, leading to lower environmental impact over the lifecycle of structures.

Keywords: geopolymer, mortar, nanoparticles, silica, silicon carbide, metakaolin, composite

1 Introduction

Geopolymers are a new family of cementitious materials that, over time, may replace traditional Portland cement

composites altogether. Geopolymer composites have lower energy in manufacturing and give much lower carbon dioxide emissions compared to Portland cement production [1]. Geopolymers are the result of the alkaline activation of a variety of source binders that are rich in silicate and aluminum, such as fly ash and metakaolin, along with GGBFS [2]. To enhance the performance and characteristics of the geopolymer, many types of nanoparticles have been introduced into geopolymers within the last 10 years. Nanotechnology has become one of the most dynamic fields of research. During the last 20 years, its importance has grown steadily due to its advanced scientific and technological innovations and practical applications. The impacts of nanoparticles on the microstructure, durability, mechanical, physical, and fresh characteristics of the geopolymer composites have been extensively studied [3].

Rathinam *et al.* assessed 10–90% slag-fly ash geopolymer after modification with 1, 2, 3, and 4 wt% of nano-silica. The addition of 1% of nano-silica achieved the best results in terms of the initial setting time, compressive and flexural strength, workability, and water absorption [4]. Coffetti *et al.* integrated nano-titanium dioxide into the alkali-activated slag-based mortar to determine the rheological and mechanical properties as well as self-cleaning ability. Under natural and accelerated conditions, self-cleaning was better after the addition of nano-titanium oxide, while there were no noticeable changes in the mechanical and rheological properties. They demonstrated that alkali content had a significant role in improving the mortar's photocatalytic efficiency [5].

Maiti *et al.* added different sizes of nano-titanium oxide to the fly ash geopolymer mortar. Five percent of nano-titanium oxide, sized 30 nm, improved the tensile and compressive strength to 6.8 and 53.6 MPa, respectively. Moreover, better durability was achieved due to a denser matrix, lower porosity, and surface roughness as a result of adding nano-titanium oxide [6].

Phokha *et al.* assessed the mechanical and magnetic properties of the geopolymer after the addition of different concentrations of nano-BaFe₁₂O₁₉ and nano-BFO (1, 3, and 5 wt%). Their results demonstrated that the resulting composite possesses ferromagnetic activity at room temperature,

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which was manifested by hysteresis loops and increasing magnetism as the percent of BFO increased. Furthermore, nano-BFO filled the gaps in the (N–A–S–H) gel structure, which contributed to a dense and compact geopolymer, resulting in about 7% increase in the composite's compressive strength as the nano-BFO increased from 0 to 3% [7].

Dişçi and Polat investigated the impact of substituting 1,2,3 wt% perlite with nano- Al_2O_3 and nano- CaO on the properties of the geopolymer under different curing conditions. According to their results, nano- Al_2O_3 improved the compressive strength significantly, especially when combining heat treatment with ambient temperature curing. Nanoparticles reduce the porosity and cracks in concrete. On the other hand, there was no significant effect on the concrete's geopolymerization pace [8].

Janowska-Renkas *et al.* examined the electrical conductivity of the fly ash–graphene–glass powder geopolymer mortar by analyzing resistivity. They used scanning microscopy and XRD to investigate the microstructure. A significant increase in conductivity was achieved as a result of adding graphene in addition to an enhancement in the strength of the geopolymer containing fly ash [9].

Tang and collaborators integrated 3-aminopropyltriethoxy silane (APTS) and graphene oxide (GO) into geopolymers to address specific challenges. They found that APTS inhibited condensation and dissolution processes, affecting workability, while GO accelerated the process of geopolymerization. In a sodium silicate alkaline environment, the GO-APTS structure remained stable, improving nanosheet dispersion within the composite. Compared to reference and pure GO, GO-APTS composites showed improved workability, transport properties, and significant tensile strength at 7 and 28 days, indicating enhanced mechanical performance [10].

Tanyildizi and coworkers examined in their study the freeze–thaw resistance of metakaolin and fly ash-based 3D-printed geopolymer mortars with different dosages (0.25, 0.5, and 0.75%) of nano-ZnO. The results indicated that the ultimate resistance to freeze–thaw of the prepared geopolymer was obtained at 0.5% nano-ZnO with a retention rate of 95.72% in the compressive strength [11].

Hashemi and coworkers developed a sulfate-resistant geopolymer mortar containing nano-silica (0, 1.0, and 2.0% binder weight replacement). The study showed that adding (1%) nano-silica causes a higher bond strength (both tensile and shear) of mixtures cured in an environment containing sodium sulfate [12].

The current work focused on studying the influence of adding oxide (SiO_2) and non-oxide (SiC) ceramic nanoparticles

at different ratios on the strengthening mechanism and the performance of the geopolymer mortar.

2 Materials

2.1 Metakaolin

Metakaolin was used as a binder in the geopolymer mortar composite preparation in this work and was purchased from Iraqi local markets. Elemental composition analysis was carried out using an X-ray fluorescence (XRF) analyzer, as illustrated in Table 1, which is compatible with the specification of ASTM C618 [13].

2.2 Fine aggregates (sand)

In this work, naturally occurring fine aggregates with a grain size of 4.75 mm were obtained from the Karbala Governorate's Al-Ekhadir region. Before use, it was cleaned and sieved. The grade, chemical makeup, and physical characteristics of the fine aggregates met the Iraqi standard requirements for IQS (No. 45/1984) [14], as illustrated in Tables 2 and 3.

