Experimental Evaluation of Nano Silica Effects to High Performance Concrete Strength in Early Age

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Abstract. Nano silicate SiO₂ (NS) in the high-performance concrete (HPC) mixture is becoming great interest because it improves the mechanical properties of concrete. As the ultra-fine SiO₂ particles with a very high ratio of surface area to mass, NS can tighten the structure and produces better quality gel products and thus concrete strength increases. However, the optimal rate of NS in high-performance concrete has not been investigated. This study experimentally evaluates the influence of NS on compressive strength and flexure strength of HPC of which nominal strength is 70MPa. Seven mixtures of HPC at NS rate of 0%, 0.5%, 1%, 1.5%, 2%, 2.5% and 3% over the total weight of the adhesive were conducted. Each mixture provides six samples for compressive tests and six samples for flexure tests. Total samples are 336 samples for four ages of concrete. Reliability of test results was carried out and results shown that at the early stage of HPC, an NS ratio of 1.5% shows the optimal value to improve the performance of concrete. This result also helps to apply NS in HPC in quality control in site application.

1. Introduction

High performance concrete (HPC) has been widely studied and applied in many countries around the world since the 1980s in the last century, bringing great efficiency for technology, economy, and society. Currently, HPC is designed to improve the workability, intensity, and durability of concrete. HPC component is formulated with a low ratio of water/binder (W/B), large aggregates with small \( D_{\text{max}} \), combined with superplasticizer and highly activated mineral additives (Silica fume, fly ash ...). According to CEB.FIP, HPC has a minimum compressive strength of 60MPa after 28 days and has high mechanical and physical properties. HPC is widely applied with intensity levels from 60 ÷ 80 MPa, for most in bridge constructions, high-rise buildings, or factories producing precast concrete structure. Therefore, the study of the use of new materials, which further improve the properties for HPC and ensure strength, durability, and environmental friendliness is becoming increasingly necessary.
In recent times, the use of NS has received a special attraction in fabrication HPC. There are many types of Nanoscale mineral materials that can be used to enhance concrete quality such as Nano SiO$_2$ [1], nano CaCO$_3$ [2], nano Al$_2$O$_3$ [3], nano Fe$_3$O$_4$ [4], nano TiO$_2$ [5] and nano Metakaolin [6]. Among these nanomaterials, NS is the most widely applied type in HPC due to its high activity and exceptionally high surface area, which improves pozzolanic activities in concrete. Moreover, NS can promote C$_3$S dissolution in cement and create highly active C-S-H products [7]. Behfarnia and Salemi [8] observed that the compressive strength of HPC containing SiO$_2$ nanoparticles was much greater than HPC containing the same content of Al$_2$O$_3$ nanoparticles. Du et al. [9] showed that the improvement of NS particles in the compressive strength of lightweight concrete in the early stages (1 - 7 days of age) is particularly noticeable, while the effect of NS may be descending with longer curing time.

The pozzolanic reaction of calcium hydroxide and silica or silica acid in HPC with the presence of NS produces more calcium silicate hydrate (CaH$_4$SiO$_4$.2H$_2$O - C-S-H) which is the main adhesive substance in concrete. Due to a very large surface area of NS, a small amount of these materials can significantly speed up the pozzolanic reaction. A small amount of NS is sufficient to improve HPC’s properties and durability [10]. Qing, Dunster, and Senf [11][12][13] suggested that adding 1kg of silica fume would reduce approximately 4kg of cement and this could be higher if using NS. Another possible application is to retain the amount of cement remains and NS to improve the properties of concrete by utilizing the special properties of NS [14][15]. Nano SiO$_2$ increases hydration reaction by combining with Ca(OH)$_2$, produce high quality C-S-H to increase the mechanical properties of concrete. Concrete with NS will be tighter with less Ca(OH)$_2$ crystals. According to Quercia [16], NS that added to mortar and concrete can have different effects. The cause of this influence is due to the very high surface area of NS and activates the precipitation of C-S-H gel [15]. As a result, NS improves the microstructure and reduces water permeability of concrete. Ji [17] experimentally showed that NS can react with Ca(OH)$_2$ crystals, reduce their size and quantity. Consequently, the interface transition zone (ITZ) of mortar and aggregate is denser [18]. The addition of NS significantly improves the structure of the transition region in concrete at an early age compared to the reduction of content [11]. Up to now, many studies are using NS to adjust the quality of concrete, optimal NS content is different in each report with the limited number of samples, and some unusual effects such as reduction of strength and slump at a high ratio of NS that need attention in further studies [1][10][19][20]. That is why the content of NS used in concrete should be studied with appropriate reliability.

Within the scope of the paper, the authors have initially studied the effects of NS content to compressive and flexural strength of high performance concrete. The methodology of this work experimentally applies various content of NS to find optimal value. A data analysis is applied with numerous experimental values for more reliable results. Since industrial production of HPC concrete needs good quality control practice application, early age of HPC is very important period that optimal ratio of NS is being interested for that purpose.

