Optimization of bentonite modified cement mortar parameters at elevated temperatures using RSM

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Abstract: The inaccessibility of industrial wastes necessitated the search for natural pozzolanic materials. This paper deals the optimization of bentonite utilization in cement mortar at elevated temperatures using response surface methodology. The substitution of calcined bentonite (0%, 10%, 20% and 30%) and temperature exposure (24°C, 200°C, 400°C and 600°C) were taken as variables. The compression strength and strength activity tests for all mixes were carried out for bentonite. Design Expert 11.0 version was utilized for modelling using RSM. The peak compressive strength was displayed by 20% replaced bentonite calcined at 200°C cement mortar after 28 days curing. Strength activity was improved upon increasing the percentage of bentonite calcined at 200°C. The generated models from RSM are significance in all the factors considered. Optimum solutions were proposed with more than 0.90 desirability.

Keywords: Bentonite, compressive strength, elevated temperatures. Response Surface Methodology and optimization.

1. Introduction:

Utilization of pozzolanic materials will improve the properties of cement mortar and concrete [1]. Presently, industrial wastes are utilizing as a pozzolanic materials in concrete to enhance the performance [2]. Fly ash is a waste material from thermal power plants. It is one of the widely using pozzolanic materials in construction industry. The fresh and hardened properties of concrete will improve by inclusion of fly ash in concrete [3, 4]. Fly ash will not be generated once the thermal power plants got shutdown permanently. So, the discovery of alternative to the pozzolanic materials generated from industrial wastes was needed.

Bentonite is a clay, can be used instead of industrial wastes in concrete. A few research was done on the assessment of the properties of bentonite modified cement mortar and concrete [5, 6]. The properties of concrete was enhanced by inclusion of bentonite in concrete [7]. The uncertainty in the impact of bentonite was detected based the source of collection [8]. So, investigation on process of treatment of bentonite is needed to estimate the influence on bentonite [9, 10]. Calcination is a method of heating a material under regulated temperature and in a monitored environment. A few studies were made to assess the effect of calcined Bentonite on the properties of cement mortar and concrete which is available in Pakistan [11, 12].

Optimization included the utilization of Response Surface Methodology (RSM) using statistical methods between variables and responses [13]. RSM was used for the optimization of the required set of objectives, like variables or responses, to execute multi-objective optimization in various concrete
materials [14]. Bashar et al. performed multi-objective optimization to accomplish a connection between variables and responses of the properties of roller-compacted concrete by keeping fly ash content as constant, considering the combined effect of both crumb rubber and nano-silica [15]. This research aimed at the optimization of calcined bentonite usage at elevated temperature in cement mortar by using RSM.

2. Experimental Program

2.1 Materials

OPC 53 grade cement was used, the properties are in the allowable limits as per the standard procedure IS12269. Sand sand was utilized in the determination of compressive strength, the properties are in the allowable limits as per the standard procedure IS650. Bentonite was gathered form Tandur (17°14ʹ27ʺN and 77°35ʹ14ʺE), southern region of India. The calcination of the bentonite was performed with muffle furnace. The chemical composition of bentonite was displayed in Table 1.

| S. No. | Component | % Mass |
|--------|-----------|--------|
| 1      | SiO₂      | 51.11  |
| 2      | Al₂O₃     | 16.38  |
| 3      | CaO       | 7.12   |
| 4      | MgO       | 7.57   |
| 5      | Fe₂O₃     | 7.65   |
| 6      | K₂O       | 1.34   |
| 7      | Na₂O      | 0.29   |
| 8      | P₂O₅      | 0.29   |
| 9      | MnO₂      | 0.14   |
| 10     | V₂O₅      | 0.07   |
| 11     | TiO₂      | 1.29   |
| 12     | LoI*      | 6.75   |