Table 1: Chemical structure of the metakaolin sample

Oxide	Metakaolin (%)
Al_2O_3	33.34
SiO_2	61.12
Fe_2O_3	2.41
CaO	1.31
MgO	0.18
K_2O	0.62
Na_2O	0.88
TiO_2	0.13

Table 2: Grading of fine aggregates

Sieve no.	Sieve size (mm)	Passing (weight%)	Spec. limit according to IQS No45/1984 zone (2)
1	9.5	100	100
2	4.75	90.55	90–100
3	2.36	78.31	75–100
4	1.18	63.1	55–90
5	0.6	43.51	35–59
6	0.3	14.64	8–30
7	0.15	0.02	0–10

Table 3: Properties of sand

Properties	Value	IQS 45-1984 Iraqi Standard limits
Density (kg/m^3)	1,598	—
Specific gravity	2.7	—
Absorption (%)	0.71	—
Fineness modulus	3.1	—
Fine materials that pass through a $75\ \mu\text{m}$ sieve	1.3	3% maximum
Sulfate content (%)	0.36	0.5% maximum

2.3 Alkaline activator

A mixture of NaOH and Na_2SiO_3 solutions serves as the alkaline activator. NaOH flakes were dissolved in distilled water and then mixed with sodium silicate at a constant $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 2.5/1. The former was made at a concentration of 16 M.

2.4 Nanoparticles

Two types of nanoparticles were used in this work: nano- SiO_2 and nano-SiC, with different quantities (1, 2, 3, and 4% by the total weight of the dry components represented by metakaolin and sand). Table 4 shows the properties of the nanoparticles used in this work.

Table 4: Properties of nano- SiO_2 and nano-SiC

Property	Nano- SiO_2	Nano-SiC
Color	White powder	Gray powder
Density (g/cm^3)	2.17	3.2
Particle size (nm)	30 nm	20 nm
Purity (%)	99.8	99.5

2.5 High-range water-reducing admixture

In this investigation, MasterGlenium®54 superplasticizer (SP), a high-range water reducer, was used to increase the workability of the geopolymer mortar. A set quantity of 3% superplasticizer was applied to each geopolymer mixture.

3 Experimental route

3.1 Mix proportion

In this study, metakaolin was mixed with nanoparticles at weight percentages of 0, 1, 2, 3, and 4%. The dry components were at a 1:1 ratio. All mixes employed a constant $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 2.5 and a constant liquid-to-binder (L/B) ratio of 0.60. Table 5 shows the mix proportions design for all mixes.

3.2 Preparation of geopolymer composites

The first step in preparing the composite was the manual mixing of the dried components, including metakaolin, fine aggregates, and nanoparticles, for approximately 5–10 min until a uniform mixture was formed. To guarantee homogeneity, the wet ingredients – the alkaline activator and superplasticizer – were next added to the dry ingredients, and the mixture was stirred once more for 5–10 min. After that, the geopolymer mixture was put into steel molds that were customized to have the cube ($50\ \text{mm} \times 50\ \text{mm} \times 50\ \text{mm}$), prism ($160\ \text{mm} \times 40\ \text{mm} \times 40\ \text{mm}$), or cylinder shape ($50\ \text{mm} \times 100\ \text{mm}$) needed for each test. A thin layer

Table 5: Mix proportions for all mixtures of the geopolymer mortar composites

Mix	Metakaolin (g)	Fine aggregate (g)	NaOH (16 M)	$\text{Na}_2\text{SiO}_3:\text{NaOH}$ 2.5:1	n- SiO_2 (wt%)	n- SiO_2 (g)	n-SiC (wt%)	n-SiC (g)
Control	450	450	64.28	160.71				
1-n SiO_2	445.5	445.5	64.28	160.71	1%	9		
2-n SiO_2	441	441	64.28	160.71	2%	18		
3-n SiO_2	436.5	436.5	64.28	160.71	3%	27		
4-n SiO_2	432	432	64.28	160.71	4%	36		
1-nSiC	445.5	445.5	64.28	160.71			1%	9
2-nSiC	441	441	64.28	160.71			2%	18
3-nSiC	436.5	436.5	64.28	160.71			3%	27
4-nSiC	432	432	64.28	160.71			4%	36

of oil was used to enhance the removal of the samples from the molds and prevent sticking, which was applied to the surface inside the walls before pouring the composite. The pouring process was done in three layers. Each layer was vibrated for 1–2 min using a vibrating table to prevent the tapping of air in the composite. After that, the sample surface was refined, covered by a plastic sheet, and left to cast at ambient temperature for 24 h. Figure 1 shows the steps of the experimental work.

3.3 Curing process

After 24 h, the samples were removed from the molds and cured in two steps. First, the samples were placed inside a furnace for 3 h at 100°C. Second, the samples were left to cure at ambient temperature for 7 and 28 days.

4 Results and discussion

4.1 Microstructural analysis

After 3 h of curing at 100°C, scanning electron microscopy (SEM) was performed. The results of SEM are shown in Figures 2 and 3. According to the SEM results, curing at such a high temperature has a considerable effect on the polymerization of the composite and promotes the gel stage creation. Fewer porosities and more uniform microstructures were achieved after 28 days of curing, unlike curing for 7 days, which means that a longer time is needed for the polymerization process to be completed and for the creation of the gel phase.