2. Materials and experiments

2.1. Materials

2.1.1. Adhesive and Additive of HPC. Materials include PC40 cement, coarse aggregate is basalt stone with Dmax = 9.5mm, fine aggregate is yellow sand with a modulus greater than 2.7, Viscocrete 3000-20M superplasticizer. NS particles were tested by SEM electron microscopy and XRD analysis at the Institute of Chemical Technology - Vietnam Academy of Science and Technology. From XRD analysis, from 16° to 30°, it was shown that the compounds were in nano form an amorphous state (Figure 1.b). NS particles are spherical with an average size of about 13nm observed in the SEM experiment (Figure 1.b).
2.1.2. HPC’s Mixture. A concrete mixture with the strength of 70MPa is designed following ACI 211 method. NS is added with different ratios of 0%; 0.5%; 1.0%; 1.5%; 2%; 2.5%; 3%. The percentage of superplasticizer additives are selected according to the manufacturer's recommendations. It is adjusted in practice to ensure the workability of the concrete mixture. The designed mixtures are in Table 1.

### Table 1. HPC Mixture with NS

| Mixture    | Components | W/B |
|------------|------------|-----|
|            | Cement (kg) | FA (kg) | CA (kg) | SF (kg) | NS (%) | SP (l) | Water (l) |
| C70NS0.0   | 544.21     | 674.68  | 1049.75 | 28.64   | 0.00   | 5.44   | 154.67   | 0.27     |
| C70NS0.5   | 541.34     | 673.68  | 1049.75 | 28.64   | 0.50   | 6.53   | 154.67   | 0.27     |
| C70NS1.0   | 538.48     | 672.67  | 1049.75 | 28.64   | 1.00   | 7.35   | 154.67   | 0.27     |
| C70NS1.5   | 535.61     | 671.67  | 1049.75 | 28.64   | 1.50   | 7.62   | 154.67   | 0.27     |
| C70NS2.0   | 532.75     | 670.66  | 1049.75 | 28.64   | 2.00   | 8.71   | 154.67   | 0.27     |
| C70NS2.5   | 529.89     | 669.66  | 1049.75 | 28.64   | 2.50   | 9.25   | 154.67   | 0.27     |
| C70NS3.0   | 527.02     | 668.65  | 1049.75 | 28.64   | 3.00   | 9.80   | 154.67   | 0.27     |

Note: FA - Fine aggregate, CA - Coarse aggregate, NS – Nano SiO$_2$, SF – Silica fume, SP-super plasticizer, W/B – water/binder.

2.2. Mixing and Sampling

As the high surface area and nanometer size of particles, NS is hard dispersion in the concrete mixture. To obtain homogeneous concrete with high stability, the trial mixing process is as follows:

- NS is mixed with 50% water, stirring at high speed to disperse the NS particles uniformly;
- Mix sand, stone, cement, silica fume in 3 minutes;
- Add 25% of water to the mixture of sand, stone, cement, and mix in 1 minute;
- Add the NS mixture, 50% of water to the above mixture and mix in 2 minutes;
- Stir the remaining 25% of water and superplastic additive then adds gradually to the mixture and mix for 2 minutes until being homogeneous.
- Stop the mixer in 1 minute to allow the reaction of super elastic additive for a better effect.
- Mix again for 2 minutes to avoid reducing slumps and ensure homogeneity of the mixture.

**Figure 2.** Preparation of materials for testing sample

The mixer was an active 60-liter mixer and the sampling was a cylinder mold with a diameter of 150mm and a height of 300mm. For the flexure test, the samples were beam type with a dimension of 150x150x600 mm. Before sampling, the inside surface of the mold must be smooth, cleaned, and lubricated. Samples were compacted using a vibration machine with a frequency of 2800-3000 rpm, amplitude 0.35 - 0.5mm then cured at 25°C in the room until removal of mold then soaking in water. Each mixture provides six samples for compressive tests and six samples for flexure tests. To evaluate the influence of NS rate in the concrete mixture of different ages, samples are enough to the age of 3, 7, 28, and 56 days which total samples are 336 samples.

**Figure 3.** Sampling and curing samples.

2.3. **Compressive and Flexural Strength Testing**

After molding and curing, compressive strength and flexural tensile strength tests adopt to ASTM C39 [21] and ASTM C78 [22], respectively. The testing apparatus is San 3000 electronic compressor with a maximum load of 3000kN and load incensement speed is 0.3MPa/s (Figure 4).
3. Results and Discussions

3.1. Reliability analysis method of the test results

Design of Experiments (DoE) and Statistical analysis with Minitab 19 software at 95% reliability level, significance level $\alpha = 5\%$. The input variables of Design of Experiments include two variables as follows: Variable 1 - age: 3 days; 7 days; 28 days; 56 days; Variable 2 - NS rate: 0%; 0.5%; 1%; 1.5%; 2%; 2.5%; 3%. Criteria need to be analyzed: compressive strength ($R_n$) and flexural strength ($R_{ku}$).

