

APPLICATION OF RESPONSE SURFACE METHODOLOGY IN THE OPTIMIZATION OF FLY ASH GEOPOLYMER CONCRETE

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A series of research experiments was designed and conducted in this study using the Box–Behnken design method of response surface methodology (RSM) to solve the optimization problem of parameters effectively in fly ash geopolymer concrete preparation. First, single-factor gradient analysis was adopted to determine the reasonable level of various factors in the response surface analysis. The 28-day compressive strength development was investigated in terms of the water–binder ratio, dosage of alkali, unit water dosage, and sodium silicate modulus. Results showed that the order of the factors in terms of their influence on concrete strength was dosage of alkali, sodium silicate modulus, and water–binder ratio, and the unit water dosage exerted a minimal influence. Second, the preparation parameters were optimized to improve the 28-day compressive strength of the concrete based on the single-factor analysis using the RSM. The optimum parameters were a water–binder ratio of 0.35, an alkali dosage of 7.9%, and a sodium silicate modulus of 1.66. This study also analyzed the response surface optimization results through a validation test to prove the effectiveness of the RSM in optimizing the preparation of geopolymer concrete.

Keywords: fly ash; geopolymer concrete; strength; preparation parameters; response surface methodology

1. Introduction

Geopolymer is a new promising inorganic polymer material that has many advantages [1-2], such as excellent mechanical properties and durability and energy conservation and environmental protection features. Considerable progress has been made in the research of geopolymer in recent years, and many valuable research results have been obtained in the fields of mix proportion of activator [3-5], curing conditions [6-8], preparation technology [9-11], mechanical properties [12-14], and durability [15-17]. However, the research on the application of geopolymer as cementitious material in concrete engineering remains limited. Cementitious material is an intermediate product, and its engineering value and material performance can be maximized better when applied in concrete engineering. The preparation technology and performance of geopolymer concrete remains a field to be exploited. Although several scholars have produced geopolymer concrete with high strength through testing [18-20], the research remains insufficient and shallow.

To prepare geopolymer concrete, the preparation parameters should be determined first. However, the experimental design methods of

existing research involve a single-variable or simple multi-factor combination experiment and an orthogonal design to determine the optimum value through a certain number of tests [21-23]. Such research focuses only on a few isolated and scattered points and could not establish a definite functional relationship between the influence factors and the response value within the entire value region of the independent variables of influencing factors to find the optimal mix proportion. In addition, the interaction among various factors could not be analyzed by the methods. Response surface methodology (RSM) uses the results of the experimental data obtained by the scientific experiment design to conduct regression analysis and establishes the functional relationship between the influence factors and the response value [24-26]. The continuous response surface model is then obtained. Combined with mathematical and statistical knowledge that consider multiple factors simultaneously, the model can approximately reflect the real function of the relationship between the factors and the response value. Within the range of the independent variable values of various influence factors, the response surface model needs to find the optimal combination through which the response value reaches its target value, thus optimizing the

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experimental program or product formula. Compared with orthogonal design and neural network, RSM has a relatively small number of test groups, thus saving manpower, reducing material consumption, reducing costs, and improving efficiency. Experimental RSM is widely used in biological, chemical, machinery, and agricultural fields, and it is an effective method to improve product performance during actual production. Nevertheless, the application of RSM is unusual in the field of civil engineering, particularly in the research on parameter optimization for building material preparation. This study uses the RSM to design experiments, investigates the relationship between concrete strength and various influence factors, and establishes the regression model between the influence parameters and concrete strength by analyzing the experimental results. This study determines the concrete optimal preparation parameters through optimization analysis and verifies the optimization results through a verification test. A convenient, practical, and effective method is proposed to optimize the parameters for preparing geopolymer concrete.

2. Materials and methods

2.1. Materials

Fly ash was utilized as raw material in the research, considering that the early activity of fly ash is low at ambient temperature, and geopolymerization requires heat curing to obtain high efficiency usually, this method not only consumes large amounts of energy but is also unsuitable for on-site construction operations. Therefore, a certain proportion of slag and cement is added to the raw materials due to its high activity, the slag and cement can quickly be dissolved by strong alkali at ambient temperature, then alkali excitation reaction occurs and the main hydration products are hydrated calcium silicate gel, hydrated calcium aluminate and calcium hydroxide etc, which provides not only the early strength of materials, but also the nucleating matrix for aluminosilicate which generated from fly ash in alkali activator solution, then prompts the further formation of a 3D network structure of geopolymer,

and accelerates the speed of geopolymerization of fly ash. In this way, the materials can achieve good setting speed and high early strength at ambient temperature.

