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**Chapter**

# Impact of Nanosilica in Ordinary Portland Cement Over Its Durability and Properties

*Gude Reddy Babu, Pala Gireesh Kumar, Nelluru Venkata Ramana and Bhumireddy Madhusudana Reddy*

#### **Abstract**

The present examination illustrates the impact on the hardened and fresh cement mortar and cement with the inclusion of nanosilica of size 40 nm in various environmental conditions (UltraTech, India). It is quite notified that an elevation in compressive strength as well as flexural strength along with an improvisation in the performance and life span of cement mortar. The samples of M5 grade blended with a ninety percentage of concrete and remaining with nanosilica was identified to have a finer working elevation in as well as in standards when collated with the conventional cement mortar. The corollary of hardened and fresh cement, strength parameters were looked upon with the aid of XRD (X-ray Diffraction). Also, the SEM (Scanning Electron Microscope) test holds a predominant role in analysis.

**Keywords:** OPC, Strength, HCl, MgSO4, XRD, nanosilica and SEM

#### **1. Introduction**

There is no other substitute for concrete that can be supplemented with an alternative because of its intrinsic qualities like get into any shape, quality and ability to consume locally available fine and coarse materials along with its strength, resistance to fire, with little support [1]. The use of concrete and its consumption is on par with wans and the activity of construction is set to have been emitting 8% of  $CO<sub>2</sub>$  [2]. Even though the substitute for concrete is not identified the area of development becomes the centre stage for identifying substitute materials. The contribution of nano technology has made big strides and effect on various aspects of science. Nano silica is preferably utilized in numerous approaches and is easily available as well.

The research and investigation in nano particles and their utilization in cement and mortar invariably becomes popular [3]. The properties of cement in both hardened as well as in fresh phase are majorly laid focus on. The density of concrete and cement can be improvised with the inclusion of nano as well as micro NS which have the tendency of acting as a material of filler, which results in up gradation of its quality [4–17]. Nano silica with separated molecule impact got an extraordinary pozzolonic property than other pozzolonic materials on comparison because of the nano size of particles and greater silica contents, accordingly both impacts are

important in making concrete [4, 6]. Expansion of Nano silica to solidify concrete and cement mortar then resulted in improvement of sharp and long haul considers and over utilisation of nano silica results in decrease in quality. Nano silica greatly improved qualities of cement and concrete in fresh and solidified stage and in durability in different environmental conditions [10–11, 18–19]. The nano particles are centered and a considerable research is carried out.

Therefore, the present research includes the impact and durability of 40 nm silica particles in cement mortar, durability existence under different environments both physical and chemical are taken up for examination [20–23]. For acquiring an accurate output, instruments of Scanning Electron Microscope (SEM) and X-ray diffraction (XRD) were resorted to. In this research, cement to sand proposition was 1: 3 and water to cement (concrete plus NS) proposition was set at 0.4.

### **2. Procedure**

In the set forth work area, the proportion of 1:3 is considered for cement-sand and 0.4 for water-cement. Also, nanosilica is incorporated along with cement starting from zero percentage and with every sample, an increase of 2% is followed until an overall 14% is achieved. Every sample is treated individually with starting with M0 and concluding with M7.

#### **2.1 Blending of nano silica with cement**

Sticking to the particulars of Jo B W [24], the blending of NS with cement mortar is done for a minute maintaining 285 rpm. Prior to the blending process, the water is treated with NS and this was put into the rotational blender which runs at 140 rpm. This was done for 30 seconds so as to solidify the mixture and addition of a fine total was done. Adding to the process, a super plasticizer was annexed to the mix when the blender was running at a speed of 285 rpm. Now, a resting phase of 90 seconds is allocated to the blender and it resumes working for a minute with the same speed before rest. Finally, the mix was set out in a hardened figure.

### **2.2 Hardening or setting interval (setting time)**

IS of code 5513-1976 was used for detecting the time taken for setting.

### **2.3 Strength determination (compressive strength)**

The part 6 of 4031-1998 was considered to find the compressive quality of mortar.

#### **2.4 Strength determination (flexural strength)**

Determining the flexural standards using the BS section 188 of 1881 of the year 1983 was done.

#### **2.5 Durability aspect**

AR graded magnesium sulphate and hydrochloric acid, which are synthetic in nature, were used in the research. The ACI of 318-99 were followed and the compound grouping was done. An exemplar of the fixations projects over a span of 120 days for 1%, 240 days for 2%, 360 days for 3% and 480 days for 4%, provided the recharge of the fixations was for each 4-month tenure.

