Influence of nano SiO$_2$ in cement mortar using taguchi analysis

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Abstract. In this research, optimization technique was employed to optimize the nano SiO$_2$ in cement mortar preparation by using Taguchi-technique. The main aim of this present investigation is to find the optimal input variables like nano SiO$_2$, w/c ratio and plasticizer to achieve cement mortar with high compressive strength and low water absorption percentage. When the input variables are more than one Taguchi -technique approach is a best method since it may give difference set of optimum level for each response. It is found that the addition of nano SiO$_2$, in cement mortar influenced high compressive strength with lower water absorption percentage. According to analysis nano-silica of 2wt. %, w/c ratio of 0.5 and plasticizer of 1wt. % (A2, B3 and C2) is found to be producing highest compression strength of 51.45MPa and lower water absorption percentage of 2.95.

1. Introduction

Cement is the most sustainable raw material in building industry with ever-increasing demand and performance [1]. New types of cement have been developed over the years as an alternative to conventional cements to increase the performance of conventional cement [2]. To enhance long term performance and adhesive properties the nano-particles could be added with cement in considerable volume [3]. This is because the growing demand for use in nano materials in recent years has brought growing popularity. Nano-oxides, ash fly, slag, and silica fume have been applied by many researchers to strengthen materials dependent on and without cement [4]. Ltifi et al. (2011) added the amorphous nano silica particle at a rate of 3 and 10% by mass of cement. According to author’s conclusion the nano-SiO$_2$ improves the strength of cement pastes and develops the process of cement hydration. Moreover, the compression strength increases with increase of nano-SiO$_2$ content [5]. Rahmani et al. (2012) found the abrasion strength increased during compression testing, the increase in water-cement ratio of micro silica concrete resulted in greater compressive strength [6]. Senff et al. [7] analysed the effect of silica fume (SF) and nano-silica (nS) on range of flow table, effective apparent porosity, and compressive strength and water absorption, un-restrained condition of shrinkage and weight loss of mortars up to 28 days using factorial design based experiments approach. Mortar with 7% of nano-silica exhibited quicker formation of structures in rheological test. The apparent porosity, compressive strength and water absorption are improved with the addition of nS 0-7%, 0-20% of SF and 0.35-0.59 of water/binder ratio. Similar study was done by Manzoor et al. [8]. The author has pointed out the use of RSM in optimizing concrete specimens by increasing compressive strength and decreasing RCPT and sorptivity. Liu et al. [9] calculated the effect of CaCO$_3$ nano particles (NC) with 2% and 1% fractional replacement of cement. It was verified that the rise in NC lowered the flow rate and shortened the specified time. Nonetheless, NC had no effect on standard cement quality water requirement. At the
age of seven days and twenty-eight days, the bending strength and compression strength of toughened cement paste with NC is maximized and the optimum NC level was only 1%.

A review of nano technology in concrete was made by Sanchez et al. [3]. The author examined the advantages of nano technology in concrete technology and its applications in modern structural industries. Moreover the results indicated that nanotechnology can boost concrete quality and promote the improvement of new sustain and progressive cement composites with improved mechanical, thermal and electrical conductivity properties. Mosaberpanah et al. [10] attempted an experimental work with the use of nano silica the ultra-high performance concrete (UHPC) strength was increased via RSM optimization. The RSM results indicated that the accumulation of nano-silica improved the drying shrinkage and 28 days compressive strength and the reduction in the flow-ability.

While the flow-ability of fresh UHPC was reduced due to the addition of nano-silica. It is found that the addition of as received nano-silica particle in cement mortar forming agglomeration during settling process. This agglomeration could initiate crack development and reduce the compression strength of mortar [8]. To avoid agglomeration and ensure uniform settling of nano-silica particle, surface modification could be an effective and needful process [11]. In surface-treatment the local attraction between nano-silica is reduced via silane attachment. This silane attachment just covers the nano-silica as a coat and prevents clustering effect. This phenomenon may improve the performance of nano particle addition in base material [12, 13]. It is known that the addition of nano-silica could improve the compression strength of mortar however; high water absorption behavior of nano-silica could affect the life of cement mortar. In surface-modification process the nano-silica is converted as a hydrophobic one from hydrophilic nature. This phenomenon would influence good compression strength along with low water absorption in cement mortar. Hence, it could be the add-on advantage for modern infrastructure industries. Because making of high compressive strength cement mortar with low water absorption yield cost effective and economical process compatibility.