* Loss on Ignition

2.2. Testing of specimens

A total number of 189 cement mortar cubes were cast and tested to failure after and 28 days curing after keeping at 24°C, 200°C, 400°C and 600°C for 3 hours in muffle furnace as per the standard procedure IS 4031 Part-6 as displayed in Figure 1. Testing of all specimens was done with compression testing machine of maximum capacity 3000 kN and rate of loading as 1kN/Sec, Figure 2 displays the test setup. The strength activity index test was conducted as per the standard procedure ASTM C311. The sorpitivity test was conducted as per the standard procedure ASTM C1585 – 20. The proportions of all mixes were calculated as per the standard procedure IS 4031 Part-6, displayed in Table 2.

| S. No | Percentage | Cement | Calcined Bentonite | Standard Sand |
|-------|------------|--------|--------------------|---------------|
| 1     | 0          | 200    | 00                 | 600           |
| 2     | 10         | 180    | 20                 | 600           |
| 3     | 20         | 160    | 40                 | 600           |
| 4     | 30         | 140    | 60                 | 600           |
Figure 1. Muffle Furnace

Figure 2. Compression strength test setup
3. Results and discussion

Analysis was performed for the values taken after testing the specimens of all mixes as per the standard procedures.

3.1 Compressive strength
The compressive strength of all mixes was calculated, the results were summarized in figure 3 after exposed 24°C, 200°C, 400°C and 600°C at 28 days of curing. The cement mortar exhibits highest compressive strength for 20% bentonite substitution among all mixes. Very less effect was observed at 200°C temperature. Compressive strengths decreased drastically for all mixes at 600°C. This attribute the pore filling effect between cement particles by bentonite addition, the particle size of calcined bentonite was less than cement. The secondary C-S-H gel formation at lateral ages of cement mortar because bentonite is a pozzolanic material.

3.2 Strength Activity Index
Strength activity index of all specimens was calculated at 28 days curing. The summary of results was displayed in Figure 4. More strength activity index was seen for the cement mortar replaced by 20% bentonite. The behavior is almost similar with compressive strength of all mixes. This may be because of pozzolanic activity occurrence upon addition of calcined bentonite.

![Figure 3. Compressive strength of all mixes](image)
4. RSM analysis

Various types of models are available like Central Composite, Box Behnken and optimal (custom) in a randomized design in RSM. Selecting a suitable model depends on the type of available data and levels for each factor. The central composite type was used for the development and investigation of a model.

4.1 Mix matrix design

In this investigation, Design Expert 11.0 version was used. The design of experiments was created by a composite design technique based on two variables (exposed temperature and bentonite replacement). Four levels of exposed temperature (Room Temperature (RT), 200°C, 400°C and 600°C) and four levels % of bentonite replacements (0%, 10%, 20% and 30%) were used. 16 combinations of mixtures were developed in RSM. Table 3 represents the details of all combination of variables. The responses of calcined bentonite blended concrete (compressive strength and strength activity index) were determined for all mixtures, considered for RSM analysis and optimization.

4.2 Analysis of variance

The summaries of ANOVA for the responses analyzed, displayed in Table 4 and 5. F-values are 107.65 and 87.1 for compressive strength and pointing all models to be significant. The cubic model was used for compressive strength and strength activity index. For compressive strength and strength activity index, few terms are insignificant term and interaction in the quadratic model. The final models for workability, compressive strength, strength activity index and sorptivity of all mixes consist of all the terms are given in Equations. 1-2, respectively. 3-dimensional (3D) response surface plot was used to illustrate the relationship between responses and independent variables. Figure 5-6 shows the 3D response surface plots illustrating the relationship between responses and independent variables for all mixes.

