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Influence of Nanomaterials on the Performance of Lightweight Concrete Containing Silica Fume Against Chemical Attacks

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ABSTRACT

This research studied the influence of nano silica (NS) on the performance and properties of lightweight concrete (LWC) containing silica fume (SF) and exposed to sulfate attack (sodium and magnesium) at a concentration of 0.3%. Although previous studies have examined the effect of adding NS to ordinary concrete, its impact on LWC with SF and exposed to double sulfate attack remains limited; therefore, this study prepared various mixtures were created using two types of cement: ordinary Portland cement (OPC) and sulfate-resistant cement (SRPC), with and without the incorporation of NS, to study the changes in weight, visual inspections, and compressive strength. LWC was produced by replacing 50% of the coarse aggregate with lightweight pumice aggregate. The results showed that the highest weight loss after exposure to sulfate solution appeared in samples containing OPC without the addition of NS, at 16.2% and 24.5% after 28 and 90 days. This is due to the high tricalcium aluminate C_3A content. As for the samples containing SRPC, they showed a lower loss of 6% and 8% after 28 and 90 days, which confirms the effect of the low content of C_3A. The addition of 4% NS reduced the strength loss in both mixtures when exposed to chemical attack. Therefore, NS is considered a material that improves the resistance of concrete to chemical influences by decreasing the $Ca(OH)_2$ content.

1. INTRODUCTION

Lightweight concrete (LWC) is a construction material whose density is less than that of normal concrete. According to European codes, the density of LWC is less than 2000 kg/m³ [1]. Consequently, LWC is an attractive option for decreasing a structure's weight and improving its insulation characteristics, such as thermal and acoustic insulation [2, 3]. To improve the strength of LWC, many cementitious materials, like SF or fly ash [4]. Concrete may be subjected to atmospheric conditions, which can contribute to the degradation of concrete structures. However, the most notable is the sulfate attack on concrete. Sulfur oxides harm concrete, particularly sulfate (SO_4), whereas reinforcing steel is harmed by chlorides [5]. Sulfate attacks come in various forms, but the most prevalent ones that interact with concrete are calcium, sodium, and magnesium sulfate, which are classified based on how aggressive they are [6]. In recent years, researchers have been working on nanotechnology forms of its use of nanoparticles and for their physical and chemical properties, which make it more useful in enhancing the performance and durability of LWC [7]. Due to its high surface area, it exhibits high pozzolanic activity like nano-silica, nano-alumina, nano-ceramic, and nano-metakaolin. Specifically, NS decreases permeability and fills micropores. It also improves the calcium hydroxide reaction to create calcium silicate gel (C-S-H), which is more impervious to chemical attack. Furthermore, nanoparticles are essential for strengthening the Interfacial Transition Zone (ITZ), which is often the weakest area between cement paste and aggregate [8, 9]. Herki [10] studied lightweight natural materials, such as pumice, used as partial replacements for coarse aggregates to produce LWC. He used replacement ratios of 10% to 50% pumice aggregates. His study included examining workability, compressive strength, density, and water absorption. He concluded that LWC containing pumice has a low density and sufficient strength for lightweight structural applications, although as the pumice content increases, workability decreases and water absorption increases compared to ordinary concrete. Abdullatif and Abdullatif [11] investigated in their study the shear and flexural strength of LWC beams made of pumice by partially replacing crushed gravel with 75%. The value of compressive strength they got was 29 MPa. Pumice aggregate has high absorption due to its high porosity, which reduces the strength and resistance of LWC to sulfate attack. Therefore, Naser and Zainab [12] concluded in their study that compressive strength would decrease by 40% when sulfates were added at a rate of 6% of the cement weight in the case of using pumice to produce LWC. For the purpose of studying the methods and extent of the effect of sulfate on concrete during sulfur attack. Liu et al. [13] conducted a study to determine the influence of chemical and physical sulfate attack on concrete using different methods, namely full immersion and partial immersion, and using several concentrations (1, 5, 10) %. The findings showed that the compressive strength will initially increase at low concentrations as a result of the formation of ettringite and gypsum that fill the pores, then it will decrease sharply at high concentrations or upon exposure for long periods due to the continued formation of gypsum and ettringite, which leads to the formation of internal stresses and thus cracks and disintegration of the structure. Therefore, there was a need to improve the performance of concrete to produce concrete with properties suitable for environments exposed to different conditions. To achieve this, Xu et al. [14] A review study of the effect of nanomaterials on ITZ, which is considered the weakest area in concrete, showed that the use of nanomaterials, especially NS, will improve the mechanical properties and microstructure of ITZ by decreasing the size and density of (CH) and increasing the formation of C-S-H, thus reducing porosity, increasing microhardness, and increasing cohesion between aggregates and cement paste, which is the main factor for improving the properties of concrete and developing its compressive strength and durability. Especially, AlTawaiha et al. [15] proved that the use of NS in concrete will improve its properties and increase its compressive strength, shear strength, and bending strength. This is due to the filling effect and the high pozzolanic interaction that NS possesses. In addition, in addition Du et al. [16] also carried out a study in which they explored the effect of incorporating NS and the extent of its influence on the characteristics of concrete. Their outcomes indicate that incorporating NS at a rate of 1-3% improves the compressive strength by 25-45%, as well as reducing total porosity and permeability, which improves the concrete's resistance to chemical attack. The loss of mass when subjected to chemical attack will decrease, and the rate of corrosion of reinforcing steel will be reduced. To study the effect of adding nano-silica to concrete when exposed to sulfur attack, Gopalakrishnan and Jeyalakshmi [17] investigated the effect of adding 5% and 10% NS on concrete exposed to sulfate attack, such as sodium sulfate at a concentration of 10% was investigated. The results showed that samples containing NS showed better resistance to sulfates compared to conventional concrete. This is due to the interaction of NS with $Ca(OH)_2$ to produce additional C-S-H, which reduces pores and the formation of gypsum and ettringite, and proves that adding 5% NS by weight of cement increases compressive strength by 40%, and adding 10z of NS enhances it by 26% at 28 days. While NS has been studied in normal concrete, its synergistic effect with SF in pumice-based LWC under combined sodium and magnesium sulfate attack remains underexplored. Therefore, the purpose of this study is to investigate the impact of NS on LWC that is subjected to sulfate attack to improve its durability and mechanical characteristics in aggressive conditions.