Taguchi optimization could be an effective tool when more number of input variables is employed in any engineering process. In this method different set of optimum level for each response could be identified at the end of optimization. The processing and mathematical modelling of Taguchi method is simple and effective on compare with other optimization techniques. Muthuramalingam et al. [14] have done an optimization study for micro machining process to optimize the process variables. According to author’s conclusion the Taguchi optimization was the effective and fastest tool to find out the optimal value of inputs and finding of dominant variable. Similarly, Jayakrishnan et al. [15] optimized the end milling of aluminium alloy input parameters using Taguchi optimization technique. The authors concluded that the Taguchi optimization is the suitable method when multi responses are arrived from the particular process. Thus the previous literature revealed that there are lot of efforts have been made by many researchers for developing different types of micro and nano particle blended cement mortar. However, very few research works is presented based on surface modified nano addition along with optimization.

Based on previous literature there are lot of efforts have been made by many researchers for developing different types of micro and nano particle blended cement mortar. However, very few research works is presented based on Taguchi-technique optimization for optimizing the process variables in new type cement mortar. Based on the research gap the present study is designed. The chief aim of this research work is to examine and optimize the influence of input process parameters and to find optimum blending proportions of cement mortar with nano silica, water cement ratio (w/c) and plasticizer using Taguchi-technique analysis. Using design expert software (DES) the model built was checked for statistical adequacy and predictive reliability by means of variance analysis (ANOVA). These optimized process parameter cement mortar could be used as highly sustainable construction material for today’s economical world [16]. Few research are followed in silane surface - treatment method [17, 18].
2. Experimental work

2.1. Materials

The ordinary port-land cement (OPC) was used. The OPC measures a specific gravity and fineness of 3.1 and 230 m²/Kg. The cement paste used here was about 31% of standard consistency. A fine aggregate with a specific gravity of about 2.62 and fineness modulus of 3.98 was obtained for this study. Polycarboxylate was used as a super plasticizer (SP) and potable water was used for mixing nano silica of size 20nm with purity of 99.44%.

2.2. Cement Mortar and specimen making

The experimental work was carefully designed and to study the characteristics of nano SiO₂ on the cement mortar properties of different water cement ratios. Super plasticizer content was maintained constant for the entire range of mix. The nano SiO₂-mortar strength depends on the water cement ratio and the nano SiO₂ percentage on mix. The mix proportion adopted was C: FA=1:3 of all kinds. The samples were prepared as total content of cement was 200gms and sand 600gms, w/c ratio of 0.48, 0.50 and 0.52 and super-plasticizer content of 0.5 and 1wt. %. Polycarboxylate was solidified in water and the silica nano particles were added and stirred at a high speed for about 2 to 3 minutes. This mixture was added with cement mortar and filled with ASTM cube specimen (50mm ×50mm ×50 mm) using a vibrating table [19].

2.3. Testing of samples

The prepared cement mortar samples were tested for void content and compressive strength. The void content percentage was calculated using theoretical and measured density values according to ASTM C 1754. For void content test the w/c ratio of 0.5, plasticizer of 1wt. % and nano SiO₂ content of 0, 1.5 and 3wt. % was used as process parameters. However, the compressive strength of nano-silica toughened cement mortar is measured using a compressive strength tester according to ASTM C 109. Figure 1 shows the prepared cubes tested machine setup. Similarly, the water absorption percentage was calculated using ASTM C 1585. However, the compression strength and water absorption percentage of cement mortar was optimized using Taguchi technique design with 9 trials to find the optimum w/c ratio, plasticizer content and nano SiO₂ since these two parameters plays a major role in structure-property relationship. Table 1 shows the selection of process parameters and levels.

![Figure 1. Cement mortar testing machine](image_url)
Table 1. Process parameters and levels

| Parameter         | Symbol | Level 1 | Level 2 | Level 3 |
|-------------------|--------|---------|---------|---------|
| Nano SiO₂ (wt %)  | A      | 1       | 2       | 3       |
| W/C ratio (%)     | B      | 0.4     | 0.45    | 0.5     |
| Plasticizers (%)  | C      | 0.5     | 1       | 1.5     |

3. Taguchi Method

Taguchi method was used to determine the optimal combination of input parameters in a systematic approach. In this approach L9 orthogonal array was used to find the optimal parameter. Since the 3 input parameters and the L9 array was deployed to execute the optimization process. Based on this L9 totally nine different kinds of cement mortar was prepared and tested for compression strength and water absorption percentage. The compression strength was calculated using a compression tester whereas the water absorption was found using the weight of cubes before and after immersion in water. The quality analysis of the output responses are valued by signal to noise ratio (S/N). This ratio is measured from the responses using suitable formulae. There are two categories of S/N ratios. They are smaller the better and larger the better. According to this present investigation the main aim is to improve the compression strength with minimal water absorption percentage. That means compressive strength is considering as larger the better whereas water absorption is consider as smaller the better. Equation 1 and 2 shows the S/N ratio of smaller the better and larger the better formulae.

