Environmentally Friendly Production and Characterization of Olivine Nano-silica for Enhancing the Compressive Strength of Self-Compacted Concrete

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Abstract

This study presents the green synthesis and characterization of olivine nano-silica and investigates its influence on the compressive strength of self-compacted concrete (SCC). Olivine nano-silica (ONS), a novel and promising nanomaterial, was prepared using a cost-effective and sustainable method. The synthesized ONS was thoroughly characterized through various analytical techniques, including X-ray diffraction (XRD) and scanning electron microscopy (SEM), to assess its morphology and crystal structure. The effects of incorporating ONS into SCC were evaluated through an experimental investigation. Different proportions of ONS particles (١٪, ٢٪, and ٣٪) were added to the SCC mixtures to study its impact on the Compressive strength of SCC after ٢٨ and ٩٠ days of curing. Moreover, the effects of ONS particles were compared with the effects of commercial nano-silica (CNS) particles available in the market. The results indicate that the addition of ONS significantly influences compressive strength of SCC positively. At optimized ONS dosage (٢٪), an enhancement in compressive strength of about ٣٤,٨٪ was observed compared to the reference SCC mixture without ONS. Also, ONS particles resulted in similar even better performance compared to the CNS particles. This research provides valuable insights into the potential use of olivine nano-silica as a sustainable and effective additive for enhancing the mechanical properties of SCC. The findings have significant implications for the construction industry, where the demand for high-performance and environmentally friendly concrete materials continues to grow.

Keywords: Olivine Nano-silica; Self-compacted concrete; X-ray diffraction.

١. Introduction

The development of self-compacting concrete (SCC) represents a remarkable achievement within the construction and concrete industry, owing to its numerous advantages when compared to conventional concrete mixes. This high-performance concrete possesses the unique ability to flow smoothly without requiring manual compaction or manipulation, setting it apart from other cement-based composites. SCC excels in passing through and filling intricate spaces without experiencing segregation or bleeding issues (Faraj et al., ٢٠١٩). The enhanced passing ability and flow characteristics of SCC are attained through the incorporation of superplasticizers, which are high-range water reducers, and various mineral admixtures such as ground granulated blast furnace slag (GGBFS), metakaolin, silica fume, and fly ash. These additions ensure the mixture remains stable and cohesive during the mixing process (Deilami et al., ٢٠١٩; Faraj et al., ٢٠٢٠). SCC presents several significant benefits over conventional concrete, including faster construction times, reduced labor costs, and improved compaction, particularly in structures featuring high concentrations of rebars.

Nanotechnology has garnered widespread attention worldwide due to its exceptional capabilities across various fields. One area where it has demonstrated remarkable effectiveness is in the construction industry, specifically through the integration of nanoparticles in concrete production. Nanoparticles are materials that can manipulate and modify matter at the atomic and molecular levels, existing within a size range of ١ to ١٠٠ nm. At this scale, they exhibit unique characteristics and phenomena, akin to single atoms or molecules, as well as bulk behavior (Zhu et al., ٢٠٠٤). By incorporating these super-fine particles into concrete, significant improvements in performance have been achieved. The exceptional outcomes are attributed to the high-density continuous packings of binder ingredients facilitated by the nanoparticles (Sobolev and Ferrara, ٢٠٠٥). Previous studies have shown that the predominant type of nanoparticles (NP) used in self-compacting concrete (SCC) composites is nanosilica (NS), either in its powder or colloidal form (Faraj et al., ٢٠٢٢a). This choice is primarily due to the fact that NS is more cost-effective and simpler to produce compared to other types of nanoparticles.