2. MATERIALS

2.1 Cement

In this work, two types of cement were used: ordinary Portland cement (Type I) with moderate C_3A content (7.9%), which is available in the Badoosh Factory, Nineveh, and the second type was sulfate-resisting Portland cement (Type V) with a (2.5%) content of C_3A, which is available at the Mass Sulaymaniyah factory in Iraq. Both types comply with Iraqi Specification No. 5/2019 [18]. The physical and mechanical properties are shown in Table 1, while Table 2 displays the chemical compositions for both types of cement.

Table 1. Mechanical and physical characteristics of cements

Physical Properties	Type I	Type V	IQS Limits 5/1984 [18]
Specific gravity	3.05	3.12	------
Blaine specific surface (m^2/Kg)	330	292.6	≥ 230 Type I ≥ 250 Type V
Initial setting time (minutes)	75.0	90.0	≥ 45minute
Final setting time (hours)	4:10	3:30	≤ 10 hours
Compressive strength, MPa at:			
3-days	19.2	19.0	≥ 15.0
7-days	27.3	26.4	≥ 23.0

Table 2. Chemical compositions of cements

Chemical Composition	Content Percentage (%)		IQS Limits 5/1984 [18]
	Type I	Type V	
SiO_2	20.99	22.66	------
AL_2O_3	5.43	4.2	------
Fe_2O_3	2.325	5.01	------
CaO	60.31	60.64	------
MgO	3.87	2.45	≤ 5.0%
Loss of ignition (LOI)	2.60	2.20	≤ 4%
SO_3	1.5	2.11	≤ 2.8% Type I ≤ 2.5% Type V
C_3S	35.68	27.45	------
C_2S	36.73	40.42	------
C_3A	7.9	2.5	≥ 5.0% Type I ≥ 3.5% Type V
C_4AF	10.5	15.23	------

2.2 Aggregate

Natural river sand was utilized as a fine aggregate in this study, which was obtained from the Mosul-Kanhash region in Iraq, with a fineness modulus of 2.8. The specific gravity of the river sand used was 2.58, and the sieve analysis for sand, which complies with ASTM C136/C136M-14 [19], is displayed in Table 3. Crushed aggregate with a maximum size of 12.5 mm was used, which is available locally in Mosul, Iraq; the specific gravity was 2.68, and its absorption was 1.85%. The sieve analysis for crushed aggregate to ASTM C136/C136M-14 [19] is shown in Table 4. Pumice is a lightweight volcanic rock with high porosity and low density (see Figure 1). It was used to get lightweight concrete. Table 5 shows the sieve analysis of pumice in which agrees with the standard specification of ASTM C330/C330M [20], while the pumice's physical properties are listed in Table 6 according to ASTM.