For smaller the better

\[ S/N \text{ ratio} = -10 \log \left( \frac{1}{n} \right) + (Yij^2) \]  

For larger the better

\[ S/N \text{ ratio} = -10 \log \left( \frac{1}{n} \right) + \left( \frac{1}{Yij^2} \right) \]  

Where,

- Yij – Values of response, n - Each experiment number of replication say ‘1’

Table 2. Design factor of L9 Orthogonal array and S/N ratio for Compressive strength and Water absorption

| Experimental Trail | Nano SiO₂ (wt%) | W/C ratio (%) | Plasticizers (%) | Compressive strength | Water absorption | S/N ratio for CS | S/N ratio for WA |
|--------------------|----------------|---------------|------------------|----------------------|------------------|------------------|------------------|
| L1                 | 1              | 0.4           | 0.5              | 35.80                | 5.29             | 31.08            | -14.47           |
| L2                 | 1              | 0.45          | 1                | 36.86                | 5.11             | 31.33            | -14.16           |
| L3                 | 1              | 0.5           | 1.5              | 37.05                | 5.05             | 31.38            | -14.07           |
| L4                 | 2              | 0.4           | 1                | 45.90                | 3.27             | 33.24            | -10.30           |
| L5                 | 2              | 0.45          | 1.5              | 43.22                | 3.98             | 32.71            | -12.00           |
| L6                 | 2              | 0.5           | 0.5              | 49.66                | 3.02             | 33.92            | -9.60            |
| L7                 | 3              | 0.4           | 1.5              | 41.55                | 3.92             | 32.37            | -11.86           |
| L8                 | 3              | 0.45          | 0.5              | 42.57                | 3.80             | 32.58            | -11.60           |
| L9                 | 3              | 0.5           | 1                | 50.21                | 3.45             | 34.02            | -10.76           |
For this present study compression strength is larger the better and for water absorption smaller the better approach was used. This consideration is assumed since for a good cement mortar the compressive strength should be higher with minimal water absorption percentage. Table 2 depicts the L9 orthogonal array for response, the normalized S/N ratio and S/N ratio values calculated from the experimental response.

4. Results and discussion

This research work analyses and studies the process parameters effect on water absorption and compressive strength of cement mortar composites. The parameters are nano SiO\textsubscript{2} (A), w/c ratio (B), plasticizers (C) at three levels as shown in table 1. The degrees of freedom (DOF) for process factors were found to be 8 (DOF = 9−1 = 8).

The hardness and tensile strength was found using Taguchi method by using 16 investigational conditions [20]. Table 3 and 4 depicts the response table for compressive strength and water absorption. The results showed that nano silica have correlation on cement mortar strength [16]. The better performance was observed for compressive strength and water absorption at higher S/N ratio as shown in Figure 2 and Figure 3. Due to the addition of surface-modified nano-silica particle of 2wt. % in cement mortar, which could fill in the voids of mortar and reduces the shrinkage and improves the compression strength [21]. The response value A\textsubscript{2}, B\textsubscript{3}, C\textsubscript{2} at Optimum process condition are represented in table 2.

| Level | Nano SiO\textsubscript{2} (wt %) | W/C ratio (%) | Plasticizers (%) |
|-------|---------------------------------|---------------|------------------|
| 1     | 31.26                           | 32.23         | 32.53            |
| 2     | 33.29                           | 32.21         | 32.86            |
| 3     | 32.99                           | 33.10         | 32.15            |
| Delta | 2.03                            | 0.89          | 0.71             |
| Rank  | 1                               | 2             | 3                |

The optimized process parameters for compressive strength and water absorption are nano SiO\textsubscript{2} at level 2, w/c ratio at level 3, and the plasticizers at level 2.

| Level | Nano SiO\textsubscript{2} (wt %) | W/C ratio (%) | Plasticizers (%) |
|-------|---------------------------------|---------------|------------------|
| 1     | -14.23                          | -12.21        | -11.89           |
| 2     | -10.63                          | -12.59        | -11.74           |
| 3     | -11.41                          | -11.47        | -12.64           |
| Delta | 3.60                            | 1.11          | 0.90             |
| Rank  | 1                               | 2             | 3                |
4.1. Analysis of variance

The experimental analysis and modelling results are obtained using the taguchi method that is a combination of arithmetical and computational techniques, which are used for analysis of results. The ANOVA table specifies 98.46% and 98.43% of significant level in compressive strength and water absorption behavior.

