Article
Mechanical Properties of High Strength Concrete Containing Nano SiO$_2$ Made from Rice Husk Ash in Southern Vietnam

Huu-Bang Tran$^1$, Van-Bach Le$^2$ and Vu To-Anh Phan$^3$*

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Abstract: This paper presents the experimental results of the production of Nano-SiO$_2$ (NS) from rice husk ash (RHA) and the engineering properties of High Strength Concrete (HSC) containing various NS contents. Firstly, the mesoporous silica nanoparticles were effectively modulated from RHA using NaOH solution, and subsequently precipitated with HCl solution until the pH value reached 3. The optimum synthesis for the manufacture of SiO$_2$ nanoparticles in the weight ratio of RHA/NaOH was 1:2.4, and the product was calcined at 550 $^\circ$C for 2 h. The EDX, XRD, SEM, TEM, FT-IR, and BET techniques were used to characterize the NS products. Results revealed that the characteristics of the obtained NS were satisfactory for civil engineering materials. Secondly, the HSC was manufactured with the aforementioned NS contents. NS particles were added to HSC at various replacements of 0, 0.5, 1.0, 1.5, 2.0, and 2.5% by the mass of the binder. The water-to-binder ratio was remained at 0.3 for all mixes. The specimens were cured for 3, 7, 28, and 56 days under 25 $\pm$ 2 $^\circ$C and a relative humidity of 95% before testing compressive and flexural strengths. Chloride ion permeability was investigated at 28 and 56 days. Results indicated that the addition of NS dramatically enhanced compressive strength, flexural strength, chloride ion resistance, and reduced chloride ion permeability compared to control concrete. The optimal NS content was found at 1.5%, which yielded the highest strength and lowest chloride ion permeability. Next, the development of flexural and compressive strengths with an age curing of 3–28 days can be analytically described by a logarithmic equation with $R^2 \geq 0.74$. The ACI code was used, and the compressive strength at t-day was determined based on 28 days with $R^2 \geq 0.95$. The study is expected to solve the redundancy of waste RHA in southern Vietnam by making RHA a helpful additive when producing high-strength concrete and contributing meaningfully to a sustainable environment.

Keywords: modulation; nano SiO$_2$; rice hush ash (RHA); high-strength concrete (HSC); chloride ion permeability

1. Introduction

Nano SiO$_2$ (NS) is well-known for its many uses in the past two decades, including in catalytic materials, dielectric materials, gas adsorbents, heavy metal ion adsorption, and inorganic carriers [1]. Besides this, it has been noted that most NS materials used in civil engineering were supplied by commercial companies, mainly from China and European countries [2]. Due to its being produced in a factory, the main qualities of commercial NS are high purity and uniformity; however, it is expensive and causes difficulty in wide use in actual construction. Thus, the current trend of locating a readily available, low-cost, silicon-rich source of materials for use as a concrete additive merits consideration and has attracted the attention academic scientific and technologists.

The rice-growing regions of the world, for example, China, India, Bangladesh, Brazil, and the far-East countries, increases daily, resulting in agricultural waste. Among them,
Vietnam is the world’s second-largest rice producer, with an estimated annual rice production of 42 billion tons. Rice husk is a by-product of rice milling, and rice husk ash (RHA) is obtained when burnt for energy production, such as for a steam boiler. The millions of tons of redundancy caused by waste RHA globally were used in specific fields, such as adsorbents, cement, catalyst, nanocomposites. It is noted that RHA is one of the most SiO₂-rich materials, with concentrations ranging from 90% to 98% in terms of volume and containing a considerable quantity of amorphous silica (85–95%) [4], making it an attractive source of raw materials for SiO₂ production [5,6]. Various methods of NS production are available and vary from expensive to cost-effective routes [7], such as sol-gel, chemical precipitation, microemulsion, and hydrothermal techniques [8].

Plain concrete has become the most-used construction material, with the broadest application range because of its high strength, additional workability, good hydrothermal stability, and plentiful raw material resources for easy mixing [9]. However, as the range of concrete uses expands, more stringent criteria for concrete characteristics (such as the compressive strength, tensile strength, flexural strength, durability, and impact resistance) are being proposed, such as those for high-rise and large-span buildings, building structures in freezing climates, and other unique applications [10,11]. Thus, the need for high-strength concrete (HSC), which is robust, sustainable, and ecologically beneficial, is increasing, because of the rapid development of infrastructure and civil engineering.

To improve the engineering properties of Portland concrete, in recent years, some researchers have used admixtures such as nanomaterials and fibers in cement-based materials to enhance its characteristics [12–16]. Regarding the use of NS particles as an additive in concrete, several studies on the addition of low-dose NS nanoparticles to replace cement in concrete mixes, aiming to improve concrete quality, have shown significantly positive results [17–21]. Due to its large specific area and intensive activity, NS particles have a high degree of pozzolanic activity in concrete. Furthermore, NS particles can function as a catalyst for pozzolanic processes, promoting the dissolution of tricalcium silicate and the production of C–S–H by providing nucleation sites for calcium silicate hydrate (C–S–H) [22,23]. Nazari and Riahi [24] used NS particles as a fractional replacement for cement. They suggested that an NS content of less than 4%, by weight, could improve the formation of C–S–H gel and refine the pore structure of the concrete; however, with a further increase in NS content of more than 4%, the strength of concrete reduced due to uneven nanoparticle dispersion. Said et al. [25] utilized colloidal NS in their experiment and revealed that a significant improvement was obtained in mixtures incorporating nano-silica in terms of reactivity, strength development, refinement of pore structure, and densification of the interfacial transition zone. Certain studies discovered that adding 0 ÷ 1.5% nano-silica particles to reinforced concrete containing polyvinyl alcohol fibers improved compressive strength, flexural strength, and fracture energies. Furthermore, tensile strength reached the greatest value at the optimum content of 2.5% nano-silica particles; however, when the concentration was larger than 2.5%, the NS was prone to self-desiccation and flocking together, resulting in microfractures and strength losses in the composite [26,27]. According to previous studies, most researchers conducted experimental results on concrete with NS, and concluded that modest dosages of NS could considerably improve the mechanical characteristics of concrete. Zhang et al. [28] investigated the influence of NS particles on the mechanical characteristics, impact resistance, chloride penetration ion and freezing–thawing resistance of concrete containing coal fly ash. In their study, NS particles were added to concrete at various contents, of 1%, 2%, 3%, 4%, and 5%, according to binder weight, and it was concluded that the compressive, flexural, and splitting tensile strengths were enhanced by 15.5%, 27.3%, and 19%, respectively. The recommended degree of NS replacement in concrete to improve chloride penetration resistance and freeze–thaw resistance was 2 ÷ 3 wt.%. Moreover, when NS was added to HSC, it had a positive impact; the compressive strength improved by 21% and 17%, respectively, above the control mixes, while the percentage of increased splitting tensile strength was about 44% and 60%, respectively, when compared to control mixes. A superplasticizer was required to
increase the workability of the concrete mixtures [29]. The HSC used in the experiment included nano SiO$_2$, micro SiO$_2$, and nano + Micro SiO$_2$, all of which were exposed to high temperatures. Subsequent studies found the following: The workability of NS concrete significantly decreased, and NS mixes require substantially more water. The amount of NS utilized was 5%, which was relatively high and increased water consumption. Additionally, most NS particles did not interact with the hydration products, which should be explored further. It is also worth noting that the NS was in an amorphous powdered condition, making mixing extremely difficult [30]. In conclusion, there was a substantial shift in the early interfacial transition zone structure when 3.0% of cement was substituted with NS. In addition, the presence of NS caused an increase in hydration heat and a decrease in CH content. Nano TiO$_2$ can also extend the time it takes for cement to hydrate. Shahbazpanahi et al. [31] reported that, with 0.5% content added to cement, a combination of 30% natural and 70% recycled coarse aggregates can produce sustainable concrete.