The impact of nanoparticles on the microstructure of the geopolymer is shown in Figures 4 and 5. By closing the gaps in the structure and creating a thick and uniform microstructure, it was found that the addition of (3%) nano-silica significantly altered the microstructure of the geopolymer, as shown in Figure 4. Also, the addition of the nano-silica to the combination enhanced the geopolymer's polymerization process and resulted in greater gel formation by increasing the degree of silicate ion dissolution in the alkaline activator. Additionally, it was found that when the addition % increased, certain gaps were created as a result of these nanoparticles clumping together, as shown in Figure 6. Figure 5, on the other hand, shows how the nano-silicon carbide affected the geopolymer's microstructure. The results indicated that the incorporation of 2% of

these particles is essential for altering the geopolymer's structure by inhibiting the grain growth through effective dispersion, particularly at an addition ratio of 2%, which yields a fine and unified microstructure.

In this context, it was shown that an increase in the addition ratio resulted in the aggregation of nanoparticles inside the structure, leading to the formation of distinct gaps and flaws in the microstructure, as shown in Figure 7.

4.2 Density measurements

The geopolymer's density changes with the curing time, and the impact of nanoparticles on the density values at day 28 of the curing process are shown in Figures 8 and 9. It was found that the curing time had a significant impact on the density change. Because the polymerization process requires sufficient time to complete the reaction process and minimize the porosity inside the structure, the density increased gradually throughout the 28 curing days before reaching a high value. It was found that the addition of nano-SiO₂ causes a discernible increase in density, which increases to 3% with the weight fraction of nano-SiO₂.

According to SEM images, this behavior is explained by the function of nano-SiO₂ in sealing the holes in the geopolymer microstructure and gel formation, and hence raising the density. The density values were then found to decrease, as shown in Figure 8. The optimal density for the addition of nano-SiC was achieved at a ratio of 1%; as the addition percentage increased, the density declines, as shown in Figure 9.

Because of their consistent distribution at low dosages (1%), the tiny particle size of these particles helps to fill the spaces within the structure and reduce porosity; hence, the density values improved. The concentration of these particles within the structure, as shown in the SEM images, is the cause of the decrease in values beyond this ratio. Moreover, nano-SiO₂ particles had a greater effect on the density than the nano-SiC particles.

4.3 Compressive strength

Compressive strength testing was done in this study in compliance with ASTM C109-2 [15]. Three cubic samples (50 mm × 50 mm × 50 mm) were assessed at 7 and 28 days post-curing in a furnace to determine the average compressive strength for each combination. The fluctuation in the compressive strength values of the geopolymer



Alkaline Activator



Mixing process



Mixtures after casting in molds according to the dimension of each test



Samples after demolding



Samples during testing

Figure 1: The steps of the experimental work.

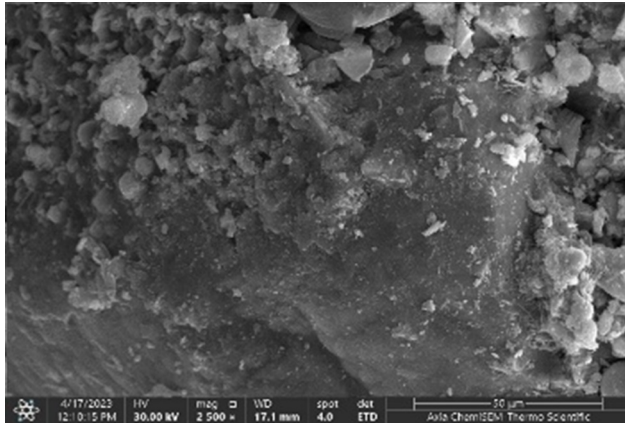


Figure 2: SEM image of the geopolymer following a 7-day cure procedure.

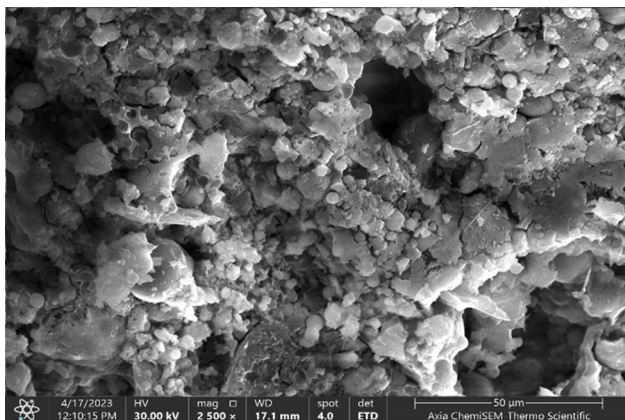


Figure 3: SEM image of the geopolymer following a 28-day curing procedure.

