Optimization and influence of parameter affecting the compressive strength of geopolymer concrete containing recycled concrete aggregate: using full factorial design approach

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Abstract. There are several parameters that influence the properties of geopolymer concrete, which contains recycled concrete aggregate as the coarse aggregate. In the present study, the vital parameters affecting the compressive strength of geopolymer concrete containing recycled concrete aggregate are analyzed by varying four parameters with two levels using full factorial design in statistical software Minitab® 17. The objective of the present work is to gain an idea on the optimization, main parameter effects, their interactions and the predicted response of the model generated using factorial design. The parameters such as molarity of sodium hydroxide (8M and 12M), curing time (6hrs and 24 hrs), curing temperature (60°C and 90°C) and percentage of recycled concrete aggregate (0% and 100%) are considered. The results show that the curing time, molarity of sodium hydroxide and curing temperature were the orderly significant parameters and the percentage of Recycled concrete aggregate (RCA) was statistically insignificant in the production of geopolymer concrete. Thus, it may be noticeable that the RCA content had negligible effect on the compressive strength of geopolymer concrete. The expected responses from the generated model showed a satisfactory and rational agreement to the experimental data with the $R^2$ value of 97.70%. Thus, geopolymer concrete comprising recycled concrete aggregate can solve the major social and environmental concerns such as the depletion of the naturally available aggregate sources and disposal of construction and demolition waste into the landfill.

Keywords: Geopolymer Concrete, Recycled concrete aggregate, Factorial design, curing condition, molarity of sodium hydroxide and Compressive Strength.

1. Introduction
In 1978, Davidovits suggested that an alkaline solution could be employed to react with the silicon (Si) and the aluminum (Al) to form a solid matrix. The polymerization action incorporates a significant accelerated chemical reaction under alkaline medium on Si-Al minerals, emerging in a three dimensional polymeric group and ring framework consisting of Si-O-Al-O bond [1]. The polymerisation process of geopolymer concrete are controlled by various parameters such as curing temperature, curing time, molarity of sodium hydroxide, ratio of alkaline liquid-to-fly ash, ratio of Si to Al, water content of the mixture, method of mixing the ingredients and method of curing [2-4]. In the past, many studies were conducted on geopolymer concrete comprising natural coarse
aggregate (NCA), but studies on geopolymer concrete by full replacement of natural coarse aggregate with recycled concrete aggregate (RCA) is reported minimum. The parameters varied in the previous studies are the molarity of sodium hydroxide [5-8], percentage RCA content [9-13], curing temperature [7] and method of curing (Ambient and heat curing) [5]. It was reported that strength of geopolymer concrete containing RCA declines with the addition of RCA [9-13]. However, variation in the curing time and combined analysis of the parameters such as molarity of sodium hydroxide, curing time, curing temperature and percentage of RCA content with optimization for a particular compressive strength value using full factorial design of experiments has been not yet reported.

2. Experimental Program

2.1 Production of Geopolymer Concrete

Geopolymer concrete was prepared using Class F low calcium fly ash obtained from Udupi thermal power plant, sodium silicate, sodium hydroxide (NaOH), river sand, NCA and RCA. Crushed concrete samples from the concrete laboratory of original compressive strength ranging from 25 – 35 MPa was used as a source of RCA and further crushed using lab model jaw crusher. The aggregates passing 10 mm sieve size and retained on 4.75 mm sieve size were used as the Coarse Aggregate. The properties of NCA and RCA are given in the Table 1.

| S. No. | Properties | NCA | RCA |
|--------|------------|-----|-----|
| 1      | Specific gravity | 2.85 | 2.65 |
| 2      | Water absorption(%) | 0.33 | 1.33 |
| 3      | Percentage adhered mortar(%) | 0 | 33.33 |
| 4      | Bulk density(kg/m³) | 1452 | 1434 |
| 5      | Abrasion value(%) | 26 | 30.5 |
| 6      | Impact value(%) | 32.82 | 30.69 |