Concerning chloride ion permeability in concrete, recent studies have investigated the effect of Nanoparticles on chloride penetration [19,32–36]. Li [33] added nano-SiO$_2$, nano-CaCO$_3$, and multi-walled carbon nanotubes to reinforced concrete and concluded that nanomaterials significantly decrease the chloride diffusion coefficients. Zhang and Li [19] revealed the pore structure and the resistance to chloride penetration that concrete possesses by adding nano-particles to the plain concrete. Similarly, autoclaved concrete significantly reduces the porosity and increases the chloride resistance by using XRD, TG-DTG, SEM, and MIP tests. However, to date, there is no research related to penetration chloride in HSC.

Based on the literature reviews mentioned earlier, the objective of this paper focused on producing NS from waste RHA, obtained in Southern Vietnam. The Sol-gel technique was employed to produce NS. Then, the physical and chemical properties of the obtained Nano-SiO$_2$ were tested and evaluated by various techniques, such as EDX, XRD, SEM, TEM, FT-IR, and BET. Secondly, the obtained NS was used as an additive replacement in HSC with various contents. To do this, HSC was prepared with the different replacement levels of 0%, 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% according to binder weight, maintaining the original silica fume (SF) content of 8% for all mixes. The water-to-binder ratio was kept at 0.3 for all the mixes. The mechanical and durability properties of HSC, such as compressive strength, flexural strength, and chloride ion permeability, were reported. The specimens were tested at 3, 7, 28, and 56 days of curing for compressive and flexural strengths, and 28 and 56 days for chloride ion permeability. Thirdly, based on the obtained results, some correlations and experimental coefficients among the obtained data were proposed, derived from this study. The results obtained from this study are expected to elucidate how NS could successfully be produced from waste RHA, reduce the agricultural waste, and help to successfully manufacture HSC. In addition, the results should also help to use waste material in construction materials, enriching the source of building materials and contributing to protecting the environment.

2. Experimental Program
2.1. Materials
2.1.1. Ordinary Portland Cement

Characteristics of the Ordinary Portland cement (PC40) utilized in this investigation, manufactured in Vietnam, as well as the physical properties and chemical compositions, are provided in Tables 1–3.

| Table 1. Chemical compositions (%) |
|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|-----------------------------|
| SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO | MgO | Na$_2$O | K$_2$O | SO$_3$ |
| 21.65 | 5.25 | 3.42 | 65.13 | 0.06 | 0.25 | 0.72 | 0.18 |
Table 2. Composition of minerals (%).

| Mineral | Composition (%) |
|---------|----------------|
| C₃S     | 51.74          |
| C₂S     | 24.20          |
| C₃A     | 8.16           |
| C₄AF    | 10.35          |

Table 3. Physical properties of OPC.

| Specific Gravity | Setting Time (min) | Compressive Strength (MPa) | Flexural Strength (MPa) |
|------------------|--------------------|-----------------------------|-------------------------|
|                  | Initial Setting    | Final Setting               | 3 Day       | 28 Day      | 3 Day   | 28 Day   |
|                  |                    |                             | 28          | 28          | 28      | 28       |
| 3.1              | 128                | 250                         | 28.5        | 55.7        | 5.8     | 8.6      |

2.1.2. Fine Aggregate

River sand is used as an aggregate in this investigation. Tables 4 and 5 detail the physical characteristics and sieve analysis, respectively, following ASTM C33 [37] and ASTM C29 [38].

Table 4. Vietnam is Dong Nai river sand properties.

| Physical Properties of River Sand | Fineness Modulus | Water Absorption (%) | Specific Gravity | Bulk Unit Weight of Sand (g/cm³) | Moisture Content (%) |
|-----------------------------------|------------------|----------------------|------------------|----------------------------------|----------------------|
|                                   | 2.5              | 0.87                 | 2.66             | 1.732                            | 2.0                  |

Table 5. Grain size distribution of fine aggregate.

| Percentage Passes of Fine Aggregates | Sieve Sizes | 4.75 mm | 2.36 mm | 1.18 mm | 600 µm | 300 µm | 150 µm |
|-------------------------------------|-------------|---------|---------|----------|--------|--------|--------|
| Cumulative (%) Passed               |             | 95–100  | 80–100  | 50–85    | 25–60  | 5–30   | 0–10   |
|                                    |             | 100     | 91.97   | 81.76    | 57.88  | 12.07  | 5.36   |

2.1.3. Coarse Aggregate

The coarse aggregate was a crushed stone collected from a local quarry and initially formed of basalt stone. The physical properties of coarse aggregate are presented in Table 6. The grain size distribution of coarse aggregate with a maximum dominant of 9.5 mm was shown in Table 7.

Table 6. Coarse aggregate property.

| D_{max} (mm) | Water Absorption (%) | Specific Gravity | Bulk Unit Weight (g/cm³) | Moisture Content (%) |
|--------------|----------------------|------------------|--------------------------|----------------------|
| 9.5          | 0.87                 | 2.78             | 1.613                    | 0.48                 |

Table 7. Grain size distribution of coarse aggregate.

| Percentage Passes of Coarse Aggregate | Sieve Size | 12.5 mm | 9.5 mm | 4.75 mm | 2.36 mm | 1.18 mm |
|---------------------------------------|------------|---------|--------|---------|---------|---------|
| Percentage Passing (ASTM C33)         | 100        | 85–100  | 10–30  | 0–10   | 0–5    |
| Cumulative (%) Passed                 | 100        | 92.5    | 10     | 5      | 2.5     |
2.1.4. Superplasticizers

Sika Viscocrete 3000-20 is a superplasticizer provided by the Sika Group. Its exceptional high water reduction capacity allows for good fluidity while retaining optimal adhesion of the mixture. According to TCVN 8826:2011 [39], this admixture is suitable, with the established criteria, for chemical additions to concrete. Sika Viscocrete 3000-20, a 3rd generation polymer-based high-tech superplasticizer with excellent porosity and simple permeability of concrete, was utilized in the experiment.

2.1.5. Silica Fume

Silica Fume (SF) was provided by a local commercial company, and the chemical composition is reported in Table 8, following ASTM C1240-04 [40]. The specific gravity of SF is 2.2. SF is an amorphous and highly reactive pozzolan.