In addition, the existence of C-H within concrete indicates an unstable outcome of the hydration procedure, where it engages with different minerals to generate fresh compounds within the microstructure of the concrete. As a result, scientists are actively investigating the incorporation of diverse minerals and chemical admixtures into the concrete mixture to transform C-H into a more beneficial substance like C-S-H gel, the introduction of nanoparticles (NPs) like nano-silica can also result in improved packing density and reduced porosity within the concrete matrix, leading to enhanced mechanical and physical properties of the concrete composite (Hamada et al., ٢٠٢٣). For instance, incorporating nano-silica in self-compacting concrete (SCC) can elevate its compressive strength, flexural strength, modulus of elasticity, and abrasion resistance (Al Ghabban et al., ٢٠١٨; Güneyisi et al., ٢٠١٥a; Güneyisi et al., ٢٠١٥b). Moreover, NPs contribute to the concrete composite's durability, providing better resistance against chemical attacks, freeze-thaw cycles, and carbonation (Balapour et al., ٢٠١٨). Overall, the integration of NPs into concrete composites can significantly augment their performance and render them suitable for diverse applications in the construction industry.

٢. Research Significance

Presently, a significant drawback of the majority of available nano-silica (NS) in the market is its high cost. Consequently, despite its highly beneficial impact on concrete properties, its extensive use in large-scale concrete production is severely limited due to economic constraints. However, ongoing research focuses on exploring more environmentally friendly and cost-effective methods for producing precipitated NS. This includes extracting it from natural dunite rocks and identifying waste sources from other processes. The expectation is that these efforts will lead to a more affordable NS in the future, making its application in concrete production more appealing (Lazaro et al., ٢٠١٣).
Previous research on the application of olivine nano-silica (ONS) in cement-based systems, especially self-compacting concrete (SCC), appears to be scarce. Therefore, this study aims to produce ONS particles from locally available Olivine rocks found in the Mountains of Kurdistan region. Additionally, the study seeks to investigate the impact of the locally produced ONS on the compressive strength of SCC.

٣. Experimental program

٣,١ Green synthesis of ONS particles

As previously mentioned, the NS particles commercially available in the market are relatively costly. Therefore, it is crucial to find alternatives to obtain locally produced NS particles from green methods. Moreover, from the practical point of view, the method that can give a higher amount of NS particles is more feasible.
Green synthesis of amorphous nano-silica $SiO_٢$ from the rock olivine $(Mg, Fe)_٢SiO_٤$, which was obtained from the Mountains of Sharbazher district, Kurdistan Region of Iraq was utilized by hydrothermal method. This procedure can be regarded as a cost-efficient and environmentally friendly manufacturing process for spherical amorphous NS particles with desirable particle size and surface characteristics from silicate minerals (Olivine rocks) (Lazaro et al, ٢٠١٢). Olivine occurs in mafic and ultramafic igneous rocks and as a primary mineral in certain metamorphic rocks. Olivine is one of the most common minerals on Earth.

٣,٢ Experimental procedure of ONS production

The process outlined in this section for generating olivine nano-silica (ONS) particles has been previously employed by Lazaro et al. (٢٠١٢) and Mardiana (٢٠١٧). Prior to commencing the production process, the olivine rock underwent grinding and crushing to obtain small particles. Sulfuric acid (٩٦ w/w%) was utilized as the reagent decomposition fluid, following the approach by Lazaro et al. (٢٠١٢). To eliminate sand, dust, and other impurities, the ground olivine was washed with warm water. The production process took place at a constant temperature of ٩٠°C.
In each round of production, approximately ٣٠g of ground olivine was mixed with ٢٥٠ mL of water and ٣M sulfuric acid, resulting in an olivine to acid mole ratio of nearly ١:٨. The solution was then heated for ٤ hours at ٩٠°C. During this time, a stirrer was employed to ensure the acid reagent interacted with all olivine particles, enhancing the degradation process effectively. Once the reaction was completed, the solution was allowed to cool down to room temperature, after which distilled water was added to separate the nano-silica from the waste olivine particles.
The suspended ONS particles in distilled water appeared as a white-colored solution, and a decantation process was performed to separate the ONS from the waste. This solution was subsequently collected and subjected to centrifugation at ٥٠٠٠ rpm for ١٠ minutes. The washing process was repeated three times until the washing pH ranged between ٥,٥ to ٧. Finally, the silica mud was dried in an oven at ١١٠°C until the solvent was completely evaporated (Lazaro et al., ٢٠٠٩; Lazaro et al., ٢٠١٢; Lazaro et al., ٢٠١٣; Mardiana, ٢٠١٧).