ANOVA table is determined by the total sum of squares of the standard deviation equal to the sum of squares of the standard deviation produced by the respective parameter [15]. Tables 5 and 6 represents the ANOVA results for compressive strength and water absorption respectively. It determines the F-ratio and P-value. The process parameter, the influence of nano SiO<sub>2</sub> plays a significant role in determining the mechanical behaviour. Compressive strength and water absorption improved by 20.33% and 21.92% respectively. Highest contribution of 88.76% was observed due to Nano SiO<sub>2</sub>, W/C ratio and plasticizers were 18.86 and 9.05 % contributions.
Table 5. ANOVA variance table for Compressive Strength.

| Source            | DF | Seq SS  | Contribution(%) | Adj SS  | Adj MS   | F-Value | P-Value |
|-------------------|----|---------|-----------------|---------|----------|---------|---------|
| Nano Silica (wt%) | 2  | 163.451 | 71.05           | 163.451 | 81.726   | 68.48   | 0.014   |
| W/C ratio (%)     | 2  | 43.383  | 18.86           | 43.383  | 21.692   | 18.18   | 0.052   |
| Plasticizers (%)  | 2  | 20.820  | 9.05            | 20.820  | 10.410   | 8.72    | 0.103   |
| Error             | 2  | 2.387   | 1.04            | 2.387   | 1.193    |         |         |
| Total             | 8  | 230.041 | 100.00          |         |          |         |         |

(R-Seq 98.96%)

Table 6. ANOVA variance table for Water absorption.

| Source            | DF | Seq SS  | Contribution(%) | Adj SS  | Adj MS   | F-Value | P-Value |
|-------------------|----|---------|-----------------|---------|----------|---------|---------|
| Nano Silica (wt%) | 2  | 5.09332 | 88.76           | 5.09332 | 2.54666  | 56.36   | 0.017   |
| W/C ratio (%)     | 2  | 0.32939 | 5.74            | 0.32939 | 0.16470  | 3.65    | 0.215   |
| Plasticizers (%)  | 2  | 0.22524 | 3.93            | 0.22524 | 0.11262  | 2.49    | 0.286   |
| Error             | 2  | 0.09036 | 1.57            | 0.09036 | 0.04518  |         |         |
| Total             | 8  | 5.73831 | 100.00          |         |          |         |         |

(R-Seq 98.43%)

4.2. Confirmation Test

Furthermore, the confirmation test verified the improvement of results and to predict the optimum performance at the selected levels (since all factors have a confident level more than 90%) of significant parameters such as A2, B3 and C2. Table 7 shows the comparison between the actual and experimental results. The predicted mean (M) values can be expressed as,

\[ M = (A2-T) + (B3-T) + (C2-T) + T, \]

Where T= average of S/N ratio (Compressive strength) \( \rightarrow 32.514 \)

\[ M= (33.29-32.514) + (33.10-32.514) + (32.86-32.514) + 32.514 \]

(Compressive strength) M= 34.222 dB

Similarly, T = average of S/N ratio (Water absorption) \( \rightarrow -12.091 \)

\[ M = ((-10.63)-(-12.091)) + ((-11.47)-(-12.091)) + ((-11.74)-(-12.091)) + (-12.091) \]
(Water absorption) M= -9.658 dB

The predicted mean of the response S/N ratio for the compressive strength lies in the range of 31.08 dB < Compressive strength > 34.22 dB at the confidence level of 95%.

The predicted mean of the response S/N ratio for the Water absorption lies in the range of -9.60 dB < Water absorption > -14.47 dB at the confidence level of 95%.

**Table 7.** The comparison between predicted and actual results of compressive strength and water absorption

| Output               | Level      | Response      | Conformation | Experimental | Difference |
|----------------------|------------|---------------|--------------|--------------|------------|
| Compressive strength | A2, B3, C2 | Observed result (MPa) | 51.45       | 49.66       | 1.79       |
|                      |            | S/N Ratio in dB | 34.222      | 33.92       | 0.302      |
| Water absorption     | A2, B3, C2 | Observed result (%) | 2.95        | 3.02        | 0.007      |
|                      |            | S/N Ratio in dB | -9.658      | -9.60       | 0.058      |

4.3. Regression analysis

The regression equation of compressive Strength = 42.536 - 5.966 A1 + 3.724 A2 + 2.241 A3 - 1.453 B1 - 1.650 B2 + 3.103 B3 + 0.141 C1 + 1.788 C2 - 1.929 C3

The regression equation of water absorption = 4.0987 + 1.050 A1 - 0.675 A2 - 0.375 A3 + 0.061 B1 + 0.198 B2 - 0.259 B3 - 0.061 C1 - 0. C2 + 0.217 C3

5. Conclusions

In this present work the influence of nano SiO2 in cement mortar were studied using the Taguchi technique. The following conclusions were drawn

- A2 B3 C2 combination represents 2% nano SiO2 W/C ratio 0.50% and plasticizers 1% obtain improved mechanical properties.

- From ANOVA table, it was observed that the effect of nano SiO2 has a better improvement on compressive strength and water absorption.

- The highest maximum value for compression strength and minimum value for water absorption was found.

- The confirmation test evidenced that the optimum combination satisfied the actual requirement of process parameters to produce high performance cement mortar.
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