Table 8. Chemical composition of SF.

| SiO$_2$ | Fe$_2$O$_3$ | CaO | MgO | Na$_2$O | C    | K$_2$O | Al$_2$O$_3$ | Other Substances |
|---------|-------------|-----|-----|---------|------|--------|-------------|------------------|
| 95.38   | 0.0063      | 0.13| 0.37| 0.28    | 0.007| 1.53   | 0.20        | 3.859            |

2.1.6. Nano SiO$_2$ Produced from Rice Husk Ash

The RHA used in this study was provided by a local commercial company in Southern Vietnam. RHA is a by-product material produced from manufacturing puffed rice, containing a large amount of iron oxide and silica, and the chemical compositions of rice husk. To obtain NS, RHA was first ground and sieved by a 2.0 mm sieve; the passing was collected and dried at 105 °C until a constant mass was reached. Then, 100 g RHA was added into a 1 L cup containing 6M NaOH solution, then stirred, swelled and heated for 4 h at 100 °C. To collect the solution, 4M HCl was added until the solution reached the value of pH~3. Next, the solution was filtered by distilled water and ethanol, then the solution was dried at 100 °C overnight and heated at 550 °C for 2 h to obtain NS. The efficiency of the NS-splitting process is determined by weighing the final product in 100 g of RHA, where more than 85% was found. The detailed properties of NS are presented in the following sections. Figure 1 illustrates the procedure of producing NS from RHA.

Figure 1. The procedure of producing Nano-SiO$_2$ from RHA.
2.2. Mix Proportions and Sample Preparation

In theory, while developing HSC components using NS, it is crucial to ensure that the necessary requirements, such as concrete strength, mix flexibility, and material, are met.

2.2.1. Design Standards and Techniques

The study used the method of ACI 211.4R-08 [41] to design the HSC component. The ACI method: The component-designing phases of HSC utilizing NS were carried out according to the manufacturer’s specifications. The slump of the concrete mixture was modified to ensure the needed slump.

2.2.2. Mix Proportions

The ACI specification was used to calculate and design the concrete composition with a specific strength of 60 MPa. SF was used in concrete to improve the strength and reduce the cement content [42]. The original binder was made of 92% cement and 8% SF for the control mix, referring to previous studies [42–44]. Then, NS was utilized in various ratios in the gradation components, including 0.5%, 1.0%, 1.5%, 2.0%, and 2.5% of the total quantity of the binder. The water-to-binder ratio was kept at 0.3 for all the mixes. The mix proportion of six mixes is presented in Table 9. It can be seen that the superplasticizer (SP) gradually increased to control the slump value of mixtures at 4 ± 1 cm.

Table 9. Mix proportions and quantities for HSC.

| Mix Code | C (kg) | S (kg) | CA (kg) | SF (kg) | NS (%) | SP (%) | Water (kg) | W/B |
|----------|--------|--------|---------|---------|--------|--------|------------|-----|
| NS0.0    | 485.46 | 685.76 | 1048.45 | 42.21   | 0.00   | 4.37   | 158.76     | 0.30|
| NS0.5    | 482.82 | 684.83 | 1048.45 | 42.21   | 0.50   | 5.31   | 158.76     | 0.30|
| NS1.0    | 480.18 | 683.90 | 1048.45 | 42.21   | 1.00   | 6.24   | 158.76     | 0.30|
| NS1.5    | 477.55 | 682.98 | 1048.45 | 42.21   | 1.50   | 7.16   | 158.76     | 0.30|
| NS2.0    | 474.85 | 682.10 | 1048.45 | 42.21   | 2.00   | 8.07   | 158.76     | 0.30|
| NS2.5    | 472.27 | 681.14 | 1048.45 | 42.21   | 2.50   | 8.97   | 158.76     | 0.30|

1 C—Cement, 2 S—Sand, 3 CA—Coarse Aggregate, 4 SF—nano SiO$_2$, 5 NS—Silica Fume, 6 SP—Superplasticizer, 7 W/B—Water/Binder (cement + SF + NS).

2.2.3. Specimen Preparation and Testing Procedures

NS has a high surface area and nanoscale particle size, and is difficult to disperse in concrete mixes. The following steps are experimental mixing procedures for making homogeneous, stable concrete, as presented in Figure 2. There are five steps to mixing the sample, including:

Step 1: NS was combined with 60% water and vigorously stirred to evenly distribute the NS particles; Step 2: A combination of sand, crushed stone, cement, and silica fume was mixed for three minutes; Step 3: A total of 20% water was added to the sand, crushed stone and cement mixture, and stirred thoroughly for one minute; Step 4: the mixture containing NS and 60% water in step 1 was added to the Step 3 mixture and mixed for minutes; Step 5: The solution containing the remaining 20% water and superplasticizer was incorporated in the Step 4 mixture for 3 min until homogeneous; Step 6: Mixer was temporarily stopped for 2 min to enable the superplastic ingredient to react, which will improve the result; Step 7: The mixture was continuously mixed for another 3 min to avoid slumps and guarantee homogeneity.

A 60-L mixer was utilized to mix the composition. A compressive strength test on a cylindrical specimen with a dimension of 150 mm × 300 mm (d × h), a bending test with a beam of 150 mm × 150 mm × 600 mm, and a chlorine ion permeability with a specimen of 100 mm × 200 mm (d × h) are among the available test specimens. The inside surface of the
mold should be smooth, clean, and lubricated before sampling. Samples were compacted using a vibrator with a frequency of 2800 ÷ 3000 rpm and an amplitude of 0.35 ÷ 0.5 mm. Then, they were cured in a room at 25 ± 2 °C for a minimum of 24 h. Finally, the molds were removed and soaked in water. The compressive and flexural strengths were tested at 3, 7, 28, and 56 days. The chloride ion permeability was conducted at 28 and 56 days. All the tests were conducted in triplicate with specimens.

2.2.3. Specimen Preparation and Testing Procedures

Experiments were conducted at the Ho Chi Minh City University of Technology’s Laboratory of Building Materials LAS-XD 238-Research Center for Industrial Technology and Equipment, Vietnam. Compressive strength and flexural tensile strength tests are performed according to ASTM C39 and ASTM C78 [45], respectively, after molding and curing. The load incensement speed is 0.3 MPa/s, and the testing instrument is a San 3000 electronic compressor with a maximum load of 3000 kN, as shown in Figure 3.

2.2.4. HSC Compressive and Flexural Strength Tests

The chlorine ion permeability test was conducted at the Construction Materials Laboratory-LAS-XD 143 Southern Institute of Water Resources Research, Vietnam. The specimens with 100 mm × 200 mm cylinders were used for each kind of concrete to evaluate the chloride ion permeability at 28 days and 56 days. The ASTM C1202 [46] was applied to determine concrete’s resistance to chloride ion permeability, as plotted in Figure 4.

| Mix Code | C (kg) | S (kg) | CA (kg) | SF (kg) | NS (kg) | SP (kg) | Water (kg) | W/B |
|----------|--------|--------|---------|---------|---------|---------|------------|-----|
| NS0.0    | 485.46 | 685.76 | 1048.45 | 42.21   | 0.00    | 4.37    | 0.30       |     |
| NS0.5    | 482.82 | 684.83 | 1048.45 | 42.21   | 0.50    | 5.31    | 0.30       |     |
| NS1.0    | 480.18 | 683.90 | 1048.45 | 42.21   | 1.00    | 6.24    | 0.30       |     |
| NS1.5    | 477.55 | 682.98 | 1048.45 | 42.21   | 1.50    | 7.16    | 0.30       |     |
| NS2.5    | 472.27 | 681.14 | 1048.45 | 42.21   | 2.50    | 8.97    | 0.30       |     |

Figure 2. Sample testing of materials.