The composition of Na₂SiO₃ was Na₂O= 15.2%, SiO₂ = 29.7% and H₂O =55.1%. The density was found to be 1.53gm/cm³. The NaOH solution was taken in the form of flakes of 98% purity which was procured from the local market. The NaOH solution of specific molarity was produced 24 hours earlier to use, as the addition of NaOH flakes and tap water, liberates large amount of heat. The Na₂SiO₃ and NaOH solutions were blended half an hour before the casting of the specimens. Conplast SP 430 based on Sulphonated Naphthalene polymer was used as high range water reducing Superplastizer with a dosage of 2% by the weight of fly ash. The quantity of materials required per cubic meter are tabulated in Table 2. Conventional mixing procedure was followed for producing geopolymer concrete. The fresh concrete was then casted in cube moulds of 100mm size. The cubes along with the mould were cured in hot air drying oven and tested after placing in a room temperature of 24 hours.

| S. No. | RCA % | Coarse aggregate | Fine aggregate | Fly ash | Sodium silicate | NaOH | Superplastizer |
|--------|-------|------------------|---------------|---------|----------------|------|---------------|
| 1      | 0     | 1201.2           | ----          | 646.8   | 380.7          | 122.4 | 48.95         | 7.614 |
| 2      | 100   | 1201.2           | 646.8         | 380.7   | 122.4          | 48.95 | 7.614         |

2.2. Factorial design of experiments

In this study, 2⁴ full factorial design analysis (four parameters and two control levels) was performed with 16 trials of experiments using Minitab® 17 statistical software. The major advantage of the factorial design was the precise estimation of the main effects of each factor and their interactions in fewer experiments [14]. Parameters and the levels used in 2⁴ full factorial design is shown in Table 3. The geopolymer synthesis parameter such as molarity of sodium hydroxide (8M and 12M), curing
time (6hrs and 24 hrs), curing temperature (60°C and 90°C) and percentage of recycled concrete aggregate (0% and 100%) and the effects of interactions between those parameters were examined. The experimental data were analyzed using second ordered interaction and the regression model form is shown in equation (1).

\[ Y = \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_4D + \beta_{AB} + \beta_{AC} + \beta_{AD} + \beta_{BC} + \beta_{BD} + \beta_{CD} \]  

(1)

Where Y is the value of the response, \( \beta_0 \) is a constant coefficient and \( \beta \) terms specify the regression coefficient estimates of main and interaction effects. The A, B, C and D are notations for the parameters as indicated in Table 3.

### Table 3. Parameters and the levels used in 2^4 full factorial design

| Notations | Parameters            | Type  | Parameter Level            |
|-----------|-----------------------|-------|---------------------------|
| A         | Molarity of sodium    | Numerical | Low level (-1) | High level (1) |
|           | Hydroxide(M)          |       | 8                         | 12             |
| B         | Curing time(Hours)    | Numerical | 6                         | 24             |
| C         | Curing temperature(°C)| Numerical | 60                        | 90             |
| D         | RCA content(%)        | Numerical | 0                         | 100            |

### 3. Results and discussion

#### 3.1 Effect of parameters on the strength of geopolymer concrete using analysis of variance (ANOVA)

The significant parameters affecting the strength of geopolymer concrete were obtained by performing analysis of variance (ANOVA). It is a statistical approach which separates the total variance in an entire data into various parts linked to definite sources of variations for the objective to test the hypotheses on the factors used in the model [14]. ANOVA results for the full factorial model are presented in Table 4. The P-values lesser than 0.05 indicates the parameters are statistically significant. Thus from the Table 4, the parameters namely the molarity of sodium hydroxide, curing time and curing temperature in the one way interaction and the combination of factors, namely molarity of Sodium hydroxide * RCA content and curing time * curing temperature in the two way interaction are statistically significant indicating their major effects on the strength of geopolymer concrete. These results are further justified by the Pareto charts and normal plot of the standardized effect as shown in Figure 1.