٣,٣ Materials

In this research, all concrete mixes were produced using Ordinary Portland cement (OPC) CEM I ٤٢,٥. The cement content accounted for ٧٠٪ by weight of the total binder content to create SCC mixtures. In order to enhance the stability of SCC in its fresh state, silica fume (SF) was employed as a supplementary cementitious material (SCM) alongside cement. The amount of silica fume used was ١٠٪ by weight of the total binder content for all SCC mixtures. This proportion of ١٠٪ SF was identified as the optimal ratio in previous studies for improving both the fresh and mechanical properties of SCC (Jalal et al, ٢٠١٣; AbdelAleem and Hassan, ٢٠١٨; Güneyisi et al, ٢٠١٠). Another commonly used pozzolanic material in SCC mixes is Ground Granulated Blast Furnace Slag (GGBFS). For this study, GGBFS was utilized at a quantity of ٢٠٪ of the total binder content. The decision to use ٢٠٪ of GGBFS was based on the fact that it had been the average content used in ٢٠٠ previous SCC mixtures to achieve desirable performance (Faraj et al, ٢٠٢٢b). To compare the results of ONS particles with CNS particles, hydrophilic Nano-Silica (NS) particles in powdered form were obtained from LUOYANG Company in China. These NS particles were extremely small, with a size range of about ٣٠ nm, and were found to significantly enhance the performance of SCC mixtures.

To achieve a flowable concrete mix, such as SCC, the addition of a high-range water-reducing admixture or superplasticizer is necessary. In this study, the researchers utilized a commercially available superplasticizer called Hyperplast PC٩٠٠, which was obtained from DCP Company. This particular superplasticizer is designed to have slump retention capabilities. It meets the ASTM C٤٩٤ standards and falls under TYPE G classification. The specific gravity of Hyperplast PC٩٠٠ is ١,١٢ g/cm٣, and its pH value ranges from ٥ to ٧.

The well-graded crushed coarse aggregates and river sand used in this study were sourced from the Qaladize district in the Kurdistan region. The coarse aggregates had a maximum size of ١٢,٥ mm and exhibited a water absorption rate of ١,٣٧٪. In the saturated surface dry condition (SSD), the specific gravity of the coarse aggregates was measured to be ٢,٦٧.As for the fine aggregates, their water absorption rate was found to be ١,٣٧٪, and their specific gravity in the saturated surface dry condition (SSD) was recorded as ٢,٧٧. The gradation of the employed aggregates was satisfies ASTM C٣٣ standard.

٣,٤ Self-compacting concrete mix proportions

Since the production of ONS particles was conducted on a laboratory scale. Therefore, a limited quantity of the material can be produced. Figure ١ shows a small sample of ONS particles along with the olivine rock. Due to the limitation of the available sample, a small number of samples were cast to determine the effect of ONS particles on the ٢٨ and ٩٠-day compressive strength of SCC concrete and compare it with the results of the commercially obtained nano-silica (CNS) particles . For this purpose, seven different SCC mixes were cast. Each mix contained a different amount of ONS or CNS particles such as ١٪ for the first mix, ٢٪ for the second mix, and ٣٪ for the third mix along with the ٠٪ for the control mixture. The mix proportions of the SCC mixtures made with different percentages of ONS and CNS particles are presented in Table ١.

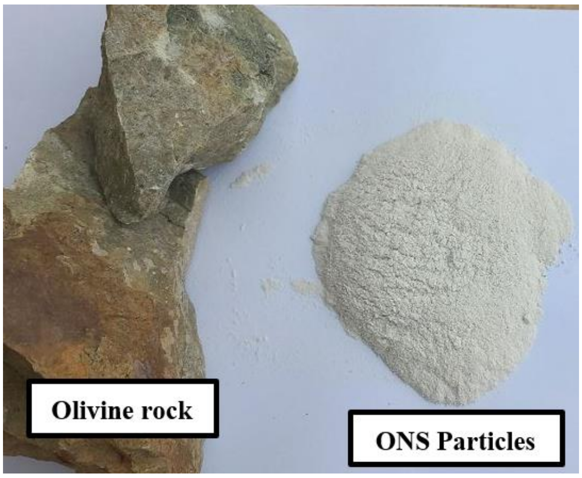

Figure ١. ONS particles sample and Olivine rock.