Compressive Strength = +55.47+2.91 A-0.8000 B+0.0107 AB-2.23 A²-0.3344 B²+0.1201 A²B-0.0588 AB²-3.47 A³-0.1442 B³ → 1

Strength Activity = +103.20+5.41 A +0.8438 B+0.0160 AB-4.15 A²-0.2937 B²+0.1621 A²B-0.1116AB²-6.47 A³ -1.07 B³ → 2
Table 3. Details of all combination of variables

| S. No | Bentonite (in percentage) | Temperature (In degree Celsius) | Compressive Strength (In MPa) | Strength Activity (Percentage) |
|-------|---------------------------|--------------------------------|-------------------------------|--------------------------------|
| 1     | 0                         | 24                             | 54.37                         | 100.00                         |
| 2     | 10                        | 24                             | 54.97                         | 101.10                         |
| 3     | 20                        | 24                             | 56.58                         | 104.06                         |
| 4     | 30                        | 24                             | 53.17                         | 97.79                          |
| 5     | 0                         | 200                            | 54.17                         | 100.00                         |
| 6     | 10                        | 200                            | 54.57                         | 100.74                         |
| 7     | 20                        | 200                            | 56.38                         | 104.07                         |
| 8     | 30                        | 200                            | 52.77                         | 97.41                          |
| 9     | 0                         | 400                            | 53.37                         | 100.00                         |
| 10    | 10                        | 400                            | 54.17                         | 101.50                         |
| 11    | 20                        | 400                            | 55.98                         | 104.89                         |
| 12    | 30                        | 400                            | 52.36                         | 98.12                          |
| 13    | 0                         | 600                            | 52.77                         | 100.00                         |
| 14    | 10                        | 600                            | 53.17                         | 100.76                         |
| 15    | 20                        | 600                            | 54.57                         | 103.42                         |
| 16    | 30                        | 600                            | 51.56                         | 97.72                          |

4.3 Optimization
Optimization was employed with the aim of getting an optimized variable of all bentonite mixes. Optimization was performed by using Design Expert Software. Table 6. Shows optimization results of bentonite mixes. The optimized mix was achieved at 20.36 % of substitution of bentonite and 435°C temperatur. The optimum values of responses were 55.65 MPa compressive strength and 104.53 strength activity index.
Figure 5. 3D response surface plot for R:1 Compressive Strength

Figure 6. 3D response surface plot for R:2 Strength Activity
Table 5. ANOVA for R:1 Strength Activity

| Source     | Sum of Squares | df | Mean Square | F-Valve | p-value  |
|------------|----------------|----|-------------|---------|----------|
| Model      | 31.22          | 9  | 3.47        | 107.65  | < 0.0001 significant |
| A-Bentonite| 5.25           | 1  | 5.25        | 162.76  | < 0.0001 |
| B-Temperature| 0.4187      | 1  | 0.4187      | 12.99   | 0.0113   |
| AB         | 0.0006         | 1  | 0.0006      | 0.0177  | 0.8985   |
| A²         | 15.65          | 1  | 15.65       | 485.76  | < 0.0001 |
| B²         | 0.3452         | 1  | 0.3452      | 10.71   | 0.0170   |
| A²B        | 0.0255         | 1  | 0.0255      | 0.7925  | 0.4076   |
| AB²        | 0.0059         | 1  | 0.0059      | 0.1841  | 0.6828   |
| A³         | 7.63           | 1  | 7.63        | 236.84  | < 0.0001 |
| B³         | 0.0137         | 1  | 0.0137      | 0.4255  | 0.5384   |
| Residual   | 0.1934         | 6  | 0.0322      |         |          |
| Cor Total  | 31.42          | 15 |             |         |          |

Table 6. Optimized calcined bentonite mixes

| Bentonite percentage | Temperature | Compressive strength | Strength activity | Index  |
|----------------------|-------------|----------------------|-------------------|--------|
| 20.36                | 435.09      | 55.65                | 104.53            |        |

5. Conclusions

The following conclusions were made based on experimental and statistical analysis, The maximum compressive strength was observed for 20 % bentonite substitution and 24°C temperature. Strength activity was improved, best strength activity was observed for the mix made with 20 % bentonite substitution and at 240°C. The model was prepared, and optimization was done by using RSM. The optimized values of variables were determined as 20.36 % bentonite substitution and 435.09°C temperature. The optimum values of responses were 55.65 MPa compressive strength and 104.53 strength activity index.

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