Figure 3. Compressive and flexural strength tests.

Figure 4. Chloride ion permeability of HSC.
3. Results and Discussions

3.1. Modulation and Characteristics of Nano SiO$_2$

3.1.1. Nano-SiO$_2$ Modulation

As shown in Figure 5, RHA was the burnt waste of the steam boiler, obtained from a factory located in Long An province, southern Vietnam. The factory burnt around 20 tons of rice husk and produced approximately 4 tons of RHA per day. This study’s first aim was to find new additives for civil engineering materials based on the waste obtained by RHA. As discussed in the earlier section, NS is a material that can potentially be extracted from RHA. The Sol-gel technique was proposed to produce NS materials with the cheapest cost and most potent tool [47].

3.1.2. Energy-Dispersive X-ray Spectroscopy (EDX)

EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample. The principle of this technique is based on an interaction between some source of X-ray excitation and a sample. Figure 7 plotted the result of the EDX spectrum. The results indicated that the NS particles were composed of Si and O, with 28.78%...
and 57.92%, respectively. The Si-O ratio was found to be approximately 1:2. Furthermore, the carbon atom, C, was also obtained due to the incomplete combustion of RHA. The obtained results also indicated that the process of preparing Nano-SiO$_2$ was pure, and suitable for use as an adsorbent and other related applications, especially for additives in civil engineering.

![Figure 6. Process of nano SiO$_2$ (NS) preparation.](image)

(a). RHA after grinding and drying.  
(b). RHA dissolved with 6M NaOH.  
(c). Filtering.  
(d). Solution after filtering.  
(e). Gel solution after dissolving solution with HCl.  
(f). Nano SiO$_2$.  

3.1.3. X-ray Diffraction (XRD)

X-ray diffraction (XRD), a versatile and nondestructive analytical technique, was used to quickly obtain detailed phase and structural information of crystalline materials. XRD is a technique that employs X-ray beams to generate diffraction maxima and minima on
solid crystal surfaces owing to the periodicity of the crystal structure. The method of X-ray diffraction (commonly shortened to X-ray diffraction) is used to study the structure of solids, materials, and other objects. In this study, the specimens were tested at the Institute of Applied Materials Science, Vietnam. The result of the XRD pattern of the Nano-SiO$_2$ sample is plotted in Figure 8. It can be seen from this figure that the sample mainly contains the SiO$_2$ crystalline phase of monoclinic lattice, and the peak result corresponds to an angle of 2$\theta$, about 19.76°. Furthermore, Figure 8 also indicated that the sample contains not only crystalline phase SiO$_2$, but also a little amorphous SiO$_2$.

![Figure 7. Result of the EDX analysis.](image1)

![Figure 8. Test results for X-ray diffraction.](image2)

3.1.3. X-ray Diffraction (XRD)

X-ray diffraction (XRD), a versatile and non-destructive analytical technique, was used to quickly obtain detailed phase and structural information of crystalline materials. The method of X-ray diffraction is based on the Bragg equation: $2d \sin \theta = n \lambda$, where $d$ is the interplanar spacing, $\theta$ is the diffraction angle, $n$ is an integer, and $\lambda$ is the wavelength of the X-rays. In this study, the specimens were tested at the Institute of Applied Materials Science, Vietnam. The result of the XRD pattern of the Nano-SiO$_2$ sample is plotted in Figure 8. It can be seen from this figure that the sample mainly contains the SiO$_2$ crystalline phase of monoclinic lattice, and the peak result corresponds to an angle of 2$\theta$, about 19.76°. Furthermore, Figure 8 also indicated that the sample contains not only crystalline phase SiO$_2$, but also a little amorphous SiO$_2$.

![Figure 8. Test results for X-ray diffraction.](image2)

3.1.4. Scanning Electron Microscope (SEM)

SEM is an electron microscope that scans a material with a concentrated stream of electrons to create a picture. When the electrons contact the atoms in a sample, they produce various signals that may be detected and include information about the sample, such as surface topography and composition. In most cases, the electron beam is scanned in a scanning field, and the position of the beam is combined with the received signal to form a picture. SEM has a resolution of more than 1 nanometer. Specimens can be examined in various environments, including high vacuum, low vacuum, moist conditions, and a wide range of temperatures. A picture depicting the surface is created by scanning the sample and detecting secondary electrons. In this work, the NS specimen was tested by SEM at the National Institute of Hygiene and Epidemiology, in Vietnam, as shown in Figure 9.
3.1.5. Transmission Electron Microscopy (TEM)

The nano SiO$_2$ sample utilized in the study was analyzed using TEM at Vietnam National Institute of Hygiene and Epidemiology, Vietnam. Figure 10 shows the TEM image of NS. Looking at the data plotted in Figures 9 and 10, an apparent crystalline grain, microscopic particles (about 10 to 15 nm), and relatively uniform dispersion were obtained.

3.1.6. Brunauer Emmett Teller (BET)

The BET theory seeks to explain the physical adsorption of gas molecules on a solid surface, and serves as the foundation for a crucial analytical approach to determining the specific surface area of materials. The nano SiO$_2$ materials used in the study have a surface area of about 258.3 m$^2$/g, as plotted in Figure 11. Furthermore, the results indicated that the NS was found to be pure and appropriate for use as an adsorbent and for other related applications, particularly as a civil engineering additive. The physical properties of NS made from RHA are presented in Table 10.

Similarly, the characteristics of NS were also obtained from the findings of the previous studies [48,49]. A crucial aspect of the NS material made from the RHA is its excellent absorption properties based on the characteristics mentioned earlier. This facilitates the mineralization process when used as an additive for concrete.

3.2. Mechanical Properties HSC Added with NS

3.2.1. Compressive Strength and Flexural Strengths at Early Curing Age

Firstly, a series of compressive and flexural tests were conducted in this study. The curing age was chosen as 3 days. Six NS contents replaced cement at various amounts such as 0, 0.5%, 1.0%, 1.5%, 2.0%, 2.5%. The 0% NS concrete sample was known as the control
specimen. Figures 12 and 13 present the compressive and flexural strength results for different replacement levels of NS, ranging from 0 to 2.5%, by weight. Looking at the data presented in these figures, the NS significantly improves the compressive and flexural strengths. The compressive and flexural strengths are in the range of 48.15–58.25 MPa and 5.68–6.77 MPa, respectively. This demonstrates that NS has a greater impact on flexural strength than compressive strength in the HSC combination. For instance, the flexural and compressive strengths of concrete grow considerably with the NS ratio from 0 to 1.5%, reaching 19.19% and 20.97%, respectively. Certain studies [50–55] also confirmed the tendency of this increase in strength. A previous study reported by Naji Givi et al. [51] found that concrete with an NS component of 1.0 ÷ 1.5% has a higher bending strength than control concrete. If the NS particle is participation is near to 2%, however, the concrete strength reduces. The explanation for this phenomenon is that the primary causes for the increased concrete strength were the pozzolanic reaction and the nano-filling effect [56]. NS particles, in particular, may interact with water molecules in the concrete mixture and produce silanol groups because of their enormous specific surface areas and ultra-high reactivity (Si-OH). Si-OH then interacts with Ca^{2+} in calcium hydroxide (Ca(OH)$_2$) crystals to produce a C-S-H gel [57]. Furthermore, the unreacted NS particles scatter in smaller areas and fill the vacancy, refining the pore structure and increasing the concrete’s compactness. Excess NS absorbs the water originally required for cement hydration, resulting in insufficient cement hydration and decreasing the strength of the concrete [58]. NS particles have extremely large specific surface areas, and when the added NS exceeds the nanoparticles, they have strong water absorption [57]. The results obtained from this indicated that the appropriate amount of NS content was 1.5%, which significantly improves the compressive and flexural strengths of HSC.