Table ١. SCC mix proportions made with ONS particles in kg/m^{٣}

Mix ID.	Cement	ONS Or CNS	SF	GGBFS	W/B	Water	CA	FA	SP
NS٠ (C)	٣٥٠	٠	٥٠	١٠٠	٠,٣٤	١٧٠	٨٤٣,٩	٨٧٥,٥	١١,٢
NS١	٣٤٥	٥	٥٠	١٠٠	٠,٣٤	١٧٠	٨٣٦,٥	٨٦٧,٨	١١,٢
NS٢	٣٤٠	١٠	٥٠	١٠٠	٠,٣٤	١٧٠	٨٢٩,١	٨٦٠,١	١١,٢
NS٣	٣٣٥	١٥	٥٠	١٠٠	٠,٣٤	١٧٠	٨٢١,٧	٨٥٢,٤	١١,٢

Regarding the mixing procedure ONS or CNS particles were dry-mixed with other cementitious materials before introducing to the mixer. After the mixing procedure was completed three samples for each mix were cast without any compaction or vibration. The samples were de-moulded ٢٤ hours after casting and stored in a water tank with temperature control of about ٢٣ °C. After ٢٨ and ٩٠ days of casting the samples were removed from the water and their surface was capped, as shown in Figure ٦. The compressive strength test was conducted after ٢٨ and ٩٠ days of casting. For each mixture, three samples were tested and the average of them was reported.

٣.٥ Compressive strength test

For compressive strength measurement of SCC mixtures, cylindrical samples of ϕ١٠٠ mm in diameter and ٢٠٠ mm in height with a height/diameter ratio of ٢ were tested concerning ASTM C٣٩-١٤ (٢٠١٤) using a ٣٠٠٠ KN capacity universal testing compression machine as shown in Figure ٧. The test is conducted on three samples from each SCC mix at ٢٨ and ٩٠ days.

٣,٦ Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is an effective tool for examining organic and inorganic materials on a nanoscale to micrometer (μm) scale. SEM utilizes a high magnification of up to ٣٠٠,٠٠٠x to create incredibly precise images of a wide variety of materials. SEM was utilized extensively to explore the impact of NPs on the microstructural improvement of different concrete composites over time by capturing high-resolution pictures. Quanta ٤٥٠ model instrument was used to conduct SEM tests, as can be seen in Figure ٨.

٣,٧ X-ray diffraction (XRD)

XRD usually offers data on the crystalline structure, the structure's essence, lattice parameters, grain size, and grain density of the crystalline. The utilized XRD machine in this study was PW٣٣٧٣/٠٠. The device specification is a Cu anode with a wavelength of ٠,١٥٤ nm, max: ٢,٢ kW, ٦٠ kV. The XRD test was conducted for the ONS particles obtained from Olivine rock.

٤. Results and discussion

٤,١ XRD and SEM of ONS particles

The ONS particles synthesized in this study were characterized with the techniques and tests explained in the previous section. Firstly, to determine the size and shape particles of the ONS particles, an SEM test was utilized. The SEM image of the ONS particles is shown in Figure ٢. It is obvious from the Figure that the ONS particles are in the agglomerated form in which the particles are clustered together due to high surface energy. The diameter of the ONS particles is generally smaller than ١٠٠ nm in which particles with higher and lower diameters are present. Moreover, an XRD analysis was performed to determine the different compounds present in the sample. The XRD analysis of the ONS particle sample is illustrated in Figure ٣. It can be seen from the XRD results that a peak of $SiO_{٢}$ is present in the sample, which means that a sample contained a higher percentage of $SiO_{٢}$. However, some lower peaks such as Mg and Fe in the form of impurities can also be found in the sample, which means that the ONS particles produced in this study contained small percentages of impurities