Figure 1. Pumice stone

Table 3. Fine aggregate's sieve analysis

Sieve Size (mm)	Passing (%)	ASTM C136 Limits [19]
9.5	100.0	100
4.75	100.0	95 - 100
2.36	89.0	80 - 100
1.18	72.0	50 - 85
0.6	45.5	25 - 60
0.3	17.6	10 - 30
0.15	4.5	0 - 10

Table 4. Crushed aggregate's sieve analysis

Sieve Size (mm)	Passing (%)	ASTM C136 Limits [19]
25	100.0	100
19	95.0	90 - 100
12.5	60.0	40 - 70
9.5	8.0	0 - 15
4.75	2.0	0 - 5

Table 5. Sieve analysis of pumice

Sieve Size (mm)	Passing (%)	ASTM C330 Limits [20]
19.5	100.0	100
12.5	92.8	90 - 100
9.5	54.5	40 - 70
4.75	12.7	0 - 20

Table 6. Physical properties of pumice

Physical Properties	Value	Standard Specification
Specific Gravity (OD)	1.9	ASTM C127-01 [21]
Specific Gravity (S.S.D)	1.38	ASTM C127-01 [21]
Water Absorption (%)	23.5	ASTM C127-01 [21]
Loosed density (kg/m^3)	560	ASTM C29 / C29 M-97 [22]
Rodded density (kg/m^3)	740	ASTM C29 / C29 M-97 [22]

Table 7. Chemical compositions of silica fume

Constituent	Result (%)	ASTM C1240 [23]
Sio_2	88.43	≥ 85
AL_2O_3	1.25	-----
Fe_2O_3	0.1725	-----
MgO	0.481	-----
CaO	0.353	-----

2.3 Silica fume

Grey-colored SF was utilized at a rate of 10% replacement by weight of cement. The specific gravity value was 2.21, and the value of Loss on Ignition (LOI) was 2.89%. Table 7 displays the chemical composition of silica fume, which was compatible with ASTM C1240 [23].

2.4 Nano silica

Figure 2 shows NS, which is a white dust composed of amorphous silica powder. NS has a particle size of less than 100 nm, which enables it to have a large surface area, high chemical reactivity, and high dispersion. In addition, the high purity of silicon oxide results in large pozzolanic activity. Amorphous silica rapidly responds with calcium hydroxide, $Ca(OH)_2$, formed by cement hydration, to form an additional hydrated calcium silicate (C–S–H) gel, which is an important element for increasing the strength of concrete. Commercial NS oxide was obtained from Emeishan Changqing New Material Co., Ltd. Table 8 shows the properties of NS.

Figure 2. Nano silica

Table 8. Properties of nano silica

Properties	Value
SiO_2 content	99.1%
Surface area (m^2/gm)	190
size of particle (nm)	20 - 30
Melting point/Melting range	> 2000℃
Retained on 325 sieves (45 μm)	0.009
Solubility in / Miscibility with water	soluble
Specific gravity	2.4

2.5 Superplasticizer (SP)

The commercial name is ViscoCrete -180 GS, which adds concrete mixtures to improve their workability. The chemical and physical properties of the superplasticizer are listed in Table 9, as per ASTM C494 [24].

Table 9. Superplasticizer information

Composition	Aqueous Solution of Modified Polycarboxylates
Appearance and color	Light brownish
Specific gravity	1.070 ± 0.02 g/cm^3
Ph-Value	4-6

2.6 Sulfate solution

Figure 3 shows sodium and magnesium sulfate used in treatment water to create aggressive environmental conditions with a concentration of 0.3%, which are among the most dangerous factors in concrete deterioration. These salts attack cement compounds, especially calcium hydroxide, $Ca(OH)_2$.