3.2. Compressive strength

Figure 5 shows compressive strength results with typical strength $f'_c = 70\text{MPa}$ with 95% Confidence Interval (CI). The analysis showed that the testing results ensure the precision and the compressive strength are different at the different ratios of NS and age.
The analysis results show that all variables and combinations of variables affect the compressive strength with statistical significance.

![Residual Plots for Rn](image)

**Figure 5.** Compressive strength diagram Rn 95% CI

**Figure 6.** Analysis chart for residual plots for Rn

The chart evaluating the residuals of the compressive strength regression function (Figure 6) shows that the residuals are very close to the normal distribution. The graph of covariance assessment shows the relationship between the residuals and the corresponding values of the regression model, random values; irregular and uniformly distributed on both sides via the "0" line so satisfying conditions for applying experimental statistical methods.
As the analysis shown in Figure 7, the compressive strength of HPC increases rapidly at the age of 3 days. After 7 days to 28 days, the compressive strength still increases and maintains the intensity development speed, but from 28 to 56 days of age, the growth rate decreases. Thereby, it clearly shows the influence of the NS on the compressive strength of concrete. The compressive strength of concrete increases with NS ratio is from 0.5 ÷ 1.5%. When using the NS ratio of 2 ÷ 3%, the compressive strength is lower than the concrete with an NS ratio of 1.5%.

3.3. Flexural Strength

Flexural strength test results are in Figure 8 with a 95% confidence interval. The analysis shows that the testing results provided a sufficient level of precision and compressive strength varies at different ratios of NS and ages.

**Figure 7.** The diagram of factor’s interacting influence on $R_n$

**Figure 8.** Flexural strength diagram 95% CI
The analysis results show that all variables and combinations of variables affect the flexural strength with statistical significance.

**Figure 9.** Analysis chart for residual plots for flexural strength

Similar to compressive strength, the chart evaluating the residuals of the flexural strength regression function (Figure 9) shows the residuals are very close to the normal distribution. The graph of covariance assessment shows the relationship between the residuals and the corresponding values of the regression model are random, irregular and uniformly distributed on both sides of the "0" line, which satisfying conditions for applying the experimental statistical methods.

**Figure 10.** The diagram of factor’s interacting influence on flexural strength

As shown in Figure 10, the flexural strength of HPC increases rapidly at the age of 3 days. After 7 days to 28 days, it still increases but from 28 to 56 days, the growth rate decreases. Thereby, it clearly shows the influence of NS on the flexural strength of concrete and the slope of the relational line between flexural strength and NS ratio of different ages is steeper than compressive strength’s one (Figure 10).

### 3.4. Discussion

It shows that in the HPC mixture, NS’s effect on flexible tensile strength is more than on compressive strength. Similarly, to compressive strength, the flexural strength of concrete increases significantly
with the NS ratio from 0.5 ÷ 1.5%. When NS is at the ratio of 2 ÷ 3%, the strength is lower than concrete with an NS ratio of 1.5%. This trend was also mentioned in some studies, such as those of Khaloo et al. [1], Chithra et al. [10], Givi et al. [23]. The results of these studies have initially confirmed the efficiency of concrete’s mechanical properties using NS. However, the undesirable effects reduce the strength when using an excess ratio of NS. Khaloo’s research which using a type of NS having a typical surface area of 200 m² / g with a ratio of 1.5% NS improved concrete’s compressive and flexural strength. Givi et al. [23] also concluded that the bending strength of concrete with NS component at the ratio from 1 to 1.5%, will be better than the control concrete. However, the concrete strength decreases if NS particle’s participant is close to 2%.

The results show that the addition of NS to HPC has increased the compressive strength and flexural strength of concrete at an early age (3 days). The flexural strength increases 21.07%; 18%; 14.82%; 13.86% in 3days, 7days, 28days and 56days age, respectively. In the period of 28 to 56 days of age, the speed of strength development is reduced compared to its early age. The concrete strength increased by a maximum of 21.07% in three days compared to control one that is corresponding to an NS rate of 1.5%. This result is a pick value that provides the optimal value of NS to increase strength of HPC at an early age. In this study, the effects of NS to pozzolanic reactions, elimination of unstable components in concrete as well as quality gel products, which concern to permeability of concrete, will be discussed in further publications.

4. Conclusions

HPC mixtures were designed and tested with seven ratios of NS from 0.5% to 3.0% to total adhesive. A total of 336 samples were in carefully controlled experiments. The numbers of samples for each mixture are six specimens that ensure the reliability of testing results. Compressive and flexure strength tests were conducted at 3, 7, 28, and 56 days of age. Results were analyzed using reliability analysis for numerous quantities of specimens and showed that this Nano-particle can improve the strength of HPC. The improvement of these strengths compared to control one reaches to the highest value of 21.07% at three days. This highest value can be obtained with ratio of NS is 1.5% to the adhesive. A lower or higher ratio of NS produces a less effective improvement of HPC in terms of compressive and flexure strengths. As a result, NS can play a positive role in HPC application at site conditions where early ages of concrete need to increased strength first.

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