#### **2.6 Temperature assessment**

The blended samples of M0 as well as M5 were tested upon for the temperature check at 900 °C in a suppress heater. Also, the blended sample out-righting 28 days were dried for a span of 6 hours at normal temperature and then they were transferred to a muffle furnace. An altering is done with temperatures where each temperature has got three samples assessed for a period of two and half hours. The altering temperatures start from 100 °C and every alteration has an add-on of 100 °C up to 600 °C. Finally, the samples were put out to cool down at a temperature of 30 °C i.e., room temperature (**Tables 1**–**5**).



#### **Table 1.**

*Constitutes of cement.*



#### **Table 2.**

*Cement attributes.*



**Table 3.** *Ennore sand attributes.*

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#### **Table 4.**

*Attributes of nanosilica.*



**Table 5.** *Attributes of water.*

### **3. Deliberation of verdict**

#### **3.1 Hardening or setting interval (setting time)**

**Figure 1** reveals the corollary on setting time due to NS and uncovers the fact of lowering the hardening time with increment in NS quantity. The initial and final setting time for M0 was 155 min and 266 min, M1 was 153 min and 263 min, M2 was 148 min and 260 min, M3 was 143 min and 255 min, M4 was 137 min and 245 min, M5 was 131 min and 240 min, M6 was 126 min and 234 min, M7 was 123 min and 227 min. The cycle was advanced because of NS has a vast surface region; subsequently, hydration measure turns out to be quick.

#### **3.2 Analysis of strength (compression)**

A rise in the strength upon inclusion of nanosilica up to 10% was portrayed in **Figure 2**. With further addition of NS, decrement in strength can be observed from the diagram. The finest of all samples is the M5 sample comprised of 90% cement and 10% NS. For 3 days, the increment strength observed was 17.8 MPa, 7 days resulted 21.58 MPa, 28 days directed a strength of 21.18 MPa, 90 days showcased 21.50 MPa, 180 days witnessed 21.32 MPa and finally, 365 days exhibited a strength of 20.66 MPa. From the analysis, it is truely clear that M6 blended with 88% of cement and the remaining with NS has seen a decrease in compressive strength whereas M5 figured the best strength notable.



**Figure 1.** *Efficacy of nanosilica over setting span.*



**Figure 2.** *Efficacy of nanosilica over strength attribute (compression).*

#### *3.2.1 Manoeuvre of XRD*

The analysis was done for a couple of samples having 0% NS and 10% NS i.e., for M0 and M5 by X-ray diffraction, XRD. The samples were of OPC type and were hydrated for a span of 28 days. **Figure 3** shows that hydroxide of calcium showed up at 18° whereas the calcium silicate hydrate showed up at 26° individually for both the samples. Concentration and strength of hydroxide of calcium was predominantly high and that of silicate hydroxides of calcium was less when compared to OPC of 90% and nano silica of 10% test. The variation of strength for the sample of M0 and the sample of M5 was due to the reaction of oxides of silica with hydroxides of calcium (finished results of cement hydrate).

#### *3.2.2 Manoeuvre of scanning electron microscope*

**Figures 4** and **5** demonstrate the results for the sample M0 and also for the sample M5 which were hydrated for 28 days. The diagram of Scanning Electron Microscope (SEM) portrays the hydroxides of calcium's needle like structure along with the silicate hydroxide of calcium which was spread widely. The calcium hydroxide's long needle like structures in **Figure 4** can be collated with **Figure 5**.

Also, a high in silicate hydroxide of calcium's content can be seen in **Figure 5** when compared to **Figure 4**. The major difference in the structure of hydroxides of calcium and quantity of silicate hydroxide of calcium was due to the presence of oxides of silica in NS. These react with hydroxides of calcium in the cement hydrate and thereby, causing the differences.



