Effects of additional nanosilica of compressive strength on mortar

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Abstract. The use of nanosilica as one of the innovations in concrete technology has developed very rapidly. Some research mentioned that nanosilica obtained from the synthesis process silica sand is a type of material that is as pozzolan when added to the concrete mix, so as to accelerate the hydration process in concrete. With the addition of nanosilica into the concrete mix, the compressive strength of the concrete can be increased and it has a high durability. This study aims to determine the effect from the addition of nanosilica on mechanical properties of concrete. Laboratory testing is conducted by making the mortar test specimen size of 50 mm x 50 mm x 50 mm. The material used is composed of silica sand, nanosilica, gravel, superplasticizer, cement, and water. Nanosilica percentage amount is added as much as 5, 10, and 15% by weight of cement. Testing of mechanical properties such as compressive strength mortar done at age 3, 7, 14, and 28 days. Based on the analysis and discussion obtained that at 28 days, mortar with the addition of 5% and 15% nanosilica has the compressive strength of 23 MPa. Addition nanosilika into the mortar to improve the mechanical properties by increasing the compressive strength of mortar. The compressive strength of mortar with the addition of 10% nanosilica is 19 MPa. The increase in compressive strength of mortar with the addition of 5% and 15% nano silica is 21% larger than the mortar with the addition of 10% nanosilica and without nanosilica. Nanosilica addition of more than 10% can cause agglomeration when mixed into the mortar so that the impact on the compressive strength of mortar.

1. Introduction

The use of concrete technology that is applied to the bridge structure has developed very rapidly. Several studies have related to high-performance concrete has been done in some countries, one of them is with the nano technology. In Indonesia, the research on nanotechnologies for concrete construction began in 2009. Some research states that nanotechnologies applied to the concrete structure can improve the compressive strength and durability so that it can extend the service life of the bridge. The concept of nano technology aims to make concrete becomes denser. Very high density of the concrete is obtained because the empty spaces that exist between particles of relatively large such as cement particles can be filled by a nanometer-sized grains of fine dust from reactive. Accordingly forming the concrete with a very dense arrangement of the structure, where the pores are formed are in the nanometer size, smaller than the size of the capillary or practically no longer containing capillary-sized pores. With the being filled pores of the concrete by nanomaterial cause the mechanical properties and durability of concrete to be better than conventional concrete [4]. In general, concrete is a mixture of water, cement, fine aggregate, and coarse aggregate. The addition of
nanomaterial as a cement substitute material causes the strength and durability of concrete is higher. In a study conducted a set of test specimen making and testing of compressive strength of mortar. Nanosilica as the material is smaller than 100 nm have the chemical and physical properties that are superior to large-size material, so that the resulting concrete is expected to have better characteristics. The use nanosilica as a partial cement substitute material capable of increasing the density and improves the mechanical properties of concrete, especially in the area of interface between coarse aggregate and mortar to form a compact structure. The development of nano technology allows nanosilica used as concrete forming materials. However, before being used in the concrete mix, the nanosilica important to know that the performance characteristics of concrete formed according to strength of plan and has no negative impact on the establishment of concrete materials [11]. Experimental test of concrete with nano-scale material substitutions have been carried out both in terms of physical properties and mechanical properties. Some of the studies that have been done related to the addition of nanomaterials to a mortar mix which are:

Byung et.al., (2007) evaluated the compressive strength of mortar with the addition nanosilica 0; 3, 6, 9, and 12%. Variations of water cement ratio used is 0.23; 0.25; 0.32; 0.35 and 0.48. The compressive strength of mortar evaluated with the addition of 5%, 10%, and 15% silica fume. The results of the study mortar at 28 days showed that the strength of mortar with nanosilica higher than the compressive strength of mortar with silica fume [1]. Khanzadi et al., (2010) to test the SEM on the concrete with and without the addition of nanosilica. SEM test results indicate that ettringite crystals that has been infiltrated by the crystal C-S-H to form a slab that is more dense, so concrete with nanosilica more homogeneous than plain concrete [2]. Nittaya and Apinon (2008) carried out tests SEM nanosilica mortar with the addition of 4% and 10%. The addition in the cement paste nanosilica cause microstructural differences pasta [7]. Nili et al., (2010) examines that the use nanosilica 6.0% as a partial replacement of cement in the concrete mix with f.a.s = 0.45 able to increase the compressive strength and reduces absorption [8]. Sobolev et al., (2006) study the mechanical properties of mortar with nanosilica were synthesized using sol-gel method. The results showed an increase in the compressive strength of the mortar in the early stages of hardening followed by a decrease in strength in the elderly. The addition of superplasticizer done to overcome these obstacles. With the addition of superplasticizer in mortar, an increase in the compressive strength of (15-20)% [10]. Luciano et al., (2009) conducted a study on the effect of nanosilica (nano SiO₂) against setting time in the cement paste. Samples made with nanosilica composition of 0%; 1%, 2% and 5% of the weight of the cement. Consistency pasta adjusted by adding superplasticizer in different amounts. In general it was observed that the addition in the cement paste nanosilica affect the process of setting time[3].
Sadrmomtazi et al. (2009) conducted a study with nanosilica and silica fume added to the mortar mix with a composition of 0; 3; 5%; Nanosilica 7% and 10% silica fume. Based on the results of the study concluded that absorption value was lowest for the mixture with nanosilica as much as 7% [9].

Configuring the microstructure of concrete is influenced by the composition of the mixture, the water-cement ratio (f.a.s), diameter, and the gradation of its constituent materials [5]. The way to determine the microstructure of concrete is cut transversely and elongated so that the configuration between fine aggregate, coarse aggregate, mortar and air pores clearly visible. If the cross section was enlarged with the test SEM (Scanning Electron Microscope), then the chemical elements C-S-H, C-A-S-H and C-H contained in the concrete microstructure can be seen clearly. Repair concrete microstructure can improve the performance of concrete. Concrete ways to improve performance is to increase or decrease the density of pores, increasing the strength of the bond between the mortar and the mortar and coarse aggregate [11]. Interface Transition Zone (ITZ) is the weakest area on the concrete material and often decline and extinction of first. This is caused by the bond between coarse aggregate and mortar are not perfect. Therefore, efforts to strengthen Interface Transition Zone is one of the solutions to obtain high-strength concrete. The concrete given uniaxial load will experience the crack perpendicular to the direction of loading. When cracks occur, the strength will be reduced very rapidly which is characterized by the instability of the concrete after the peak load. Behavior of concrete begins to deviate from linear conditions along with the start cracking in concrete, which initially raised in the transition area aggregates and paste. The crack propagation resistance weakens the concrete to the work load, thus forming a stress strain diagram of the curve. Mortar is the material used for the construction which consist of a mixture of cement and fine aggregate. The mix of cement and aggregates using specific comparison so that the mortar resistance to stress and pull will be higher or maximum. Based on data specifications, the largest content of silica sand as fine aggregate used is a compound with a composition of 99.09% SiO$_2$. In addition as a mixture of mortar, sand is used as nanosilica material processed through the synthesis process and nanosiasi. In normal concrete, mortar or bonding strength between the mortar and aggregate are relatively weaker than the strength of aggregate. This study aims to determine the increase in compressive strength when added to nanosilica as a partial cement substitute material.

2. Methodology

The methodology used in this research begins with the synthesis and characterization of nanosilica conducted at the Center for Ceramics. Making test specimen carried in Bridge Structures Laboratory. Test specimen was made in the form of mortar size of 50 mm x 50 mm x 50 mm. Making mortar is done with addition of nanosilica variation of 5, 10, and 15% by weight of cement OPC (Ordinary
Portland Cement). Mortar with addition of 5, 10, and 15% respectively nanosilica identified as NS5, NS10, and NS15. Preparation of test specimens for each mortar is 3 specimen. Fine aggregate used was silica sand from Bangka island with a mesh size 325. Each variation uses a mixture of silica fume (SF) with a percentage of 5% of the weight of cement. The addition of superplasticizer conducted to ease the workability of mortar mix with a maximum use of 2% to weight of cement. The testing of mortar compressive strength is done at the age of 3, 7, 14, and 28 days with the amount each of the 3 test specimen. Testing equipment used in making mortar consists of ovens, scales, mixers, vicat, measuring cup, cone mold, leveling instrument, vibrators, dryers fine aggregate, mortar mold, and other equipment. Mortar mix made with the following composition:

- Cement OPC = 475 g (21%)
- Silica sand from Bangka = 1392 g (60%)
- Silica Fume (5%) = 25 g (1%)
- Water = 399.8 g (18%)

SEM testing aims to determine the configuration of the mortar structure microscopically and element content in mortar. SEM testing for mortar without nanosilica done with a magnification of 1000 times, 5000 times, 10000 times, 15000 times, 20000 times, and 50000 times. Making the mortar mix made with reference to the SNI-03-6825-2002 (method of testing the compressive strength of Portland cement mortar for civil works).