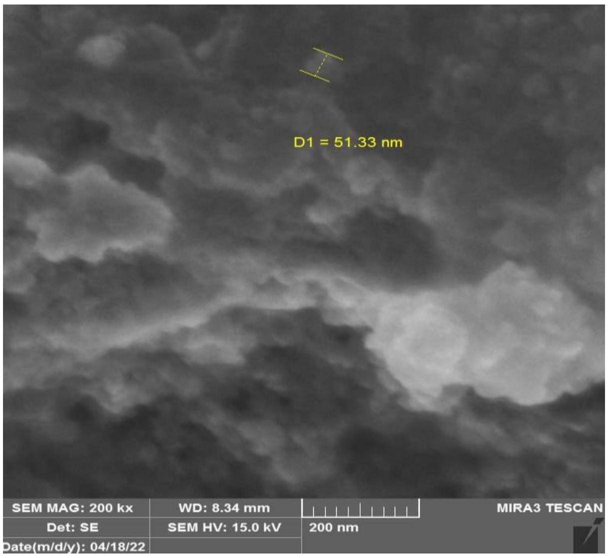

Figure ٢. SEM image of the ONS particles.

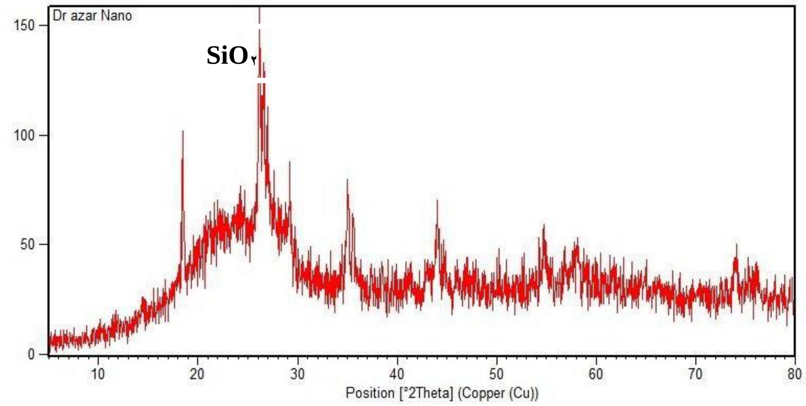

Figure ٣. XRD analysis of the ONS particles.

٤,٢ Compressive strength

The results of the ٢٨ and ٩٠-day compressive strength of SCC mixtures produced with various percentages of ONS particles are illustrated in Figure ٤. It is obvious from the results that similar to the commercial NS particles (CNS), due to the addition of ONS particles the CS of SCC mixes was considerably improved. For example, after ٢٨ days of curing, the CS of the control mixture was ٥٨,٦ MPa, this value was significantly elevated to ٧٤,٢ MPa, ٧٩ MPa, and ٧٦,١ MPa due to the utilization of ١٪, ٢٪, and ٣٪ of ONS particles, respectively. This means that the addition of ١٪, ٢٪, and ٣٪ of ONS particles caused an improvement of about ٢٦,٦٪, ٣٤,٨٪, and ٢٩,٨٪, respectively. Moreover, after ٩٠ days of curing, the compressive strength of the control mixture was ٦٣,٢ MPa. This value was also elevated to ٧٥,٨ MPa, ٨٥,٤ MPa, and ٨٠,٢ MPa after the incorporation of ١٪, ٢٪, and ٣٪ of ONS particles, respectively. The improvement percentages were about ١٩,٩٪, ٣٥,١٪, and ٢٦,٨٪ due to the incorporation of ١٪, ٢٪, and ٣٪ of ONS particles, respectively. Therefore, based on the results obtained in this study, the optimum percentage for ONS particle addition was ٢٪. Higher percentage replacement was no longer beneficial for improving the CS. The study's optimum percentage aligns with similar findings in the literature (Moghaddam et al., ٢٠٢١; Niewiadomski and Hoła, ٢٠٢٠; Hameed et al., ٢٠٢٠). Some prior research reported different optimal percentages. For instance, Beigi et al. (٢٠١٣) and Güneyisi et al. (٢٠١٥b) found that ٤٪ NS particles improved FRSCC performance.

