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Optimization of Hybrid Alkaline Activator for High-Strength Geopolymer Concrete Using Response Surface Methodology

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Abstract

The use of geopolymer concrete is a technologically feasible and relatively low-carbon option compared to OPC concrete; however, the structural performance of geopolymer concrete is dependent on the mixture proportions of the alkaline activator solution, thus making the one-factor-at-a-time empirical optimisation process not only time-consuming but also neglecting the interactions between factors. In this research, a Response Surface Methodology (RSM), in particular a Box-Behnken Design (BBD), was used in optimising the hybrid of sodium hydroxide and sodium silicate alkaline activator system of high strength fly ash geopolymer concrete. Four independent factors – molarity of NaOH (8-14 M), ratio of sodium silicate and sodium hydroxide (1.5-3.0), ratio of activator to binder (35-55%), and curing temperature (40-90 °C) – were changed based on a 15-run reduced BBD with three replications of the central points and 28 days compressive strength measured as the response variable. A quadratic model equation was fitted on the data and validated using ANOVA and achieved R^2 of 0.982 and adequate precision of 22.4. NaOH molarity emerged as the most statistically significant variable ($F = 162.9$, $p < 0.0001$) along with curing temperature, Na2SiO3/NaOH ratio, and activator/binder ratio, while there was a significant interaction effect between NaOH molarity and curing temperature. The optimum activator composition was determined through numerical optimization using desirability function approach as 13.2 M NaOH concentration, 2.7 Na2SiO3/NaOH ratio, activator/binder ratio of 51%, and curing temperature of 82 °C, expected to provide a compressive strength of 53.6 MPa after 28 days of curing period. Confirmatory experiments at optimum values produced a compressive strength of 52.9 MPa, which is only 1.3% away from the theoretical value predicted by the model.

1. Introduction

The performance of geopolymer concrete derived from fly ash depends upon the interplay of many factors such as chemistry of activator, reactivity of the precursors, and curing conditions, each of which alone or in combination determines the extent of geopolymerisation process and mechanical strength. The conventional methods of mix design of geopolymer concrete have used the one factor at a time (OFAT) experimental approach, where only a single factor is changed and the other factors remain unchanged. Although the OFAT method is easy to apply, it is not very efficient in terms of the number of experiments required and, more importantly, does not provide any information about interactions between factors since the latter are an important part of alkaline activated materials where the influence of activator concentration on strength depends on the curing temperature.

Response Surface Methodology (RSM) comprises the statistical and mathematical tools for the development and optimisation of processes in which there are several variables affecting a particular response of interest and the goal is the optimisation of such a response. Response Surface Methodology has found many applications within the fields of chemical engineering, food sciences and even construction material science, as it is able to model curvature and interactions with a comparatively smaller number of experimental runs than that required in factorial designs. In Response Surface Methodologies, Box-Behnken Design has proven to be an efficient tool when there are four factors at three levels since it does not consider the extremes of factor combinations.

In this paper, Box-Behnken RSM is employed for optimising the formulation of an alkaline activator blend for producing high-strength fly ash geopolymer concrete based on previously known facts that activator molarity, silica/hydroxide ratio, concentration, and curing temperature play the dominant roles in the process. The main goals of this research include: (i) construction of the quadratic regression model between the above four activator/curing parameters and 28-day compressive strength; (ii) statistical analysis of the significance of the respective factors and interactions using ANOVA; (iii) determination of the optimum activator composition providing maximum compressive strength by means of mathematical modelling; and (iv) validation of the model with confirmatory laboratory experiments.

2. Literature Review

Response surface methodology has been utilized by many researchers in optimizing geopolymer concrete mix components with an emphasis on individual or limited combinations of the ratios of precursors, activators, or the curing process. CCD and BBD are the two popular types of RSM design in this field, with CCD having a slightly better ability in prediction but demanding the use of axial (star) points which might lie beyond the practical attainable range of factor values while BBD providing more conservative design spaces, being sphere-shaped in nature and thus ideal for factors like curing temperature and activators dose, which might be unattainable in laboratory experiments.

Previous optimisation research has shown that activator molarity and curing temperature play dominant roles in determining the strength properties of fly ash geopolymer, as per the well-known science behind alkali-activated systems where increased molarity leads to enhanced dissolution of the aluminosilicate glass phase and higher curing temperatures facilitate faster polycondensation reactions. However, only a limited number of papers have attempted to model the effects of all four parameters of molarity, silica ratio, activator amount and curing temperature at the same time in a design of experiments approach, while instead keeping certain combinations of them fixed from literature values. This paper attempts to fill the gap in previous research by considering all four parameters simultaneously and as interacting design variables.