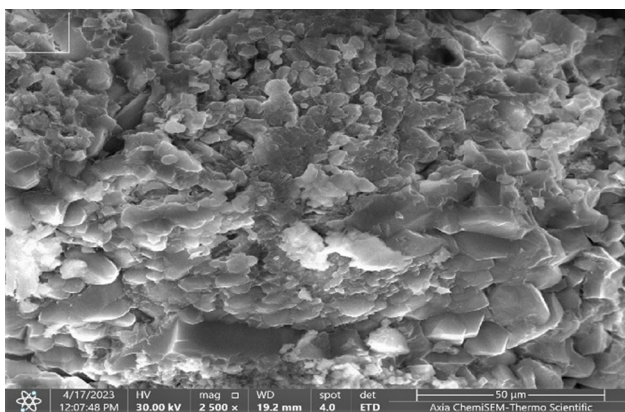


Figure 4: SEM image of the geopolymer filled with 3% n-SiO₂ after a 28-day cure procedure.

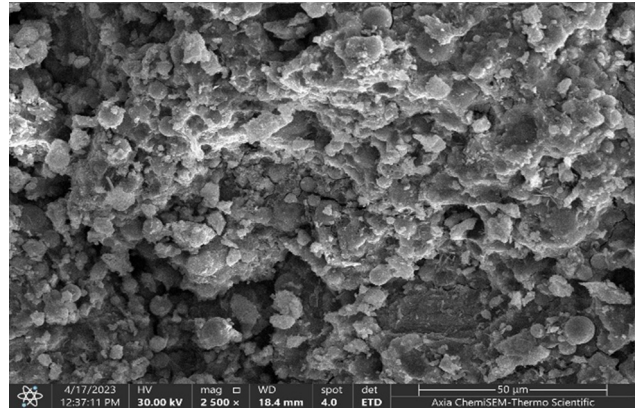


Figure 5: SEM image of the geopolymer filled with 2% n-SiC after a 28-day curing procedure.

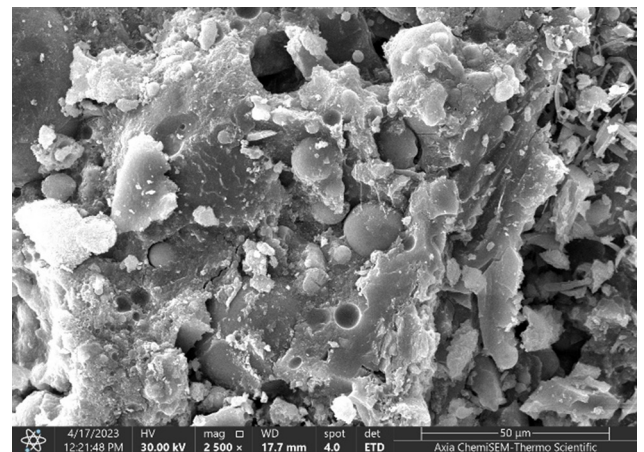


Figure 6: SEM image of the geopolymer filled with 4% n-SiO₂ after a 28-day curing procedure.

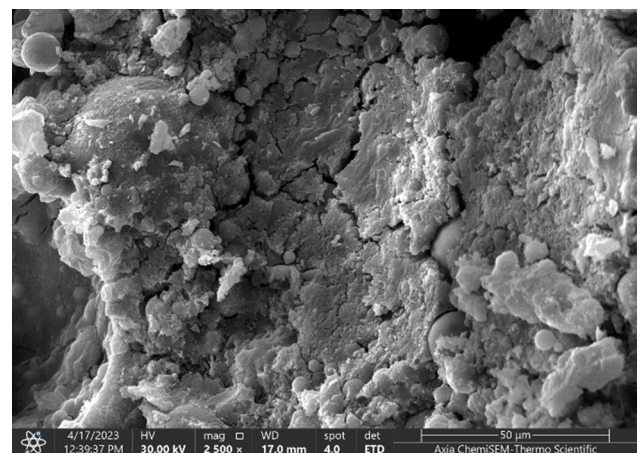


Figure 7: SEM image of the geopolymer filled with 4% n-SiC after a 28-day curing procedure.

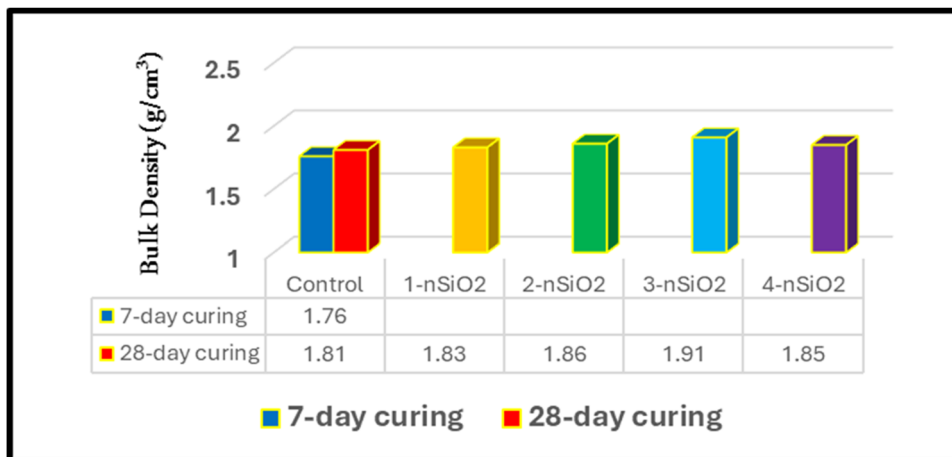


Figure 8: Density values of the geopolymer at various nano-SiO₂ weight fractions.