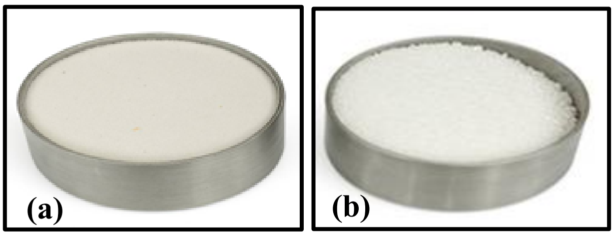


Figure 3. (a) Sodium sulfate, and (b) Magnesium sulfate

3. EXPERIMENTAL PROCEDURE

In this work, pumice aggregate was used as a lightweight coarse aggregate to produce LWC with a mixing ratio of 1:2:4, where 50% of the coarse aggregate was replaced with lightweight pumice aggregate. Two types of cement were used: OPC and SRPC, in addition to sand and water with a w/c of 0.45, superplasticizer at 0.5%, and SF of 10% replacement of the cement weight. NS was added as a partial replacement of the cement weight at a rate of 4%. The mixing process can be summarized in the following steps:

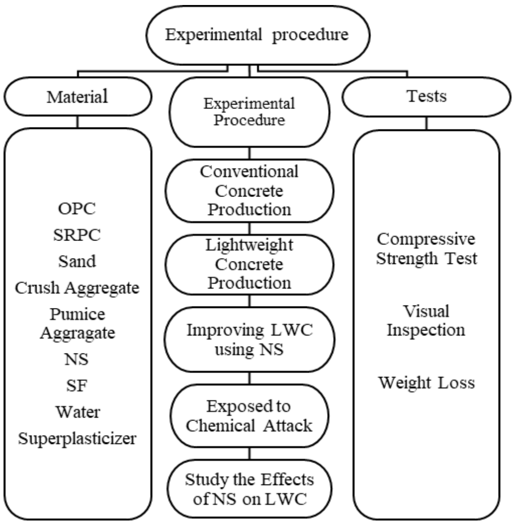


Figure 4. Flowchart for the experimental work

- The mixer was checked for cleanliness, and its internal surface was moistened before adding the materials.
- The pumice aggregate was weighed and then soaked in water. Before using it in mixing, it was removed from the water, and its surface was dried to obtain SSD.
- The materials to be used, such as sand, gravel, SF, NS, superplasticizer, and water, were weighed.
- The dry materials were mixed for approximately two minutes.
- About two-thirds of the water was added to the mixture, and it was mixed for an additional two minutes. Then, the superplasticizer was added to the remaining water and added to the mixture. The mixing process continued for 2–3 minutes to ensure homogeneity.
- After preparing cubic samples 10 × 10 × 10 cm and cylindrical samples 10 × 20 cm for mechanical tests, they were cast into molds and compacted.

After the concrete hardened, the samples were removed and placed in the curing, which is normal water or a salt solution with a concentration of 0.3%. It contains sodium and magnesium sulfate. to simulate aggressive environmental conditions and was treated for different periods of time, 7,28 and 90 days, for the purpose of conducting mechanical tests, such as compressive strength on it. Table 10 illustrates the lightweight mixtures used in this work. Figure 4 summarizes the experimental work in this study.

Table 10. Lightweight concrete mix proportions (Kg/m^3)

Mix	OPC Kg/m^3	SRPC Kg/m^3	Sand Kg/m^3	Gravel Kg/m^3	Pumice Kg/m^3	Silica Fume Kg/m^3	Nano Silica %	Nano Silica Kg/m^3	Type of Curing
M1	298.2	----	662.7	661.5	338.1	33.13	----	----	water sulfate
M2	284.9	----	662.7	661.5	338.1	33.13	4	13.25	water water
M3	----	298.2	662.7	661.5	338.1	33.13	----	----	water sulfate
M4	----	284.9	662.7	661.5	338.1	33.13	4	13.25	water