However, considering the higher cost of NPs compared to cement, it is more feasible to optimize mixtures using lower NP percentages. The improved CS in SCCs with the addition of ONS can be attributed to various factors. Firstly, the pozzolanic reaction between ONS and C-H crystals leads to the formation of additional C-S-H gel, which is a crucial source of CS in cement-based materials. Secondly, the small size of ONS particles allows them to fill the nano voids and pores in the C-S-H gel, enhancing the density and packing of the paste's micro and nano structure (Li et al., ٢٠٠٤; Silvestre et al., ٢٠١٦). Thirdly, ONS and other nanoparticles act as nuclei, forming a strong bond with C-S-H gel particles, which improves the stability of hydration products and enhances mechanical characteristics (Beigi et al., ٢٠١٣). However, it's important to note that exceeding the optimum amount of NPs doesn't necessarily lead to further CS improvement. High NP dosage can cause agglomeration due to high surface energy, resulting in poor dispersion, formation of voids, and weak zones, ultimately reducing CS (Silvestre et al., ٢٠١٦).
To compare the results of the CS obtained due to the addition of ONS particles with CS for similar mixtures obtained due to the incorporation of CNS particles after ٢٨ and ٩٠ days of testing Figure ٥ and Figure ٦ are provided. Figure ١٣ shows the comparison between ONS and CNS mixtures after ٢٨ days of curing. The results illustrated that the CS of SCC mixtures made with ONS particles was slightly higher than those of CNS particles for all percentage additions. The ONS particles gave higher improvements of about ٢,٧١٪, ٢,٨٦, and ١,٧١٪ compared to the CNS particles for the mixtures made with ١٪, ٢٪, and ٣٪ of both NS particles, respectively. Moreover, after ٩٠ days of curing, the comparison is also reported in Figure ١٤. Negligible improvements can be found in ONS compared to CNS mixtures at the age of ٩٠ days. There were about ١,٨٪ improvements in the CS when ٣٪ of ONS particles were utilized instead of CNS particles. However, the mixture produced with ١٪ and ٢٪ ONS particles had slightly lower CS than similar mixtures made with CNS particles after ٩٠ days of curing. The above-mentioned results were crucially important to produce SCC mixtures with the NS particles which were obtained from green methods. Because these NS particles such as ONS are more cost-effective and environmentally friendly compared to the most commercially available NS particles. The failure mode of SCC mixtures made with ONS particles was also similar to the CNS particles. The shear (cone) mode of failure with brittle nature was also observed for the SCC samples made with different percentages of ONS particles, as shown in Figure ٧. Since the addition of ONS particles shifted the concrete behaviour from normal strength SCC to high strength SCC. Its mode of failure was changed to a very brittle failure with an explosion of the samples during testing.

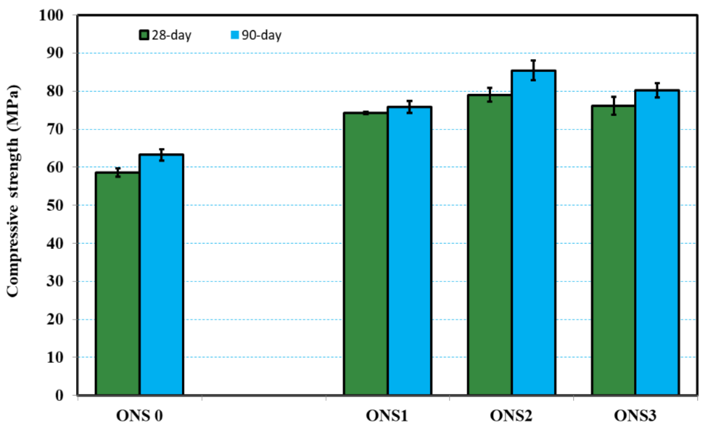

Figure ٤. CS of SCC mixtures made with various percentages of ONS particles.