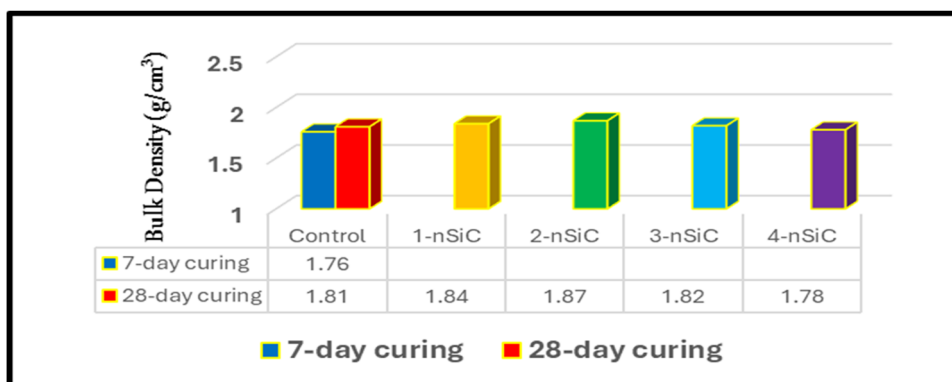


Figure 9: Density values of the geopolymer at various nano-SiC weight fractions.

mortar with varying weight fractions of nano-SiO₂ following the curing process is shown in Figure 10. The experimental findings showed that the concentration of the nano-silica and the curing conditions had a significant impact on the compressive strength values, with an improvement rate of 43%.

It was found that as the curing time increased, the dense microstructure formed as a result of the completion of the polymerization process and the formation of the gel that bonded the constituents together, increasing the geopolymer's compressive strength.

In the same situation, a 3% increase in nano-silica concentration raises the compressive strength of the geopolymer mortar. Following that, there was a modest decline in the strength at a ratio of 4%. Reactive nano-silica, which has a more exothermic interaction with metakaolin's components, is the cause of this increase

in strength. Because they can operate as nucleation sites for the development of additional (N-A-S-H) gel, which gives the combination its harder qualities, and this reaction enhances the geopolymerization mechanism [16]. More compressive strength values will result from the components' increased bonding due to the excessive gel formation.

The density of a material is closely linked to its compressive strength, with increased density resulting in enhanced compressive strength. The use of nano-silica powder occludes the nano- and micropores of the structure, hence enhancing the compressive strength values. It was observed that the compressive values diminished slightly at 4% weight, attributable to the presence of an unreacted silica ratio resulting from the concentration of these particles. The impact of nano-SiC at various weight fractions on the compressive strength of mortar made of geopolymer materials is shown in Figure 11.

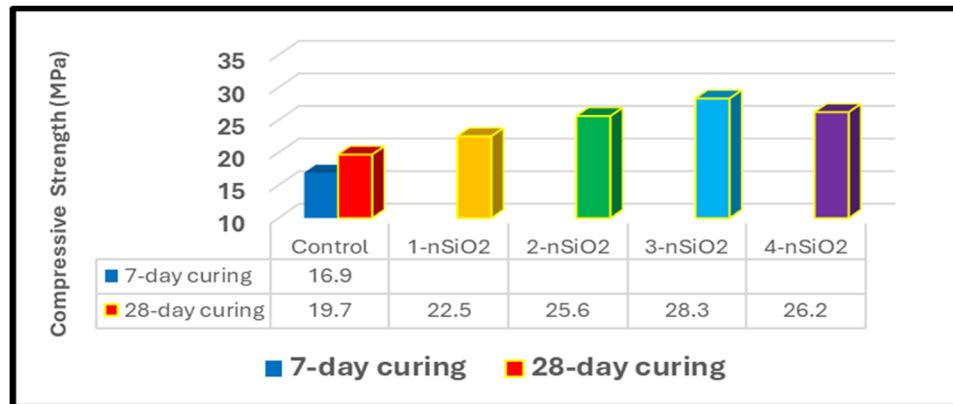


Figure 10: Compressible strength values of the geopolymer materials at different weight fractions of nano-SiO₂.

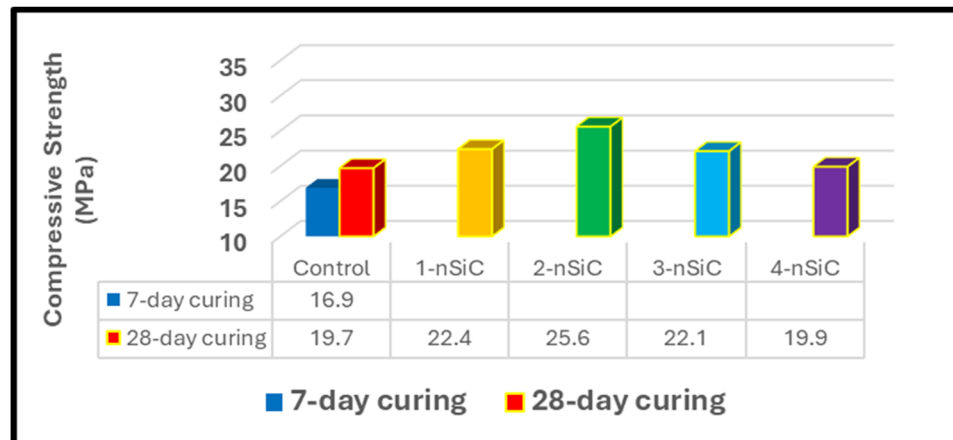


Figure 11: Compressible strength values of the geopolymer materials at different weight fractions of nano-SiC.