### Table 4. Analysis of variance (ANOVA)

| Source            | DF | Adj SS  | Adj MS  | F-Value | P-Value |
|-------------------|----|---------|---------|---------|---------|
| Model             | 10 | 2953.65 | 295.37  | 20.93   | 0.002   |
| Linear            | 4  | 2669.91 | 667.48  | 47.29   | 0.000   |
| A                 | 1  | 991.78  | 991.78  | 70.27   | 0.000   |
| B                 | 1  | 1582.65 | 1582.65 | 112.13  | 0.000   |
| C                 | 1  | 93.94   | 93.94   | 6.66    | 0.049   |
| D                 | 1  | 1.54    | 1.54    | 0.11    | 0.754   |
| 2-Way Interactions| 6  | 283.74  | 47.29   | 3.35    | 0.103   |
| A*B               | 1  | 3.16    | 3.16    | 0.22    | 0.656   |
| A*C               | 1  | 24.53   | 24.53   | 1.74    | 0.245   |
| A*D               | 1  | 102.57  | 102.57  | 7.27    | 0.043   |
| B*C               | 1  | 152.71  | 152.71  | 10.82   | 0.022   |
| B*D               | 1  | 0.78    | 0.78    | 0.06    | 0.824   |
| C*D               | 1  | 0.00    | 0.00    | 0.00    | 0.991   |
| Error             | 5  | 70.57   | 14.11   |         |         |
| Total             | 15 | 3024.22 |         |         |         |
3.1.1 Main effect. The main effect shows variations of the mean among the high and low values of each factor. The magnitude of slope represents the intensity of the effects that each factor exerts. When the slope is positive, the response increases for higher levels of that factor and vice-versa [15]. The main effect plot for the mean strength of geopolymer concrete is shown in Figure 2. It is seen that all the factors show a slope of positive response increase to the higher level. The mean strength of geopolymer concrete reduces gradually in the order of curing time > molarity of NaOH > curing temperature > RCA content. As compared to other parameters, curing time parameter with steeper slope plays a major role in strength enhancement of geopolymer concrete. Thus, prolonged curing for 24 hours may boost up the geopolymeric reaction (polymerization reaction) and forms a stronger bond between alumina (Al) and silica (Si) compounds. The molarity of sodium hydroxide is the second parameter which played a vital part in the disintegration (leaching) of Al and Si atoms. By the dissolution process, the free Al and Si atoms are released in the solution, enhancing the reaction mechanism pertaining to the strength increase at higher molarity. The curing temperature ranks third in influencing the strength of geopolymer concrete. The strength variation at higher temperature is not as identical as compared to the curing time and molarity of NaOH solution. The possible reasons may be, at higher temperature the breakdown of the Al and Si bonds occurs with the evaporation of the water (OH ions). The last parameter, namely RCA has negligible influence on the strength of geopolymer concrete with minimum slope line. But, this result is contradictory to the previous research work predominantly by the decrease in strength, as the RCA content is increased to 100% [9-13]. The possible reasons are the use of good quality RCA as tabulated in Table 1 with the properties of the RCA are almost similar to NCA.
3.1.2 Interaction effect. The interaction effect indicates the combined interactive effect of two parameters, when a response of one parameter (low to high) depends on the level of a second parameter. Graphically, two parallel lines of parameters indicate no interaction between them, whereas non-parallel lines suggest that the two parameters interact together. From the Figure 3, it is noted that the molarity of Sodium hydroxide * RCA content and curing time * curing temperature interacts with each other showing non parallel lines. Further, the mean strength for all the interaction parameters increase from lower level to higher level.

![Interaction Plot for Compressive strength](image)

**Figure 3.** Interaction effect for the mean compressive strength

3.2 Full factorial model and optimization of the parameters

An empirical regression model equation (second-order) was generated using the experimental data as shown in equation (2) and the strength of geopolymer concrete was predicted with an $R^2$ value of 97.7%. The fitted line plot between the experimental and predicted values of the geopolymer concrete strength is shown in Figure 4. A response optimization tool from Minitab software was used to optimize the parameters for the goals of maximum and target strengths of geopolymer concrete as shown in the Table 5.