**Figure 3.** *Depiction of M0 & M5 samples XRD (hydrated for 28 days).*



**Figure 4.** *Depiction of M0's SEM diagram (28 days of hydration).*



**Figure 5.** *Depiction of M5's SEM diagram (28 days of hydration).*

#### **3.3 Analysis of flexural strength**

The NS impact on flexural strength can be identified from the **Figure 6**. It was evident that cement with less than 10% NS exhibited expand in flexural strength by 10% whereas cement with NS in more than 10% showcased decline in the strength. The samples were identified upon M5 blend with a higher expansion rate with time. It was seen that 2 MPa was recorded for 3 days, 2.2 MPa for 7 days, 2.5 MPa for 28 days, 2.4 MPa for 90 days, 2.4 MPa for 180 days and finally 2.6 MPa for 365 days. From the analysis, we can notice that samples has shown a decrease in strength with NS percentage more than 10 but the value was higher when compared with the strength of sample with zero percentage of NS. The major concerns that led the expansion of the strength characteristics were the size of the particles and content of oxides of silica. An extra quantity of silicate hydroxide of calcium was witnessed due to the action of hydroxides of calcium and oxides of silica. Furthering this, the NS acts as a framework's filler material of cement mortar.

#### **3.4 Probes on durability aspect**

#### *3.4.1 Corollary of magnesium sulphate*

**Figures 7** and 8 render the effect of Magnesium Sulphate (MgSO<sub>4</sub>) over compressive strength of the samples. If one observes in **Figure 7**, by and large, cement mortar strength quality increased irrespective of age, the equivalent can be observed in M0 examples of no MgSO $_4$  fixation.<sub>,</sub> Compressive strength qualities decreased as for time and focus with less than 4% fixation, and 4% centralization of MgSO4 has resulted in the greatest quality decrease.

M5 blend examples equivalents the above and were provided in **Figure 8**. Concerning the concentration and age of  $MgSO<sub>4</sub>$ , M0 blend examples get altogether result in quality decrease compared to M5 blend examples Also the M0's compressive strength for 4% was noted to be 57.30 MPa for 120 days, 53.55 MPa for 240 days, 48.50 MPa for 260 days and finally 44.16 MPa for 480 days. In the similar context, the strength values of M5 were 78.26 MPa for 120 days, 74.1 MPa for 240 days, 67.0 MPa for 260 days and finally 64.0 MPa for 480 days. The contrast of strength characteristic between M0 and M5 was 21, 20.4, 20.3 and 19.7 MPa respectively.



**Figure 6.** *Efficacy of nanosilica upon strength (Flexural).*



**Figure 7.** *Corollary of MgSO4 over Sample M0.*



**Figure 8.** *Corollary of MgSO4 over Sample M5.*

#### *3.4.2 Manoeuvre of X-ray diffraction*

The blended samples of OPC's M0 and M5 were hydrated for 28 days and the samples were kept in water with 4% of MgSO<sub>4</sub> for a span of 360 days. The results of the sample M0 and the sample M5 were represented as charts in the **Figure 9** where magnesium silicate hydrate was presented at 48°; at 18°, the hydroxide of calcium turned up and at 33°, magnesium's hydroxide was presented and finally the silica hydroxide of calcium was shown at 26°. It was noted that the hydroxide of calcium exhibited a high force of ascent and, additionally, the lower power ascent of silicate hydroxide of calcium of M0 diversified from M5. The contrast happened because of nanosilica quality. Also, hydroxide and silicate hydrate of magnesium were directed because of the reaction of MgSO4 with hydroxide and silicate hydroxide of calcium.. The quality of silicate hydrate of magnesium declined due to the non-binding nature, but M5 exhibits a finer standard when collated with M0 due to the inclusion of nanosilica.

#### *3.4.3 Manoeuvre of scanning electron microscope*

The blended samples of OPC's M0 and M5 were hydrated for 28 days and the samples were kept in water with 4% of  $MgSO<sub>4</sub>$  for a span of 360 days. The results



**Figure 9.** *Depicting M0 & M5 samples XRD (Sample placed for 360 days in water with 4% MgSO4).*



**Figure 10.**



were represented as Scanning Electron Microscope diagrams in the **Figures 9** and **10** where hydroxide of calcium, silicate hydroxide of calcium, hydroxide of magnesium and silicate hydrate of magnesium were presented. Calcium hydroxide's needle structure was indicated in the **Figure 10** and further, it was made in contrast with the **Figure 11** whereas the content of calcium silicate hydroxide is high when compared to **Figure 10**. In further addition, magnesium's silicate hydrate and hydroxide witnessed a fine and more elegant outlook when collated with **Figure 11**. The inclusion of NS shows diversified nature of OPC. Also, placing the sample in water mixed with  $MgSO_4$  adds up to the work.