4. DISCUSSIONS AND RESULTS

4.1 Weight loss

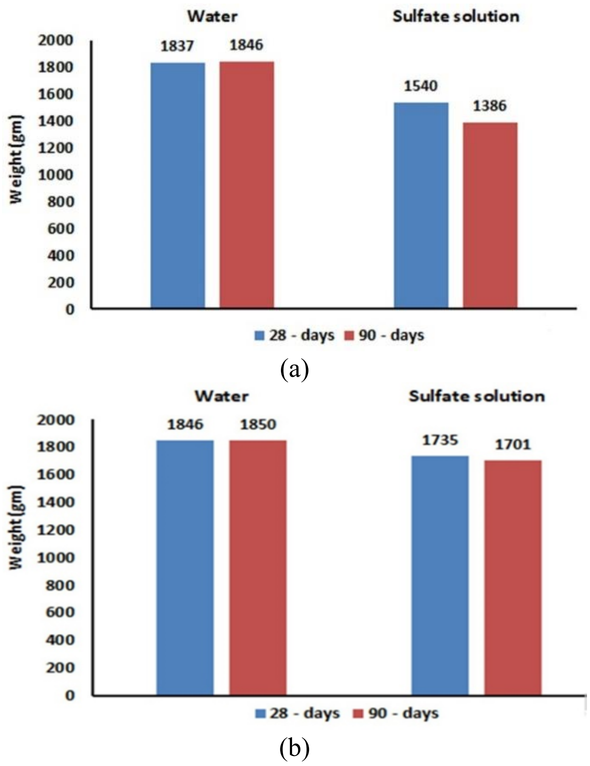
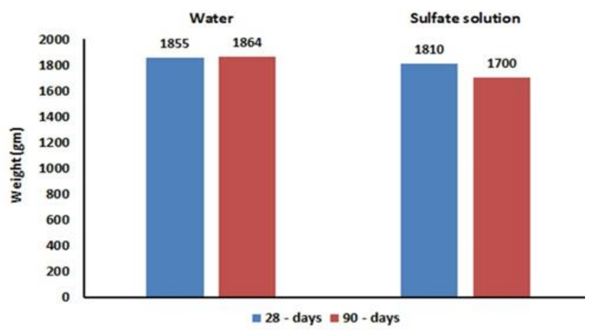
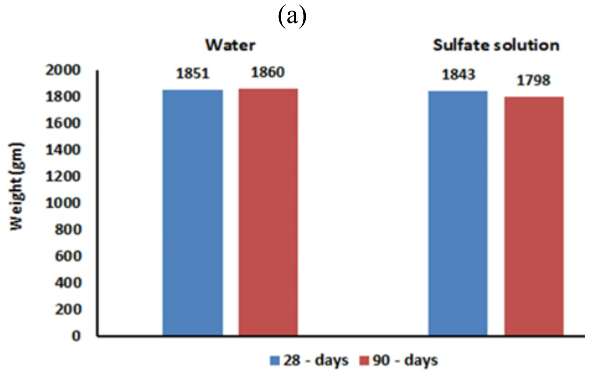


Figure 5. Weight change of (a) OPC and (b) SRPC concrete samples without nano-silica after 28 and 90 days of curing in water and a 0.3% combined sulfate solution

Three cubic samples (100 mm) from each type of cement, without and with the NS, were weighed after 28 and 90 days of moist curing. Then, the samples were subjected to two kinds of curing: with water and a combined solution of sodium and magnesium sulfate, until the time of testing. Figure 5 illustrates the specimen's weight of OPC and SRPC without the impact of NS. Samples containing OPC and without NS recorded the highest weight loss of approximately 16.2% at 28 and 24.5% after exposure for 28 and 90 days, respectively. This is due to the high content of tricalcium aluminate (C_3A), which in turn increases the rate of formation of ettringite and gypsum upon sulfate reaction. As for LWC with SRPC cement and without NS, it showed a lower weight loss of approximately 6.0% and 8.1% after 28 and 90 days of exposure. This confirms the low content of C_3A in the cement, which increased the resistance to sulfate attack. The influence of NS against chemical attack exposure is depicted in Figure 6, where the weight loss has been significantly improved. This is due to the large filling effect and its high pozzolanic activity.



(a)



(b)

Figure 6. Weight change of (a) OPC and (b) SRPC concrete samples with nano-silica after 28 and 90 days of curing in water and a 0.3% combined sulfate solution

4.2 Visual inspection of specimens

From the visual inspection of the concrete sample after immersion for 28 and 90 days in the sulfate solution, it was observed that all samples were severely deteriorated at late ages (see Figure 7). Salt precipitation and the formation of a white layer on the surfaces of the immersed specimen can be observed, as well as salt deposition in the immersion tanks. Additionally, specimens with OPC displayed more degradation than those with SRPC. When 4% NS is added to the concrete samples, their surface appearance is significantly better than that of the specimens that don't have NS.