Compressive strength = - 30.4 + 2.48 A + 3.044 B + 0.091 C + 0.251 D - 0.0247 AB + 0.0413 AC - 0.02532 AD - 0.0228 BC + 0.00049 BD + 0.00001 CD \ (2)
Table 5. Optimization of parameters for target and maximum compressive strength

| S. No | RCA content (%) | Molarity of NaOH (M) | Curing time (Hrs) | Curing temperature (°C) | Compressive strength (MPa) | Goals   |
|-------|-----------------|----------------------|------------------|------------------------|---------------------------|---------|
| 1     | 0               | 9.35                 | 6                | 60                     | 30.0                      | Target  |
| 2     | 100             | 8.55                 | 6                | 60                     | 30.0                      | Target  |
| 3     | 0               | 12                   | 24               | 90                     | 68.6                      | Maximum |
| 4     | 100             | 12                   | 24               | 90                     | 64.6                      | Maximum |

3.2.1 Goodness of model fit. Figure 5 shows the goodness of model fit by checking the normality and equal variance assumptions. The normal probability plot is a graphical description for concluding whether the data are allocated normally indicated by the closer accumulation of points [16]. Figure 5a shows the data are assigned normally, as all the points are closer to the straight line.

The residual values describe the changes among the predicted values (model) and the experimental values. Standardized residuals greater than +2 and less than -2 are usually considered as outliers. Figure 5b shows the spread of residual values in the plots are within this limits and on both sides of 0 indicating no outliers in the data. Thus the predicted model specified an insignificant divergence in the fitted value from the experimental value [17].
3.3 Validation of full factorial model

The factorial model generated for the particular set of parameters with the predicted response are checked with the new intermediate level of a particular parameter, namely the molarity of sodium hydroxide (10M). Other parameters such as curing time, curing temperature and RCA content were kept constant. The predicted compressive strength using the generated model is compared with the experimental value and the percentage deviations are tabulated in table 6. It is noted that the deviation of the predicted compressive strengths from the experimental value are within the acceptable range of (± 6 %), thereby validates the generated model.

| S. No | Molarity of NaOH (M) | Curing time (Hrs) | Curing temperature (°C) | RCA Content (%) | Compressive strength (MPa) Predicted Value | Experimental Value | Deviation (%) |
|------|---------------------|------------------|-------------------------|----------------|------------------------------------------|-----------------|--------------|
| 1    | 10                  | 24               | 90                      | 0              | 57.41                                    | 60.12           | -4.72        |
| 2    | 10                  | 24               | 90                      | 100            | 58.50                                    | 62.08           | -6.12        |
| 3    | 10                  | 24               | 60                      | 0              | 58.80                                    | 57.3            | 2.55         |
| 4    | 10                  | 24               | 60                      | 100            | 59.80                                    | 61.69           | -3.16        |
| 5    | 10                  | 6                | 90                      | 0              | 44.13                                    | 52.81           | -6.07        |
| 6    | 10                  | 6                | 90                      | 100            | 44.34                                    | 41.57           | 6.25         |
| 7    | 10                  | 6                | 60                      | 0              | 33.13                                    | 40.4            | -3.83        |
| 8    | 10                  | 6                | 60                      | 100            | 33.30                                    | 31.83           | 4.41         |

4. Conclusion

The $2^4$ full factorial design of experiment was successfully used to generate prediction model for geopolymer concrete. The expected responses of the generated model showed a satisfactory and rational agreement to the experimental data with the $R^2$ value of 97.70%. The analysis of variance (ANOVA) implies that the curing time and the molarity of the sodium hydroxide were the most significant parameters affecting the strength of geopolymer concrete. The percentage of RCA content was statistically insignificant and had a negligible effect on the compressive strength of geopolymer concrete. The set of parameters required to develop the maximum strength of geopolymer concrete containing 100% RCA content is as follows: Molarity of NaOH = 12M; Curing time = 24 Hrs; Curing temperature = 90°C. Thus, it may be inferred that the 100% replacement of natural coarse aggregate with recycled concrete aggregate does not impact the strength of geopolymer concrete. Overall, geopolymer concrete comprising recycled concrete aggregate can solve the major social and environmental concerns such as the depletion of the naturally available aggregate sources and disposal of construction and demolition waste into the landfill.

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