3. Results and Discussion

Penambahan nanosilika ke dalam campuran mortar dimaksudkan untuk mengisi rongga atau pori yang terkandung dalam mortar. By being filled voids or pores are expected structural configuration within the mortar could be more solid so that the compressive strength of the mortar is higher than conventional mortar. Nanosilica are put into a mortar mix should be reactive, because in addition to functioning as a filler, it acts as a binder nanosilica. To determine the level of reactivity nanosilica, it must be tested XRD. If the test result shows the XRD amorphous chart pattern, then the nanosilica are reactive and can be added to the mortar to improve the compressive strength than mortar. Nanosilica used in this study are shown in Figure 1.
Based on the test results of XRD, the diffraction pattern of the resulting nanosilica is amorphous as shown in Figure 2. Nanosilica obtained from the synthesis when it is separated from impurities (i.e. Al₂O₃) will produce the compound SiO₂ greater than 99%. If it does not continue the synthesis process, the test results will show the XRD diffraction pattern of crystalline. The sharp diffraction patterns indicate that the process of synthesis which do not produce the amorphous nanosilica.

With the diffraction pattern as shown in Figure 1 is proven that nanosilica used as a cement substitute material is reactive, so that when added to the mortar will cause an increase in the compressive strength. The amorphous properties of that shown in Figure 2 indicates that the levels of SiO₂ compound contained in such nanosilica in greater than 99%. At the time of the hydration process, a mortar mix consisting of silica sand, cement, and water will react to primary products calcium silicate.
hydrate (3CaO.2SiO2.3H2O) gel and calcium hydroxide. The main elements of this paste directly influence the strength of the mortar and to function as a space filler between the cement grains, filling the pores of the capillaries thus diminishing the porosity and increasing strength.

The test results of mortar to the age of 3 days, 7 days, 14 days and 28 days are shown in Table 1.

| No | Sample   | 3 days | 7 days | 14 days | 28 days |
|----|----------|--------|--------|---------|---------|
| 1  | NS0 (1)  | 13     | 16     | 19      | 21      |
| 2  | NS0 (2)  | 14     | 15     | 17      | 20      |
| 3  | NS0 (3)  | 14     | 15     | 19      | 19      |
|    | Average  | 14     | 15     | 18      | 20      |
|    | Standard deviation | 0.64 | 1.01 | 1.50 | 0.63 |
| 1  | NS5 (1)  | 15     | 17     | 20      | 23      |
| 2  | NS5 (2)  | 15     | 18     | 21      | 24      |
| 3  | NS5 (3)  | 14     | 17     | 20      | 24      |
|    | Average  | 15     | 17     | 20      | 24      |
|    | Standard deviation | 0.79 | 0.73 | 0.59 | 0.61 |
| 1  | NS10 (1) | 12     | 18     | 16      | 21      |
| 2  | NS10 (2) | 11     | 17     | 14      | 20      |
| 3  | NS10 (3) | 11     | 15     | 16      | 18      |
|    | Average  | 12     | 17     | 15      | 20      |
|    | Standard deviation | 0.20 | 1.57 | 0.93 | 1.19 |
| 1  | NS15 (1) | 15     | 16     | 23      | 27      |
| 2  | NS15 (2) | 13     | 17     | 25      | 26      |
| 3  | NS15 (3) | 11     | 17     | 21      | 18      |
|    | Average  | 10     | 11     | 16      | 17      |
|    | Standard deviation | 4.97 | 6.89 | 8.90 | 8.70 |

Remarks:
NS0 is a mortar without nanosilica
NS5 is a mortar with the addition of 5% nanosilica
NS10 is a mortar with the addition of 10% nanosilica
NS15 is a mortar with the addition of 15% nanosilica