![BET coordinates of SiO2 sample plotted on a line.](image)

**Table 10.** Physical properties of NS made from RHA.

| Properties                  | Value  |
|-----------------------------|--------|
| Average particle size (nm)  | 15 ± 3 |
| Specific surface area (m$^2$/g) | 258.3  |
| Specific gravity (g/cm$^3$) | 2.2    |
| Apparent density (g/L)      | 52     |
| pH value                    | 3      |
reactivity (Si-OH). Si-OH then interacts with Ca\textsuperscript{2+} in calcium hydroxide (Ca(OH)\textsubscript{2} crystals to produce a C-S-H gel \[57\]. Furthermore, the unreacted NS particles scatter in smaller areas and fill the vacancy, refining the pore structure and increasing the concrete’s compactness. Excess NS absorbs the water originally required for cement hydration, resulting in insufficient cement hydration and decreasing the strength of the concrete \[58\]. NS particles have extremely large specific surface areas, and when the added NS exceeds the nanoparticles, they have strong water absorption \[57\]. The results obtained from this indicated that the appropriate amount of NS content was 1.5%, which significantly improves the compressive and flexural strengths of HSC.

Figure 12. Compressive strength of HSC with various NS contents.

Figure 13. Flexural strength of HSC with various NS contents.

Figures 14 and 15 present the development of the compressive strength with a curing age in a range of from 3 to 56 days for various NS ratios of 0, 0.5%, 1.0%, 1.5%, 2.0%, 2.5%. In general, a longer HSC curing age offers a higher compressive strength due to its formation through chemical condensation before the final step of gel hardening. Figure 14 shows that the 28-day specimens containing 0%, 0.5%, 1.0%, 1.5%, 2.0%, 2.5% NS have mean compressive strengths of 71.25 MPa, 74.65 MPa, 76.75 MPa, 81.25 MPa, 75.24 MPa, 73.45 MPa, respectively. Similarly, Figure 15 shows that the 28-day specimens containing 0%, 0.5%, 1.0%, 1.5%, 2.0%, 2.5% NS have mean flexural strengths of 7.05 MPa, 7.21 MPa, 7.35 MPa, 7.67 MPa, 7.46 MPa, 7.26 MPa, respectively. In general, looking at the data plotted in Figures 14 and 15, the development, compressive and flexural strengths with an age curing of 3–28 days can be analytically described by a logarithmic equation, Equation (1), as follows:

\[
f_{ci} = a \times \ln(t) + b
\]  

(1)
where \(a\) and \(b\) are the experimental coefficients; \(t\) is curing age, days; \(f_{ci}\) is the compressive strength at the curing age of \(t\)-day, MPa.

![Compressive strength of HSC with curing age.](image1)

**Figure 14.** Compressive strength of HSC with curing age.

![Flexural strength of HSC with curing age.](image2)

**Figure 15.** Flexural strength of HSC with curing age.

In addition, the coefficients of determination, \(R^2\), are found to be greater than 0.95 for compressive strength, which confirms that the logarithmic equations are highly reliable when predicting the compressive strength development of HSC with a curing age of 7–28 days. However, the coefficients of determination, \(R^2\), are greater than 0.74 for flexural strength, which should be considered with further specimens.

Figures 14 and 15 also indicated that adding NS to HSC significantly enhanced the compressive strength of concrete at an early age, from 3 to 7 days. An NS content of 1.5% yields the best compressive strength value, irrespective of the curing period. At a 1.5% content of NS, the increase in compressive strength at 3, 7, 14, and 28 days of curing was...
20.8%, 19.5%, 14.04%, and 12.06%, compared to the control sample. Similarly, the flexural strength of specimen containing 1.5% NS at a curing time in the range of 3–56 days increased by 19.19%, 16.43%, 8.79%, and 8.12% compared to that of control concrete.

The 28-day compressive strength was preferential for use in practical civil engineering. Based on the obtained data, this study used ACI [59], Equation (2), to determine the compressive strength at t-day based on 28 days:

\[
f_{ci} = \frac{t}{\alpha + \beta t} f'_c
\]  

where \(\alpha\) and \(\beta\) are the experimental coefficients; \(t\) is curing age, days; \(f_{ci}\) is the compressive strength at the curing age of \(t\)-day, MPa; \(f'_c\) is the compressive strength at 28-day.

The non-linear regression method was analyzed based on the experimental data. The values of \(\alpha\), \(\beta\), and \(R^2\) were shown in Table 11.

| Mix     | NS Content | \(\alpha\) | \(\beta\) | \(R^2\) |
|---------|------------|------------|-----------|---------|
| NS0.0   | 0.0        | 1.78       | 0.91      | 0.95    |
| NS0.5   | 0.5        | 1.48       | 0.93      | 0.97    |
| NS1.0   | 1.0        | 1.41       | 0.92      | 0.98    |
| NS1.5   | 1.5        | 1.46       | 0.93      | 0.97    |
| NS2.0   | 2.0        | 1.15       | 0.94      | 0.98    |
| NS2.5   | 2.5        | 1.14       | 0.93      | 0.98    |

3.2.2. Effect of NS Content on Strength Development

The compressive and flexural strengths, both without and with NS contents, are presented in Figures 16 and 17. In general, with an NS content in a range of 0–2.5%, the developed compressive and flexural strengths follow with a parabolic function. At 28 days of curing, the compressive and flexural strengths were in the range of 71.25–81.25 MPa and 7.05–7.67 MPa, respectively. As seen in these figures, the strengths significantly increased when NS content increased from 0 to 1.5%; however, the strength decreased with a further increase in NS content. Thus, the appropriate NS content was to be found 1.5%. These results were similar to the previous finding reported by Zhang et al. [26], who investigated polyvinyl alcohol fiber content and NS particles in terms of flexural strength; however, the optimum NS content was revealed to be 1.2%. As with the current results, Zhang et al. [28] indicated that the NS replacement was in the range of 2–3%; the compressive strength, splitting tensile strength, and flexural strength obtained the highest values in coal fly ash concrete. There are two reasons for the improvement in HSC strength, including the pozzolanic reaction and nano-filling [56]. In this study, the SF and NS react with Ca(OH)$_2$ to form CaSiO$_3$, which noticeably improves the microstructure of the mixtures [26]. Additionally, NS particles are smaller than cement particles, and can effectively enhance the interfacial structure properties. When NS content is greater than 1.5%, the sizeable molecular force was beyond the optimum content for NS addition in cementitious composites, which tend to flock and reduce strength, as explained by Abbasi et al. [60].