4.3 Compressive strength

The compressive strength test was done at the 7-day, 28-day, and 90-day ages, involving testing in the sulfate-exposed environment in addition to the curing in water for each kind of cement, and without and with NS 4% dosage. The compressive strength of specimens for two cement types without NS in a solution of water and sulfate is displayed in Figure 8. The percentage decrease in compressive strength of the mixtures with OPC without NS upon exposure to sulfates is 49.6% at 90 days, and after adding 4% of NS, it decreased to 42.6% at 90 days. For the mixtures containing SRPC without NS, the percentage decrease in compressive strength is 32% after 90 days, and after the addition of 4% NS, it decreased to 29%. This is due to the decrease in C3A content in this type of cement, which reduced the interaction with sulfates and the formation of gypsum and ettringite. On the other hand, the surface area of NS is very high, which enables it to achieve two mechanisms. The first is a filling effect, where NS fills the microscopic voids in the internal zone (ITZ), resulting in reduced porosity and a less permeable structure, thus minimizing the penetration of sulfate ions inside the concrete. The second mechanism is that NS promotes the pozzolanic reaction, accelerating hydration and rapid reaction with $Ca(OH)_2$, increasing C-S-H production and reducing the amount of CH that is converted to gypsum and ettringite in a sulfate medium (see Figure 9). Previous studies have shown that NS improves the resistance of conventional concrete to sulfate attack, but most of these studies were conducted on conventional concrete and not LWC. Therefore, compared tothe study [17], we find that the percentage decrease in compressive strength is higher. This is due to the difference in curing conditions and the type of aggregate used, as pumice aggregate is more porous, allowing greater sulfate penetration compared to the ordinary aggregate used in conventional concrete.

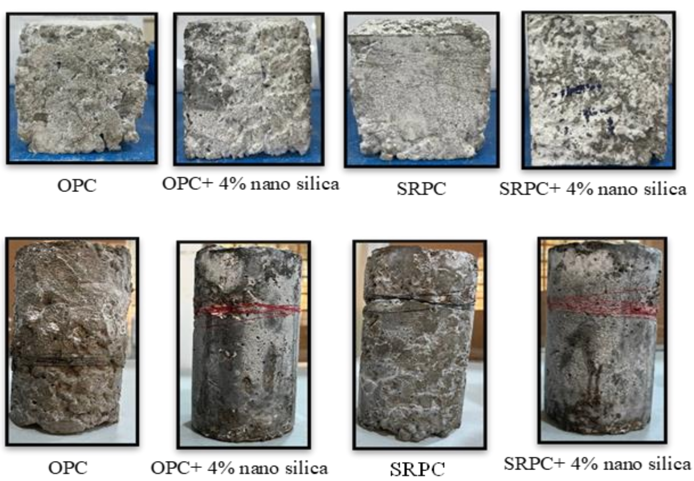
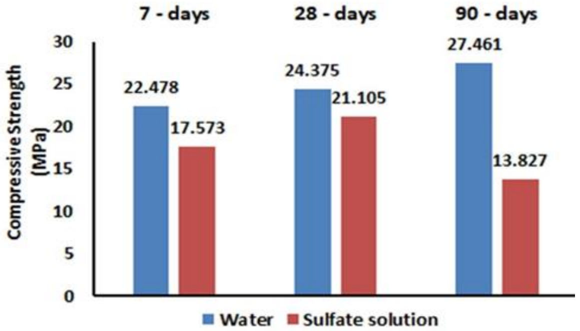
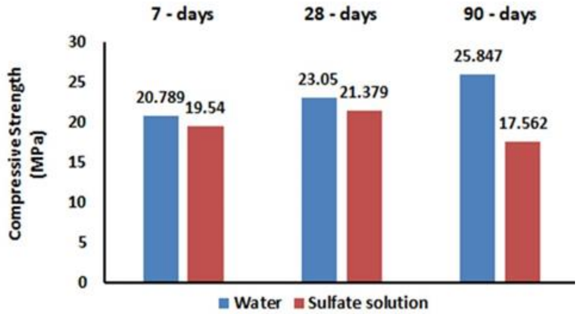


Figure 7. Concrete specimens after 28 and 90 days of immersion in sulfuric solution