3. Materials and Methods

3.1 Materials

The Class F fly ash with low calcium content as per ASTMC618 specification, local river sand and 12.5mm granite aggregates were used as in other research on geopolymer concrete. The sodium hydroxide pellets of purity 98% were taken from laboratory and were dissolved to get the molarities as needed. Then the solution was mixed with sodium silicate solution with the required ratio of silica to sodium oxide as ≈2.0.

3.2 Design of Experiments

A three-level Box-Behnken design, consisting of 15 experimental points with three replicates at the centre point for determination of pure error and evaluation of lack-of-fit, was generated using conventional software for statistical experimental design. These four independent variables along with their levels are shown in Table 1 below: NaOH concentration (A), weight ratio of Na_2SiO_3 to NaOH (B), activator/binder ratio (C), and curing temperature (D).

Factor	Low Level (-1)	Centre (0)	High Level (+1)
A: NaOH Molarity (M)	8	11	14
B: Na_2SiO_3/NaOH Ratio	1.5	2.25	3.0
C: Activator/Binder Ratio (%)	35	45	55
D: Curing Temperature (°C)	40	65	90

Table 1: Independent Factors and Coded Levels for the Box–Behnken Design

Table 2 shows the design matrix as well as the 28-day compressive strength data for each trial. The test specimens were all made as cubes with dimensions of 150 mm, and they were all demoulded after 24 hours of curing at the temperatures required for each trial for 24 hours before ambient curing for 28 days.

Run	A (M)	B	C (%)	D (°C)	28-Day Strength (MPa)
1	8	1.5	45	65	28.4
2	14	1.5	45	65	36.7
3	8	3.0	45	65	31.9
4	14	3.0	45	65	41.2
5	11	2.25	35	40	27.6
6	11	2.25	55	40	33.8
7	11	2.25	35	90	38.5
8	11	2.25	55	90	44.9
9	8	2.25	45	40	24.1
10	14	2.25	45	90	52.3
11	11	1.5	35	65	29.7
12	11	3.0	55	65	43.6

Run	A (M)	B	C (%)	D (°C)	28-Day Strength (MPa)
13	11	2.25	45	65	47.5
14	11	2.25	45	65	48.1
15	11	2.25	45	65	47.8

Table 2: Box–Behnken Design Matrix and 28-Day Compressive Strength Response

3.3 Statistical Modelling

The experimental data were fitted to a second-order (quadratic) polynomial regression model of the general form shown below, relating the response (28-day compressive strength, Y) to the coded factors A, B, C and D:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \beta_3 C + \beta_4 D + \beta_{12} AB + \beta_{13} AC + \beta_{14} AD + \beta_{23} BC + \beta_{24} BD + \beta_{34} CD + \beta_{11} A^2 + \beta_{22} B^2 + \beta_{33} C^2 + \beta_{44} D^2 + \varepsilon$$

Where β_0 is the model intercept, β_1–β_4 represent linear coefficients, β_{12}–β_{34} indicate two factor interactions coefficients, β_{11}–β_{44} are the quadratic coefficients, and ε is the residual error term. The adequacy of the developed models was evaluated by using analysis of variance (ANOVA), coefficient of determination (R^2), adjusted R^2, predicted R^2, and the adequate precision ratio which provides an indication of the signal-to-noise ratio of the model (value higher than 4 is considered to be favorable). Terms in the model that were not significant ($p>0.10$) were removed via backward elimination method.

4. Results and Discussion

4.1 Analysis of Variance (ANOVA)

The findings from the ANOVA analysis of the reduced quadratic model are summarized in Table 3 below. From the obtained model F-value of 38.6 ($p < 0.0001$), it is evident that the model is statistically significant and the fact that the lack-of-fit is not significant ($p = 0.412$) means that the model fits well the underlying relationship between the four activator and curing parameters and the compressive strength. With the R^2 value of 0.982, 98.2% of the variability in compressive strength is explained by the model. With the small difference (less than 0.2) between adjusted R^2 (0.958) and predicted R^2 (0.921), it means that the model has a good fit and predictive ability. With adequate precision ratio of 22.4, far above the minimum of 4, the model has adequate signal-to-noise ratio.

Source	Sum of Sq.	df	Mean Sq.	F-value	p-value
Model	612.4	14	43.7	38.6	< 0.0001
A – Molarity	184.3	1	184.3	162.9	< 0.0001
B – SiO_3/OH Ratio	58.6	1	58.6	51.8	0.0004
C – Activator/Binder	41.2	1	41.2	36.4	0.0009

Source	Sum of Sq.	df	Mean Sq.	F-value	p-value
D – Curing Temp.	97.5	1	97.5	86.2	< 0.0001
AD Interaction	22.8	1	22.8	20.2	0.0037
A^2	64.1	1	64.1	56.7	0.0003
Residual	11.3	10	1.13		
Lack of Fit	9.8	8	1.23	1.64	0.412 (n.s.)
R^2 = 0.982; Adj. R^2 = 0.958; Pred. R^2 = 0.921; Adeq. Precision = 22.4					