3.2.3. Correlation between Compressive Strength and Flexural Strength

The flexural strengths of concrete were established based on the compressive strengths obtained for different countries. Table 12 presents some empirical formulations obtained for plain concrete [61].

Based on the data obtained in this study, an empirical formulation has been established between flexural strength and compressive strength, in Equation (3), as follows:

\[
f_{fr} = k \sqrt{f'_c}
\]
where $k$ is the values presented in Figure 18, which depends on the NS content and curing age.

![Figure 16. Compressive strength of HSC with NS content.](image1)

![Figure 17. Flexural strength of HSC with NS content.](image2)

| No | Country      | Standard  | Formulation          |
|----|--------------|-----------|----------------------|
| 1  | India        | IS: 456-2000 | $f_{r} = 0.7\sqrt{f_{c}}$ |
| 2  | USA          | ACI       | $f_{r} = 0.62\sqrt{f_{c}}$ |
| 3  | New Zealand  | NZS-3101  | $f_{r} = 0.60\sqrt{f_{c}}$ |
| 4  | Europe       | EC-02     | $f_{r} = 0.201f_{c}$   |
| 5  | Britain      | BS-8110   | $f_{r} = 0.60\sqrt{f_{c}}$ |

Table 12. Empirical formulation between flexural strength and compressive strength of plain concrete [61].

where $f_r$ is flexural strength at 28-day, MPa; $f_c$ is cube compressive strength at 28-day, MPa; $f'_c$ is the cylinder compressive strength, MPa.
3.2.3. Correlation between Compressive Strength and Flexural Strength

Based on the data obtained in this study, an empirical formulation has been established between flexural strength and compressive strength, in Equation (3), as follows:

\[ f_\text{flex} = k \times f_\text{comp} \]

where \( f_\text{flex} \) is the flexural strength at 28-day, MPa; \( f_\text{comp} \) is the cylinder compressive strength, MPa; \( k \) is the values presented in Figure 18, which depends on the NS content and curing age.

The flexural strengths of concrete were established based on the compressive strengths obtained for different countries. Table 12 presents some empirical formulations published between flexural strength and compressive strength, in Equation (3), as follows:

| Country Standard Formulation | Corresponding Empirical Formulation |
|-----------------------------|-------------------------------------|
| 1 USA ACI 501-10             | 3-day flex strength = k \times 3-day compress strength |
| 2 USA ACI 501-10             | 7-day flex strength = k \times 7-day compress strength |
| 3 New Zealand NZS-3101       | 3-day flex strength = k \times 3-day compress strength |
| 4 Europe EC-02               | 28-day flex strength = k \times 28-day compress strength |
| 5 Britain BS-8110            | 56-day flex strength = k \times 56-day compress strength |

3.3. Chloride Ion Penetration

Figure 19 presents the results of the permeability of chloride ions with various NS contents. It indicated that when the number of NS particles in the concrete increases, the chloride penetration resistance of the concrete gradually improves. When the percent substitution of NS increases, the chloride ion permeability of the concrete first drops, then increases. The data presented in Figure 19 indicated that the permeability values of chloride ions are in the range of 361–882 C and 264–678 C for 28 and 56 days of testing, respectively, irrespective of the NS content used. The chloride ion permeability was at its lowest when the NS replacement amount was 1.5%. The HSC containing 1.5% NS had the best anti-chloride ion penetration capabilities, of 59% and 61%, respectively, for 28 and 56 days, compared to the control sample. The chloride ion permeability of concrete increased gradually as the replacement level of NS climbed to 2.0% and 2.5%, respectively, but was still reduced by 38% and 25%, respectively, compared to the control concrete at 28 days. The tendency of reducing the chloride ion permeability was also reported in many previous studies [56,62]. This phenomenon can be explained by NS being a nanoscale substance that may change hazardous pores into safe ones and lower the porosity of concrete. When added to the concrete as a nanoscale material, with an average particle size smaller than that of cement in concrete, it can prevent the formation of concrete pores, refine pore size, and make concrete microstructures denser and more homogeneous. Furthermore, the NS in the cement matrix can effectively block or cut off capillaries in the concrete, resulting in tortuosity and more disconnected transport channels, which improve the concrete sample’s chloride permeability resistance [18,28]. As previously stated, the NS particles will agglomerate and become unable to disperse evenly in the cement matrix after being introduced to the mixture at larger replacement ratios of NS, meaning that the chloride ion permeability will be lower than that of the concrete with the optimal threshold NS.

Figure 18. K-value with various NS contents.

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Figure 19. Permeability of chloride ions with various NS contents after 28 and 56 days.

4. Conclusions

This study presents experimental research on Nano-SiO$_2$ modulation from the waste RHA, mechanical engineering properties, and chloride ion permeability of HSC containing various Nano-SiO$_2$ contents. Based on the obtained data, the following conclusions can be drawn.

The Nano SiO$_2$ was produced from RHA in southern Vietnam using the sol-gel method. Specific techniques have evaluated the physical and chemical properties of NS. EDX spectroscopy data indicated that SiO$_2$ has a primary atomic composition of Si (28.78%) and O (57.92%), with a Si:O atom ratio of about 1:2. The XRD results revealed that NS material was mainly crystalline, and amorphous SiO$_2$ was mixed in with the crystalline SiO$_2$ phase in the sample.

BET methods found the specific surface area of nano SiO$_2$ to be about 258.3 m$^2$/g. SEM and TEM techniques showed that the NS material made from rice husk ash includes tiny particles, ranging from approximately 10 to 15 nm; SiO$_2$ is in the form of crystals, made up of numerous microscopic particles that cluster together to create porous SiO$_2$ blocks. Based on the obtained results, it can be concluded that the NS from RHA could be applied as a suitable application as a binder for building materials, enriching the source of building materials and contributing to protection of the environment.

HSC containing NS contents ranging from 0.5% to 2.5% had good performances in terms of its compressive and flexural strengths. The addition of NS to mix significantly improved the strength of concrete at the short-term curing age of 3 days compared to longer curing ages. The compressive and flexural strengths reached their highest values with the NS replacement level of 1.5%. HSC containing 1.5% NS yielded the compressive and flexural strengths of 81.25 MPa and 7.67 MPa, respectively, at 28 days of curing. There are two possible reasons for the increase in strength: the interaction of NS particles with Ca(OH)$_2$ generating more C-S-H gel, and the densification of the microstructure significantly improving the mechanical properties of concrete specimens.

Compared to control concrete, a small dose of NS can significantly improve the chloride penetration resistance of the HSC. A homogeneous dispersion of NS particles was obtained at the 1.5% replacement level. The reduction in the chloride ion permeability was because the NS could cut off capillaries in the concrete, resulting in tortuosity and more disconnected transport channels. The chloride ion permeability reached its lowest when the NS replacement amount was around 1.5%, and the concretes had the best anti-chloride ion penetration capability at 59% and 61%, respectively, for 28 and 56 days.