Based on Table 1 the value of the average compressive strength of mortar N0, N5, N10, and N15 at 28 days respectively was 20; 24; 20 and 17. Barchart against the concrete compressive strength test results in Table 1 can be seen in Figure 3.
Figure 2 shows the development of the compressive strength of mortar by age. All mortar composition has the behavior of different compressive strength at age 3, 7, 14, and 28 days. NS10 mortar compressive strength at age 3 days is the most low of 12 MPa and the highest is NS5 at 15 MPa. At the age of 3 days, mortar with the addition of 5% nanosilica (NS5) 15 MPa compressive strength is almost the same as the mortar without nanosilica amounted to 14 MPa. Furthermore, at the age of 7 to 28 days of mortar that contains nanosilica progressing compressive strength faster than the compressive strength of mortar without nanosilica. Nanosilica addition of more than 10% of the compressive strength will not increase because during the process of mixing the mortar will occur agglomeration. This is caused by nanosilica is fine and reactive material so that when mixed with calcium oxide (CaOH) free produce secondary reactions that form new cement paste. The development compressive strength of mortar with 10% nanosilica at the age of 3 days up to 28 days has a different strength than other mortar. At the age of 3 day compressive strength of 12 MPa was the lowest compared to other mortar. After reaching the age of 7 days, the compressive strength of mortar (NS10) an increase of 17 MPa. But at the age of 14 days, compressive strength being dropped 15 MPa and at 28 days to 20 MPa. The increase in compressive strength of mortar with the addition of 15% nanosilica (NS15) occurred in the age of 7 to 28 days. At the age of 3 days, the compressive strength of mortar NS15 is smaller (i.e 13 MPa) than without nanosilica (14) MPa. It is caused the hydration reactions at beginning of age is still dominated by the cement hydration reaction with water. At the age of 28 days, the compressive strength of mortar with the addition of 15% nanosilica (NS15) at 23 MPa less than the compressive strength of mortar with 5% nanosilica (NS5) is 24 MPa. But at the age of 14 days, the compressive strength of mortar NS15 larger (ie 23 MPa) compared with 5% nanosilica mortar (NS5) with a compressive strength of 20 MPa. The increase in compressive strength of mortar with the addition of 5% nanosilica is the most optimum for the increase is continuous from the age of 3, 7, 14 up to 28 days. Mortar with the addition of a 10% increase in compressive strength nanosilica
experience a discontinuity at the age of 14 days. This could be caused during the process of mixing mortar occur agglomeration nanosilica so it does not react in the optimum. From some of the tests conducted, if nanosilica mixed with water and water will immediately be absorbed. While the percentage increase of 15% nanosilica to mix mortar for compressive strength increase is not significant at 28 days. To determine microscopic structure of the mortar test SEM (Scanning Electron Microscope)/EDS (Electron Dispersion Spectrometry). Testing SEM/EDS was conducted to determine the composition and structure of chemical compounds in micro mortar. Figure 4 shows the test results of SEM/EDS mortar without addition of nanosilica. Magnification of mortar structure made up to 1000, 5000, 10000, 15000, 20000 and 50000 times.
Based on Figure 4 can be seen that the mortar is still a small pipes at a magnification of 20000 times. Small pipes have a hollow (or pore) that causes mortar not be impermeable. The composition of elements and chemical compounds contained in mortar shown in Figure 5.

The number of elements and chemical compounds in the mortar of Figure 4 can be seen in Table 2.

Table 2. Total composition of elements and compounds in a mortar without addition nanosilica.

| Element Line Type | Apparent Concentration | k Ratio | Wt% | Wt% Sigma | Standard Label | Factory Standard |
|-------------------|------------------------|---------|-----|-----------|----------------|------------------|
| 1                 | K series               | 4.75    | 0.04746 | 10.54 | 0.43 | C Vit | Yes |
| O                 | K series               | 76.77   | 0.25833 | 55.07 | 0.34 | SiO2 | Yes |
| Mg                | K series               | 0.96    | 0.00636 | 0.67  | 0.03 | MgO  | Yes |
| Al                | K series               | 1.65    | 0.01186 | 0.91  | 0.03 | Al2O3 | Yes |
| Si                 | K series               | 38.85   | 0.30783 | 18.04 | 0.15 | SiO2 | Yes |
Based on Table 2, the element oxygen (O) contained in the compound in a mortar SiO$_2$ is the highest of 55.07% and element content of silica (Si) in the compound was 18.04%. Total SiO$_2$ composition of each of the elements oxygen and silica amounted to 73.11%. The percentage of the element silica comes from silica sand used as fine aggregate. While the composition of the structure in a mortar with the addition of nanosilica (for the number of nanosilica 5% by weight of the binder) can be seen from the test results of SEM as shown in Figure 6.