According to the data, the addition of these nanoparticles by up to 2% clearly increased the compressive strength. The strong connection with the matrix and the excellent dispersion of these particles are responsible for this increase. In composite materials, bonding plays a crucial role in the load transmission process from the matrix to the reinforcing phase (particles), which raises the material's compressive strength. However, the presence of these ceramic particles prevents the production of a fine microstructure with high compressive strength by impeding the growth of grains inside the geopolymer matrix [17].

The compressive strength then noticeably decreases when the weight fraction of these particles is increased to 4% because of the clear agglomeration of particles caused by their heterogeneity and difficulty in dispersing within the matrix, which creates some structural gaps [18]. Additionally, it was discovered that nano-SiO₂ played a larger role in increasing the compressive strength than

nano-SiC. This is because nano-SiO₂ contributes to the polymerization process and the formation of gels that boost strength, whereas nano-SiC strengthens the material by forging a strong bond with the matrix and has no influence on the polymerization process.

4.4 Flexural strength

The flexural strength tests of the geopolymer mortar with different percentages of nano-SiO₂ during the curing process were performed according to ASTM C 293-03 [19] standard, and the results are shown in Figure 12. An average of three prism specimens, each measuring 160 mm × 40 mm × 40 mm, was used to test the flexural strength of different geopolymer mortar formulations. After being treated in the furnace, the specimens were evaluated at two distinct ages: 7 and 28 days.

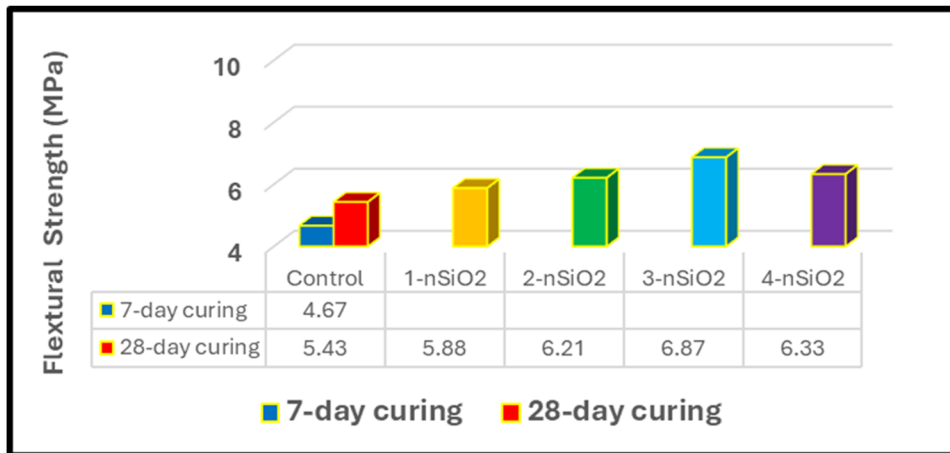


Figure 12: Flexural strength values of the geopolymer at various weight fractions of nano-SiO₂.

The results demonstrate that as the curing time increases, the flexural strength enhances. Moreover, the integration of nano-SiO₂ improves the flexural strength slightly until 2 wt% because of the strong bonds between the components caused by nano-SiO₂. At 3 and 4 wt% of nano-SiO₂, the strength decreased slightly. The increase in the geopolymer stiffness and brittleness may be the cause of the decrease in flexural strength. Conversely, it was found that the addition of nano-SiC, as shown in Figure 13, significantly increased the flexural resistance values, which increased to 2% wt as the weight fraction of these particles increased. This increase in flexural strength is the result of these hard particles' ability to improve the brittle fracture behavior of geopolymer cement by delaying and halting the formation of fractures caused by stress. Because of the previously described agglomeration

tendency of these particles, which results in cavities in the structure, the flexural strength value decreases as the weight fraction increases by 4% wt.

4.5 Splitting tensile strength

The splitting tensile test was performed in this investigation to evaluate the fracture resistance of the geopolymer mortar, following the ASTM C496 [20] standard. Following the curing procedure, cylindrical mortar specimens of 50 mm × 100 mm were made and examined. While the geopolymer mortar exhibits excellent performance under compressive loads, it is also crucial to evaluate its tensile properties as they determine its resistance to cracking. The

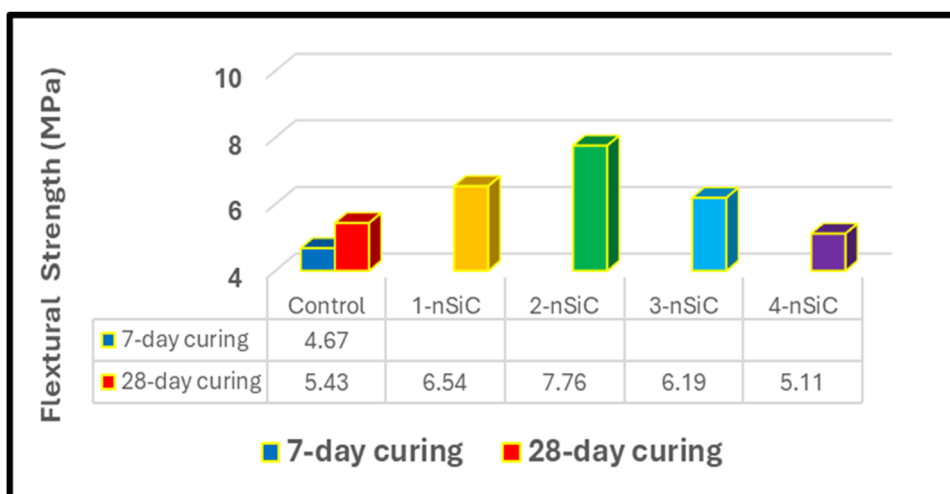


Figure 13: Flexural strength values of the geopolymer at various weight fractions of nano-SiC.