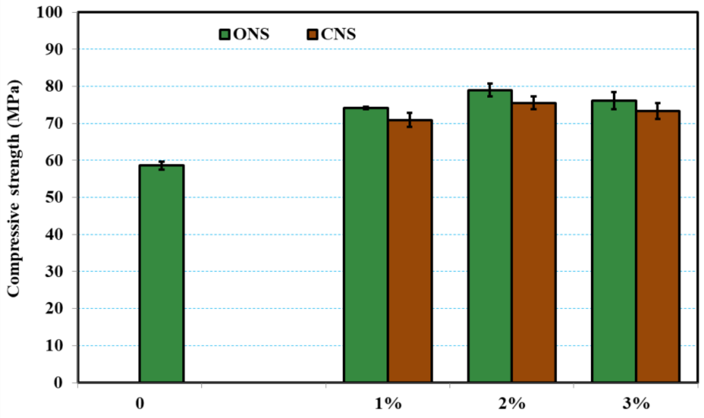

Figure ٥. Comparison of the SCC mixtures made of ONS particles and CNS particles after ٢٨ days of curing.

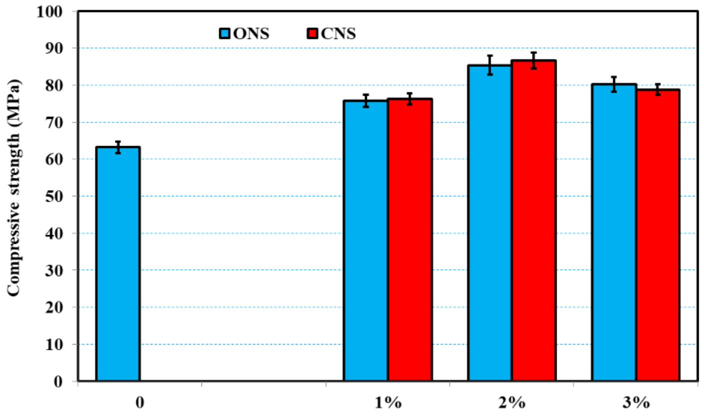

Figure ٦. Comparison of the SCC mixtures made of ONS particles and CNS particles after ٩٠ days of curing.

Figure ٧. Failure pattern of SCC mixture made with ٢٪ of ONS particles at ٢٨ days.

٥. Conclusions

Drawing conclusions based on the outcomes of this experimental investigation, the following key findings and implications can be inferred:

- The successful green synthesis of olivine nano-silica (ONS) using a cost-effective and scalable method highlights its potential as an environmentally friendly and sustainable nanomaterial for concrete applications.

- Thorough characterization of the synthesized ONS through advanced analytical techniques, such as X-ray diffraction (XRD) and scanning electron microscopy (SEM), ensures its desired morphology and crystal structure, laying the foundation for its effective use as a concrete additive.

- The incorporation of ONS into self-compacted concrete (SCC) has a significant and positive impact on the compressive strength, with a notable ٣٤,٨٪ enhancement observed at the optimized ONS dosage of ٢٪ compared to the reference SCC mixture without ONS.

- Comparative analysis with commercially available nano-silica (CNS) particles reveals that ONS performs on par or even surpasses CNS, offering a viable and efficient alternative for enhancing the mechanical properties of SCC.

- The promising results of this study open new avenues for the practical application of olivine nano-silica in the construction industry, where the demand for high-performance and environmentally friendly concrete materials is continually growing.

- The successful green synthesis of ONS and its demonstrated positive effects on SCC's compressive strength contribute to the ongoing efforts towards sustainable construction practices, aligning with global environmental goals.

- The findings of this research hold significant implications for the development of durable and resilient concrete infrastructure, promoting the adoption of greener and more efficient building materials to address the challenges of modern construction.

- Future research endeavors may focus on optimizing the ONS dosage for specific concrete mix designs, investigating its influence on other essential concrete properties, and assessing its long-term performance and durability under diverse environmental conditions, ensuring its reliable and widespread implementation in real-world construction projects.

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