![Figure 6. SEM test results mortar with the addition of 5% nanosilica.](image)

Based on Figure 6, the structural arrangement mortar with the addition of 5% nanosilica there are the pipes as the capillary cavity. At a magnification of 20000 times, look more resistant mortar. The composition of elements and compounds contained in the mortar can be seen from the test results of EDS as shown in Figure 7.
Figure 7. The composition of the configuration (a) and the number of element content in mortar with 5% nanosilica (b).

Based on Figure 7, the element oxygen (O) contained in the mortar is the most dominant element. Total the composition each of the elements and compounds contained in a mortar with the addition of 5% nanosilica shown in Table 3. From Table 2, the element oxygen (O) contained in the compound in a mortar SiO\(_2\) is the highest of 48.56% and element content of silica (Si) in the compound was 14:03%. Total SiO\(_2\) composition of each of the elements oxygen and silica amounted to 62.59%. The percentage of the element silica comes from silica sand used as a fine aggregate and nanosilica. This is less than the amount of SiO\(_2\) mortar composition without nanosilica. By the SiO\(_2\) content as the dominant chemical compounds, nanosilica potentially reducing calcium hydroxide in mortar.

Table 3. Total composition of elements and compounds in a mortar with the addition of 5% nanosilica.

| Element | Line Type | Apparent Concentration | k Ratio | Wt% | Wt% Sigma | Standard Label | Factory Standard |
|---------|-----------|-------------------------|---------|-----|-----------|----------------|------------------|
| C       | K series  | 9.18                    | 0.09181 | 18.13 | 1.09      | C Vit          | Yes              |
| O       | K series  | 52.36                   | 0.17621 | 48.56 | 0.70      | SiO2           | Yes              |
| Mg      | K series  | 0.76                    | 0.00507 | 0.54  | 0.03      | MgO            | Yes              |
| Al      | K series  | 2.95                    | 0.02122 | 1.66  | 0.04      | Al2O3          | Yes              |
| Si      | K series  | 29.10                   | 0.23057 | 14.03 | 0.22      | SiO2           | Yes              |
| K       | K series  | 3.26                    | 0.02761 | 1.16  | 0.03      | KBr            | Yes              |
| Ca      | K series  | 40.37                   | 0.36070 | 14.75 | 0.22      | Wollastonite   | Yes              |
| Fe      | K series  | 2.28                    | 0.02283 | 0.99  | 0.04      | Fe             | Yes              |
| Cu      | K series  | 0.41                    | 0.00408 | 0.18  | 0.04      | Cu             | Yes              |
| Rb      | K series  | 0.00                    | 0.00000 | 0.00  | 0.00      | Rb (v)         | Yes              |
| W       | L series  | 0.00                    | 0.00000 | 0.00  | 0.00      | W              | Yes              |
| Total:  |           | 100.00                  |         |       |           |                 |                  |

This reduction continued because the chemical reaction between compounds of silica dioxide (SiO\(_2\)) with calcium hydroxide to form a calcium silicate hydrate (CSH) new. It can be concluded that
nansilica freely mixed with calcium hydroxide will happen further reaction that forms a new cement paste. This gives effect to the relationship between the surface of the mortar with the zone to be stronger and provide increased compressive strength in mortar.

4. Conclusion

The highest mortar compressive strength at 28 days resulting in a mixture with nanosilica 5% ie 23.5649 MPa.

Nanosilica addition of more than 10% can cause agglomeration when mixed into the mortar so that the impact on then compressive strength of mortar. It was proven that the compressive strength of the mortar with the addition of more than 10% nanosilica be lower than conventional mortar. Mortar compressive strength with the addition of 15% nanosilica at 17 MPa lower than conventional mortar which is 20 MPa. The decrease in the compressive strength can be caused when the distribution mixing of nanosilica were added to the mortar uneven, in part, on the agglomeration and therefore can not fill the pores in the mortar.

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