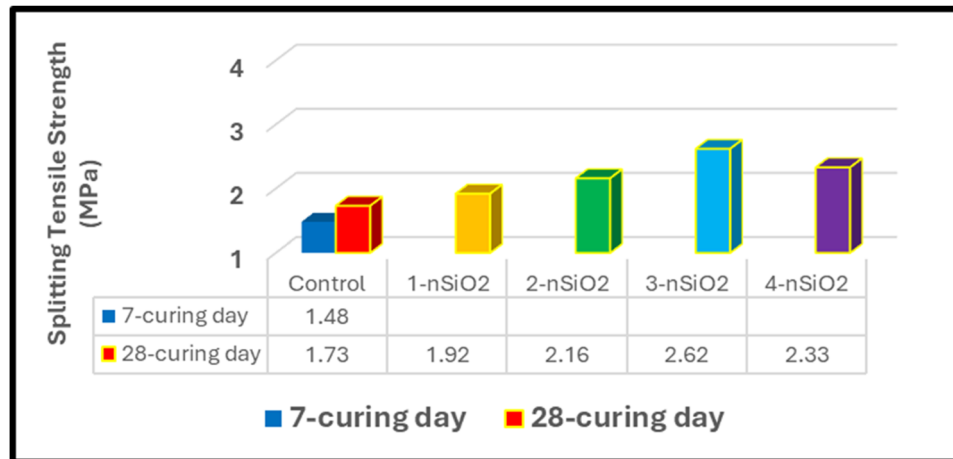


Figure 14: Splitting tensile strength values of the geopolymer at various weight fractions of nano-SiO₂.

splitting tensile strength results for all geopolymer mixtures are shown in Figures 14 and 15. These results exhibited a similar trend to the flexural test, confirming the previous findings that nano-SiC outperformed nano-SiO₂ in terms of enhancing geopolymer mortar's fracture resistance [21].

4.6 Abrasion resistance

The experiment was carried out following the British standard EN-1338:2003 [22]. The procedure involved subjecting the top surface of the samples to controlled abrasion using fused alumina. For the abrasion, a large steel wheel of 200 mm in diameter and 70 mm in breadth was employed. Its Brinell hardness ranged from 203HB to 245HB. A minimum of 100 mm × 100 mm × 70 mm should be the

size of the test specimens. The abrasion index, which quantifies the abrasion resistance, was calculated using the following equation:

$$\text{Abrasion index} = \frac{WL}{WI} \times 100,$$

where WL represents the weight loss, and WI represents the initial weight of the sample.

The impact of nanoparticles on the synthesized geopolymer's resistance to wear is shown in Figures 16 and 17. Compared to the effect of nano-SiO₂, the results show that the addition of nano-SiC has a significant impact on the wearing resistance values. This result may be ascribed to the high hardness and stiffness of nanoparticles, which are interconnected reliably to the geopolymer framework, bringing about a high interfacial region between the geopolymer matrix and nano-SiC particles that leads to impeding mortar abrasion [23].

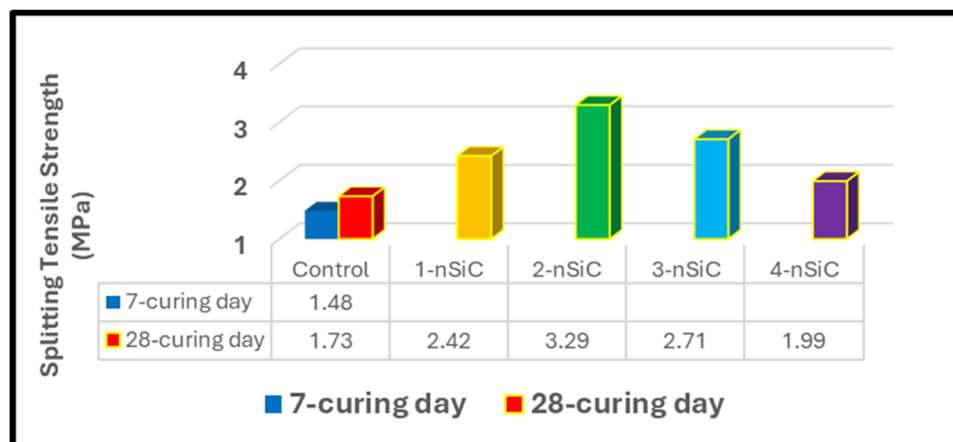


Figure 15: Splitting tensile strength values of the geopolymer at various weight fractions of nano-SiC.