The compressive and flexural strengths developed with an age curing of 3–28 days by a logarithmic rule, with $R^2 \geq 0.74$. Based on ACI, the compressive strength at 28 days was employed to determine the compressive strength at t-day, with $R^2 \geq 0.95$. The flexural–compressive correlations were established from the obtained data; however, the results had limited accuracy. Further studies should include more results.
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References
1. Chen, J.-F.; Ding, H.-M.; Wang, J.-X.; Shao, L. Preparation and characterization of porous hollow silica nanoparticles for drug delivery application. Biomaterials 2004, 25, 723–727. [CrossRef]
2. Wu, L.; Lu, Z.; Zhuang, C.; Chen, Y.; Hu, R. Mechanical Properties of Nano SiO₂ and Carbon Fiber Reinforced Concrete after Exposure to High Temperatures. Materials 2019, 12, 3773. [CrossRef] [PubMed]
3. Tambichik, M.A.; Mohamad, N.; Samad, A.A.A.; Bosro, M.Z.M.; Iman, M.A. Utilization of construction and agricultural waste in Malaysia for development of Green Concrete: A Review. IOP Conf. Ser. Earth Environ. Sci. 2018, 140, 012134. [CrossRef]
4. Hossain, S.S.; Mathur, L.; Roy, P.K. Rice husk/rice husk ash as an alternative source of silica in ceramics: A review. J. Asian Ceram. Soc. 2018, 6, 299–313. [CrossRef]
5. Le, V.H.; Thuc, C.N.H.; Thuc, H.H. Synthesis of silica nanoparticles from Vietnamese rice husk by sol–gel method. Nanoscale Res. Lett. 2013, 8, 58. [CrossRef]
6. Venkateswaran, S.; Yuvakkumar, R.; Rajendran, V. Nano Silicon from Nano Silica Using Natural Resource (Rha) for Solar Cell Fabrication. Phosphorus Sulfur Silicon Relat. Elem. 2013, 188, 1178–1193. [CrossRef]
7. Fares, G.; Khan, M.I. Nanosilica and its Future Prospects in Concrete. Adv. Mater. Res. 2013, 658, 50–55. [CrossRef]
8. Luo, Z.; Cai, X.; Hong, R.Y.; Wang, L.S.; Feng, W.G. Preparation of silica nanoparticles using silicon tetrachloride for reinforcement of PU. Chem. Eng. J. 2012, 187, 357–366. [CrossRef]
9. Wang, L.E.I.; Guo, F.; Yang, H.; Wang, Y.A.N.; Tang, S. Comparison of fly ash, PVA fiber, MGO and shrinkage-reducing admixture on the frost resistance of face slab concrete pore structural and fractal analysis. Fractals 2020, 29, 2140002. [CrossRef]
10. Zhang, P.; Li, Q.-F.; Wang, J.; Shi, Y.; Ling, Y.-F. Effect of PVA fiber on durability of cementitious composite containing nano-SiO₂. Nanotechnol. Rev. 2019, 8, 116–127. [CrossRef]
11. Wang, L.E.I.; Jin, M.; Guo, F.; Wang, Y.A.N.; Tang, S. Pore structural and fractal analysis of the influence of fly ash and silica fume on the mechanical property and abrasion resistance of concrete. Fractals 2020, 29, 2140003. [CrossRef]
12. Qin, Y.; Zhang, X.; Chai, J.; Xu, Z.; Li, S. Experimental study of compressive behavior of polypropylene-fiber-reinforced and polypropylene-fiber-fabric-reinforced concrete. Constr. Build. Mater. 2019, 194, 216–225. [CrossRef]
13. Qin, Y.; Li, M.; Li, Y.; Ma, W.; Xu, Z.; Chai, J.; Zhou, H. Effects of nylon fiber and nylon fiber fabric on the permeability of cracked concrete. Constr. Build. Mater. 2021, 274, 121786. [CrossRef]
14. Wang, L.; Jin, M.; Wu, Y.; Zhou, Y.; Tang, S. Hydration, shrinkage, pore structure and fractal dimension of silica fume modified low heat Portland cement-based materials. Constr. Build. Mater. 2021, 272, 121952. [CrossRef]
15. Fu, C.; Xie, C.; Liu, J.; Wei, X.; Wu, D. A Comparative Study on the Effects of Three Nano-Materials on the Properties of Cement-Based Composites. Materials 2020, 13, 857. [CrossRef]
16. Aggarwal, P.; Singh, R.P.; Aggarwal, Y. Use of nano-silica in cement based materials—A review. Cogent Eng. 2015, 2, 1078018. [CrossRef]
17. Costel, S.L.; O’Connor, D.; Barnes, P.; Mayes, E.L.; Mann, S.; Freimuth, H.; Ehrfeld, W. Functional micro-concrete: The incorporation of zeolites and inorganic nano-particles into cement micro-structures. J. Mater. Sci. Lett. 2000, 19, 1085–1088. [CrossRef]
18. Du, H.; Du, S.; Liu, X. Durability performances of concrete with nano-silica. Constr. Build. Mater. 2014, 73, 705–712. [CrossRef]
19. Zhang, M.H.; Li, H. Pore structure and chloride permeability of concrete containing nano-SiO₂ particles. Constr. Build. Mater. 2011, 25, 608–616. [CrossRef]
20. Xu, W.; Lo, T.Y.; Wang, W.; Ouyang, D.; Wang, P.; Xing, F. Pozzolanic Reactivity of Silica Fume and Ground Rice Husk Ash as Reactive Silica in a Cementitious System: A Comparative Study. Materials 2016, 9, 146. [CrossRef]
21. Mussa, M.H.; Abdulhadi, A.M.; Abbood, I.S.; Mutalib, A.A.; Yaseen, Z.M. Late Age Dynamic Strength of High-Volume Fly Ash Concrete with Nano-Silica and Polypropylene Fibres. Crystals 2020, 10, 243. [CrossRef]
22. Rai, S.; Tiwari, S. Nano Silica in Cement Hydration. Mater. Today Proc. 2018, 5, 9196–9202. [CrossRef]
23. Jo, B.W.; Kim, C.H.; Lim, J.H. Investigations on the development of powder concrete with nano-SiO₂ particles. KSCE J. Civ. Eng. 2011, 7, 37–42. [CrossRef]
24. Nazari, A.; Riahi, S. Microstructural, thermal, physical and mechanical behavior of the self compacting concrete containing SiO₂ nanoparticles. Mater. Sci. Eng. A 2010, 527, 7663–7672. [CrossRef]
25. Said, A.M.; Zeidan, M.S.; Bassuoni, M.T.; Tian, Y. Properties of concrete incorporating nano-silica. Constr. Build. Mater. 2012, 36, 836–844. [CrossRef]
26. Zhang, P.; Ling, Y.; Wang, J.; Shi, Y. Bending resistance of PVA fiber reinforced cementitious composites containing nano-SiO₂. Nanotechnol. Rev. 2019, 8, 690–698. [CrossRef]
27. Ling, Y.; Zhang, P.; Wang, J.; Taylor, P.; Hu, S. Effects of nanoparticles on engineering performance of cementitious composites reinforced with PVA fibers. Nanotechnol. Rev. 2020, 9, 504–514. [CrossRef]