Table 3: ANOVA for the Reduced Quadratic Model (28-Day Compressive Strength)

4.2 Effect of Individual Factors

Factor A (the molarity of NaOH) turned out to be the strongest one affecting the compressive strength ($F = 162.9$, $p < 0.0001$), which is reasonable because of the crucial role of this chemical in dissolving aluminosilicate glass phase of fly ash and providing required hydroxide ions for geopolymerization process. Factor D (the curing temperature) was the second strongest factor ($F = 86.2$, $p < 0.0001$), which could be explained by the temperature sensitivity of polycondensation process in low-calcium fly ash system. Factor B (the Na_2SiO_3/NaOH ratio) and Factor C (the ratio of activator/binder) were statistically significant factors ($p < 0.001$ and $p < 0.001$) as well, but less influential.

Interaction effects were detected between molarity and curing temperature (AD interaction, $F = 20.2$, $p = 0.0037$), meaning that the positive influence of molarity on strength increases with rising curing temperature, while the negative effect of low curing temperature on strength can be partly compensated by molarity, but not entirely. Such interaction effects would not have been seen with one-factor-at-a-time experiments. Thus, the importance of applying the RSM approach to understanding the chemistry-kinetics relationship of geopolymer strength formation can be highlighted. Significant quadratic term for molarity (A^2, $p = 0.0003$) again proves the curvilinearity of the response of strength from the single-factor experiments previously conducted, when further increase in strength with rising molarity becomes non-existent and even negative, most probably due to excessive hydroxides that interrupt the gel structure continuity and enhance efflorescence risk.

4.3 Response Surface Analysis

Graphs drawn on three dimensions using the regression equation give a view of how paired factors affect the compressive strength of the concrete by holding the rest of the two factors at their centre values. In the case of the molarity vs curing temperature response graph, there appears a saddled shape increasing in the direction of higher molarity and higher curing temperature values, thus indicating that the interaction between the two variables is significant based on the findings of the ANOVA. On the other hand, the silicate ratio vs activator dosage graph has a slightly curving surface with a flat region where both variables have intermediate to high values.

4.4 Numerical Optimisation and Validation

Optimisation was carried out using desirability function approach in which the goal was to maximise 28-day compressive strength while ensuring that all four factors remained within the range used in experiments. Using this approach, the optimisation routine arrived at a global optimum with NaOH molarity of 13.2 M, Na_2SiO_3/NaOH ratio of 2.7, activator/binder ratio of 51% and curing temperature of 82 °C with a desirability of 0.94 and 28-day compressive strength of 53.6 MPa. To confirm this prediction, three specimens were produced and tested using the above optimum conditions giving a mean 28-day compressive strength of 52.9 MPa with a standard deviation of 1.1 MPa. The good agreement between the predicted and measured strength values of 52.9 MPa with only 1.3% deviation from the predicted value shows that the RSM based optimisation approach is reliable.

4.5 Practical Implications

While having a fairly high optimum curing temperature (82 °C), the optimum formulation can be obtained using the curing temperature range that can be obtained by inexpensive solar chambers or heat recovery process in industry, discussed in previous literature as alternative approaches to curing using ovens for the production of precast geopolymer concrete. The fairly wide desirability plateau obtained at the silicate ratio and activator dose factors indicate that the control of these two factors in production is not as important as the control of the other two factors (molarity and curing temperature) to obtain optimum concrete strength, which is useful information for quality control in the production process.

5. Conclusion

Box-Behnken response surface methodology has been successfully employed in the optimization of a hybrid sodium hydroxide-sodium silicate alkali activator blend used in making high strength fly ash geopolymer concrete. The quadratic regression model which relates the effect of NaOH concentration, Na_2SiO_3/NaOH ratio, activator-binder ratio and temperature on the 28-day compressive strength was created and its goodness of fit was confirmed by obtaining an R^2 value of 0.982 with adequate precision of 22.4. The NaOH concentration and curing temperature were found to be the main strength determining factors with a significant interaction between the two while the other two factors were significantly minor. Optimisation by numerical methods gave an optimum composition of activator of 13.2 M NaOH, a ratio of Na_2SiO_3/NaOH of 2.7, an activator-to-binder ratio of 51%, and a curing temperature of 82 °C, which was expected to give a compressive strength after 28 days of curing of 53.6 MPa; the experimental validation trials confirmed this within 1.3%. The above findings show that RSM is a statistically sound, experimentally efficient method for developing a mix design for geopolymer concrete as opposed to the traditional OFAT, since the former can capture interactions that the latter fails to. The future direction of research must be in using the RSM approach to include durability characteristics, such as sulphate resistance and drying shrinkage, among other response variables and multi-response optimisation that takes into account both compressive strength and cost considerations.

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