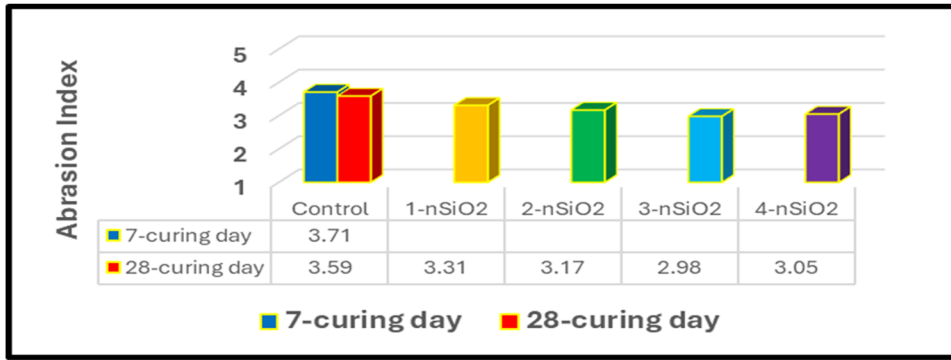


Figure 16: Abrasion index values of the geopolymer at weight fractions of various nano-SiO₂.

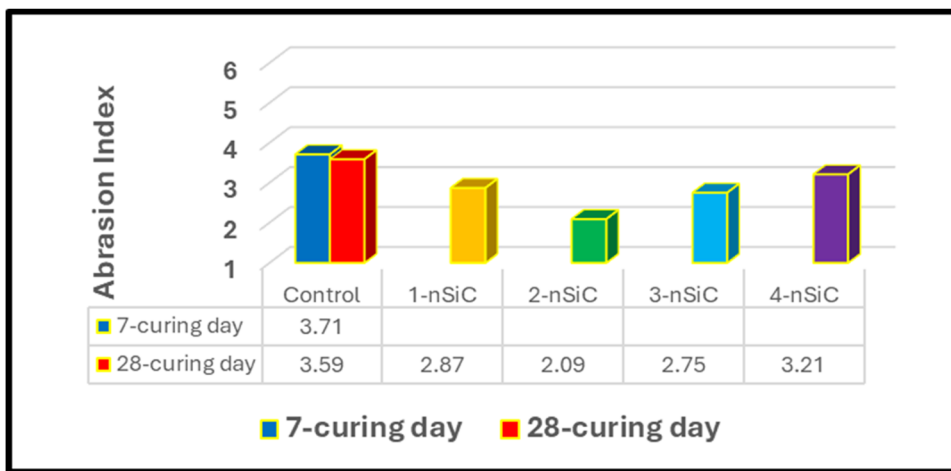


Figure 17: Abrasion index values of the geopolymer at various weight fractions of nano-SiC.

Also, the nanoparticles enhance abrasion resistance by improving the microstructure, strengthening the interfacial bonding, and reducing the friction due to the smooth and hard surfaces of these particles (e.g., SiC). Moreover, nanoparticles act as crack arrestors within the geopolymer matrix. When microcracks form due to abrasive forces, the nanoparticles can bridge these cracks, preventing them from propagating further. This crack-bridging mechanism enhances the toughness and durability of the material, making it more resistant to abrasion. It was also noticed that there is a similar behavior as in the rest of the properties, where the wear resistance value decreases gradually at high percentages of nanomaterials added and for the same reasons that were previously explained.

5 Conclusions

1. A key element in enhancing the characteristics of geopolymer mortar is the curing process parameters, such as temperature and duration.
2. The enhancement in geopolymer's performance depends principally on the role of the nanoparticles in the geopolymerization process, their homogeneous distribution within the geopolymer structure, and their ability to impede the movement of the cracks and the growth of granules.
3. The addition of nanoparticles plays a vital role in improving the mechanical properties in different ways. Nano-SiO₂ had the greatest effect on improving the compressive strength and density by increasing the degree of silicate ions dissolved in the alkaline activator, producing more N-A-S-H gel responsible for strength. Instead, nano-SiC enhances the flexural and splitting strength through its role in hindering the emergence and movement of cracks.
4. The reinforcement mechanism depends on the type and effectiveness of the nanoparticles. Silica is a reactive powder that contributes to the geopolymerization process and improves the strength, while silicon carbide does not contribute to the polymerization process because it is inert with high hardness and strength,

dispersed within a structure that contributes to hindering the formation and movement of cracks.

5. The optimal dosages of nano-SiO₂ and nano-SiC in geopolymer composites are determined by their respective roles in the geopolymerization process, dispersion characteristics, and their impact on the mechanical properties and workability. Nano-SiO₂ at 3% maximizes geopolymerization and microstructure refinement, while nano-SiC at 1–2% provides the best balance of mechanical reinforcement without causing agglomeration or excessive brittleness.
6. The addition of nanoparticles enhanced the abrasion resistance of the geopolymer mortar, with a noticeable influence of nano-SiC on the wear resistance values compared to nano-SiO₂.
7. Nanoparticles tend to agglomerate due to their high surface energy, which can negatively affect the homogeneity and performance of the composite, in addition to the difficulty of achieving uniform dispersion in large-scale production.
8. This study presented high specification building materials that can be used in construction and infrastructure, fire-resistant materials, and the oil and gas industry.

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Conflict of interest: The authors state no conflict of interest.

Data availability statement: All data generated or analyzed during this study are included in this published article.

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