28. Zhang, P.; Sha, D.; Li, Q.; Zhao, S.; Ling, Y. Effect of Nano Silica Particles on Impact Resistance and Durability of Concrete Containing Coal Fly Ash. Nanomaterials 2021, 11, 1296. [CrossRef]

29. Amin, M.; Abu el-hassan, K. Effect of using different types of nano materials on mechanical properties of high strength concrete. Constr. Build. Mater. 2015, 80, 116–124. [CrossRef]

30. Shah, A.H.; Sharma, U.K.; Roy, D.A.B.; Bhargava, P. Spalling behaviour of nano SiO$_2$ high strength concrete at elevated temperatures. MATEC Web Conf. 2013, 6, 01009. [CrossRef]

31. Shahbazpanahi, S.; Tajara, M.K.; Faraj, R.H.; Mosavi, A. Studying the C–H Crystals and Mechanical Properties of Sustainable Concrete Containing Recycled Coarse Aggregate with Used Nano-Silica. Crystals 2021, 11, 122. [CrossRef]

32. Ghafoori, N.; Moradi, B.; Najimi, M.; Hasnat, A. Transport properties of nano-silica contained self-consolidating concrete. Concr. Compos. Part B Eng. 2020, 115, 17465. [CrossRef]

33. Li, G.; Zhou, J.; Yue, J.; Gao, X.; Wang, K. Effects of nano-SiO$_2$ and secondary water curing on the carbonation and chloride resistance of autoclaved concrete. Constr. Build. Mater. 2020, 235, 117465. [CrossRef]

34. Wang, B.; Zhao, R. Effect of graphene nano-sheets on the chloride penetration and microstructure of the cement based composite. Constr. Build. Mater. 2021, 301, 124060. [CrossRef]

35. Li, C. Chloride permeability and chloride binding capacity of nano-modified concrete. J. Build. Eng. 2021, 41, 102419. [CrossRef]

36. Li, G.; Ding, Y.; Gao, T.; Qin, Y.; Lv, Y.; Wang, K. Chloride resistance of concrete containing nanoparticle-modified polymer cementitious coatings. Constr. Build. Mater. 2021, 299, 123736. [CrossRef]

37. Li, G.; Zhou, J.; Yue, J.; Gao, X.; Wang, K. Effects of nano-SiO$_2$ and secondary water curing on the carbonation and chloride resistance of autoclaved concrete. Constr. Build. Mater. 2020, 235, 117465. [CrossRef]

38. ASTM C1202.

39. ASTM C78.

40. ASTM C1240.

41. ASTM C29.

42. ASTM C197.

43. ASTM C1202.

44. ASTM C856.

45. ASTM C1140.

46. ACI 211.4R-08.

47. Mazloom, M.; Ramezanianpour, A.A.; Brooks, J.J. Effect of silica fume on mechanical properties of high-strength concrete. Concr. Compos. Part B Eng. 2020, 131, 102419. [CrossRef]

48. Jianyong, L.; Pei, T. Effect of slag and silica fume on mechanical properties of high strength concrete. Concr. Compos. Part B Eng. 2021, 129, 123065. [CrossRef]

49. Esposito, S. “Traditional” Sol-Gel Chemistry as a Powerful Tool for the Preparation of Supported Metal and Metal Oxide Catalysts. Materials 2019, 12, 668. [CrossRef] [PubMed]

50. Nair, P.A.K.; Vasconcelos, W.L.; Paine, K.; Calabria-Holley, J. A review on applications of sol-gel science in cement. Constr. Build. Mater. 2021, 291, 123065. [CrossRef]

51. Shakhmenko, G.; Juhelevica, I.; Korjakins, A. Influence of Sol-Gel Nanosilica on Hardening Processes and Physically-Mechanical Properties of Cement Paste. Procedia Eng. 2013, 57, 1013–1021. [CrossRef]

52. Khaloo, A.; Mobini, M.H.; Hosseini, P. Influence of different types of nano-SiO$_2$ particles on properties of high-performance concrete. Constr. Build. Mater. 2016, 113, 188–201. [CrossRef]

53. Naji Givi, A.; Abdul Rashid, S.; Aziz, F.N.A.; Salleh, M.A.M. The effects of lime solution on the properties of SiO$_2$ nanoparticles binary blended concrete. Compos. Part B Eng. 2011, 42, 562–569. [CrossRef]

54. Rong, Z.; Sun, W.; Xiao, H.; Jiang, G. Effects of nano-SiO$_2$ particles on the mechanical and microstructural properties of ultra-high performance cementitious composites. Cem. Conc. Compos. 2015, 56, 25–31. [CrossRef]

55. Ganesh, P.; Murthy, A.R.; Kumar, S.S.; Reheman, M.M.S.; Iyer, N.R. Effect of nanosilica on durability and mechanical properties of high-strength concrete. Mag. Conc. Res. 2016, 68, 229–236. [CrossRef]

56. Seraq, M.I.; Yasien, A.M.; El-Feky, M.S.; Elkady, H. Effect of Nano Silica on Concrete Bond Strength Modes of Failure. Int. J. GEOMATE 2017, 12, 73–80.

57. Ngo, V.-T.; Bui, T.-T.; Lam, T.-Q.-K.; Nguyen, T.-T.-N.; Nguyen, V.-H. Experimental Evaluation of Nano Silica Effects to High Performance Concrete Strength in Early Age. IOP Conf. Ser. Mater. Sci. Eng. 2020, 869, 032011. [CrossRef]

58. Balapour, M.; Joshaghani, A.; Althoey, F. Nano-SiO$_2$ contribution to mechanical, durability, fresh and microstructural characteristics of concrete: A review. Constr. Build. Mater. 2018, 181, 27–41. [CrossRef]

59. Hosseinpourpia, R.; Varshoee, A.; Soltani, M.; Hosseini, P.; Ziaei Tabari, H. Production of waste bio-fiber cement-based composites reinforced with nano-SiO$_2$ particles as a substitute for asbestos cement composites. Constr. Build. Mater. 2012, 31, 105–111. [CrossRef]
58. Sanjuán, M.A.; Argiz, C.; Galvez, J.C.; Reyes, E. Combined effect of nano-SiO$_2$ and nano-Fe$_2$O$_3$ on compressive strength, flexural strength, porosity and electrical resistivity in cement mortars. *Mater. Constr.* 2018, 68, 9. [CrossRef]

59. Committee, A.C.I. Prediction of Creep, Shrinkage, and Temperature Effects in Concrete Structures. *ACI Symp. Publ.* 1997. [CrossRef]

60. Abbasi, S.M.; Ahmadi, H.; Khalaj, G.; Ghasemi, B. Microstructure and mechanical properties of a metakaolinite-based geopolymer nanocomposite reinforced with carbon nanotubes. *Ceram. Int.* 2016, 42, 15171–15176. [CrossRef]

61. Beeby, A.W.; Naranayan, R.S. *Designers Handbook to Eurocode 2 Part I: Design of Concrete Structures*; Thomas Telford Services Ltd.: London, UK, 1995.

62. Zhang, P.; Wan, J.; Wang, K.; Li, Q. Influence of nano-SiO$_2$ on properties of fresh and hardened high performance concrete: A state-of-the-art review. *Constr. Build. Mater.* 2017, 148, 648–658. [CrossRef]