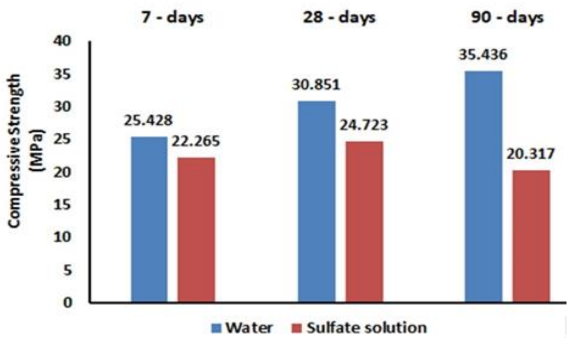


(a)

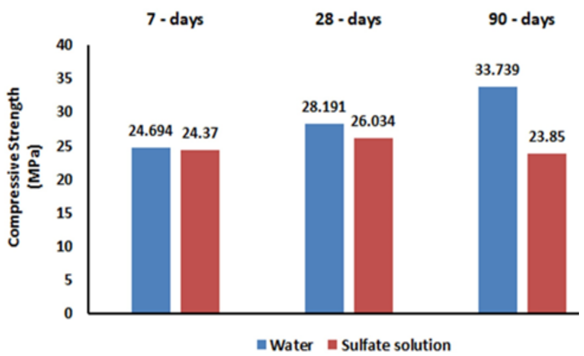


(b)

Figure 8. Compressive strength of (a) OPC and (b) SRPC concrete samples without nano-silica after 7, 28 and 90 days of curing in water and a 0.3% combined sulfate solution



(a)



(b)

Figure 9. Compressive strength of (a) OPC and (b) SRPC concrete samples with nano-silica after 7, 28 and 90 days of curing in water and a 0.3% combined sulfate solution

5. CONCLUSION

According to the findings and discussions, the following can be concluded:

1. The lightweight concrete samples containing OPC and without NS had the highest mass (10%) reduction when immersed in sulfate solution due to the high content of C3A, which increases the production of ettringite and gypsum. In the case of using SRPC, it reduced the mass loss to about 8%, which confirms that the low C3A content has a positive influence on the resistance to sulfate attack. Compared to samples with added NS, where NS demonstrated a significant role in reducing of weight of samples.
2. Visual examinations after 28 and 90 days showed the production of a soft white layer on the surface of the samples as a result of the migration of dissolved salts and their crystallization on the surface.
3. Water treatment improved the compressive strength of LWC mixes. Adding 4% NS improved the compressive strength by 31.5%, 26.6% and 29% at 7, 28 and 90 days for mixes containing OPC and by 17%, 22.3% and 30.6% at 7, 28, and 90 days for mixes with SRPC due to the formation of additional C-S-H and improving the ITZ between the paste and lightweight aggregate.
4. Mixtures impregnated with sodium and magnesium sulfate with OPC and without NS recorded a decrease in compressive strength of 19.7%, 13.35% and 49.6% at 7, 28, and 90 days, while mixes containing RSPC recorded a decrease of 18%, 7.3%, and 32% at 7, 28, and 90 days. Adding 4% of NS reduced this decrease in compressive strength. The percentage of decrease was 19%,20% and 42% at 7, 28, and 90 days % in OPC mixes and about 8%, 7% and 29% at 7, 28, and 90 days in SRPC mixes.
5. All mixtures exhibited decreased strength due to the production of gypsum and ettringite under chemical attack by sulfate solution; however, the degree of deterioration was heavily reliant on the type of cement and the number of NS. The use of SRPC provided more protection against sulfate and magnesium attack due to its low C_3A content, which limited the formation of expansive ettringite phases.
6. NS has proven its effectiveness in reducing the effect of chemical attack by reducing $Ca(OH)_2$, which reduces the formation of gypsum and ettringite, and increases its resistance to MgSO4. This means increasing its resistance to chemical attack, which confirms its effective role in enhancing durability and mechanical performance.

Future proposal: This study relied on mechanical measurements, weight loss, and visual inspections without taking into consideration the microscopic examinations, such as Scanning Electron Microscopy (SEM) or X-ray Diffraction (XRD), to visually confirm the improvement of the ITZ zone. Furthermore, the period of sulfate exposure was limited to 90 days. Therefore, future research suggests incorporating microscopic examinations and studying longer sulfate exposure periods. It is also recommended to study the effect of other types of nanomaterials, such as nano clay and alumina, on lightweight concrete.

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