#### **3.5 Corollary of hydrochloric acid**

**Figures 12** and **13** render the effect of Hydrochloric Acid (HCl) over compressive strength of the samples. By and large, concrete mortar quality expanded irrespective of age, the equivalent can be seen at sample M0 which was placed in zero HCl fixations. It was also seen that a decline in compressive quality with concentration and time in **Figure 12** when M0 blend examples kept in less than 4% convergence of HCl and examples were given for HCl centralization with 4% has the greatest quality decrease.



**Figure 11.** *Depicting M5's SEM diagram (Sample placed for 360 days in water with 4% MgSO4).*



**Figure 12.**



**Figure 13.** *HCl Corollary over Sample M5.*

The equivalent to above can likewise be seen in M5 blend examples which appear in **Figure 13**. Yet, M0 blend examples get noteworthy quality decrease contrasted with that of M5 blend examples. M0's and M5's compressive quality for a focus of 4% was is 55.35 MPa and 77.33 MPa for 120 days, 49.00 MPa and 72.30 MPa for 240 days, 42.60 MPa and 65.55 MPa for 260 days and 37.31 MPa and 56.30 MPa for 480 days. The differentiation in strength at 4% fixation for 120 days was 21.3 MPa, 240 days was 23.25 MPa, 260 days was 22.82 MPa and for 480 days was 18.96 MPa.

#### *3.5.1 Manoeuvre of X-ray diffraction*

The blended samples of OPC's M0 and M5 were hydrated for 28 days and the samples were kept in water with 4% of HCl for a span of 360 days. The results were represented in the **Figure 14** where hydroxide of calcium, silicate hydroxide of calcium, chlorides of calcium, Friedel's mixes of salt of M0 sample and M5 sample were presented individually at 18°, 26°, 33°, 37° and finally at 48.5°. It was notified that the ascent power of calcium hydroxide is high whereas the force ascent of silicate hydroxide of calcium is more for M5 than M0. The observed variation in ascent of M0 sample and M5 sample was majorly due to the expansion taking place in cement mortar due to the inclusion of NS. Adding to it, the reaction of hydroxides of calcium with HCl and chlorides of calcium with  $C_3A$  frames the chlorides of calcium and Friedel's mixes of salt. Silicate hydroxide of calcium got destabilized due to its reaction with HCl resulting a decline in the quality of strength but the performance of M5 was way too good when collated to that of M0 because of essence of NS in cement mortar.

#### *3.5.2 Manoeuvre of scanning electron microscope*

The blended samples of OPC's M0 and M5 were hydrated for 28 days and the samples were kept in water with 4% of HCl for a span of 360 days. The results were represented as Scanning Electron Microscope diagrams in the **Figures 15** and **16** where hydroxides of calcium, silicate hydroxides of calcium, chlorides of calcium were shown. A significant improvement in case of above mentioned compounds can be noted in the **Figure 15** when contrasted with the **Figure 16**. The diagrams of Scanning Electron Microscope test uphold the investigation of X-ray Diffraction.



**Figure 14.** *Depicting M0 & M5 samples XRD (Sample placed for 360 days in water with 4% HCl).*



#### **Figure 15.**

*Depicting M0's SEM diagram (Sample placed for 360 days in water with 4% HCl).*



#### **3.6 Assessment of temperature corollary**

Temperature's corollary on the blend samples of M0 and M5 were shown in the **Figure 17**. It was evidently clear that an increase in temperature of M0 and M5 samples results the compressive strength to lower. The strength values noted for M0 sample and M5 sample at various temperature conditions are 62.93 MPa and 84.12 MPa at 27 °C, 51.90 MPa and 73 MPa at 400 °C, 28.30 MPa and 48 MPa at 600 °C, 15.40 MPa and 28.41 MPa at 800 °C. The M0 sample and M5 sample exhibits decrease in compressive strength quality because of between layer water, losing free water and synthetically reinforced water. Furthermore warm extension of cement mortar and was unique. The quality decreases in case of compressive strength for sample of M0 and sample of M5 was critical over a temperature of 400 °C. But, more compressive quality was exhibited by M0 blend examples than M5 blend examples.





### **4. Conclusion**

The strength qualities (compression and flexural) expanded by and large due to the NS of 40 nm in case of M5 blend. Cement's extraordinary pozzolonic property and its property as a good material for filling of NS instigated the rise in strength with cement and brilliant filling material.

NS has quickened setting measure because of its enormous surface area. M5 mix examples have given much better execution in solidness in different environmental conditions than that of M0 blend examples.



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