Combined with the previous research results of this study [27], the cementitious material composition was 60% fly ash, 25% slag, and 15% cement. Fly ash with a density of 2.12 g/cm³ and fineness of 10.8% was obtained from Fuxin Power Plant in China. Slag with a density of 2.68 g/cm³ was obtained from Fuxin Jinfujiye Concrete Co. Ltd. OPC with a grade of 42.5 conforming to the Chinese standard GB175-2007 was applied in the test. The raw materials in research are shown in Fig. 1, and the chemical compositions and loss of ignition of materials are presented in Table 1.

A combination of sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) was utilized as the alkaline activator in this study. Commercially available sodium hydroxide (NaOH) with purity higher than 96% was used. Commercially available industrial-grade sodium silicate (Na₂SiO₃) with 8.97% Na₂O, 29.16% SiO₂, and 61.87% water was also utilized. The required modulus was obtained by adding NaOH solution during the test.

Table 1

Chemical compositions (Wt. %) of materials

Composition	Fly ash	Slag	Cement
SiO ₂	53.75	33.16	22.56
Al ₂ O ₃	29.37	15.33	4.64
CaO	3.68	37.15	61.28
Fe ₂ O ₃	5.64	1.36	2.35
MgO	1.08	9.07	2.04
Na ₂ O	-	0.36	0.6
K ₂ O	0.68	0.39	0.75
SO ₃	1.29	1.27	2.83
LOSS	3.58	0.91	2.01

River sand from the town of Baiyudu in Fuxin with a density of 2.57 g/cm³ was used as the fine aggregate. Gravel from the town of Gongguanyingzi in Fuxin with specification of 5 mm to 31.5 mm and a density of 2.78 g/cm³ was used as the coarse aggregate. The sand percentage in this study was 33%. Tap water was used as the the concrete mixing water. The polycarboxylate superplasticizer produced by the Tsingtao Hongsha High Polymer Material Limited Company was utilized in this study at 1.2%.



Fig. 1 - Raw materials in research: (a) Fly ash, (b) Slag, and (c) Cement.

2.2. Test method

2.2.1. Compressive strength test of concrete

In reference to the fly ash geopolymer concrete compressive strength test indicated in *Standard for test method of mechanical properties on ordinary concrete* (GB/T 50081-2002), geopolymer concrete samples were prepared as follows. The test materials, which were in accordance with the preliminary mix proportion design, were mixed fully in a concrete forced mixer before casting in 100 mm × 100 mm × 100 mm steel molds. The samples were subjected to vibration to remove air bubbles. Thereafter, the samples were demolded and stored in a standard curing room at 20±2 °C and with more than 95% relative humidity until the age of 28 d. Compressive strength tests were then performed.

2.2.2 Univariate analysis

The experimental point area in the experimental design should include the best experimental conditions to optimize the preparation parameters effectively using the RSM. Thus, we must first conduct a single-factor experimental study to determine the reasonable affecting factors and the experimental value interval. In accordance with the existing research and the results of early experiments, water–binder ratio, alkali dosage (percentage rates for total mass of Na₂O in alkaline activator to fly ash mass), unit water dosage, and sodium silicate modulus were selected as the examination factors. This study conducted a

univariate analysis of each factor using the index of the 28-day compressive strength of geopolymer concrete to determine the influence law of the changes in each factor on the properties of geopolymer concrete and the reasonable level of various factors in the response surface analysis.

2.2.3 Response surface experimental design

Experiments were designed using the Box–Behnken design (BBD) method based on the single-factor analysis. Design-Expert software V8.0.6.1 was used to conduct a response surface regression analysis of the experimental results, in which the 28-day compressive strength was the response variable. The functional relationship was fitted using a secondary polynomial, and the response surface model was established as

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i < j} \beta_{ij} X_i X_j + \sum_{i=1}^k \beta_{ii} X_i^2,$$

where Y is the response value; X is the independent variable of the factors; β₀ represents the intercept; and β_i, β_{ii}, and β_{ij} represent the coefficients of the linear, square, and interactive terms, respectively.

3. Results and discussion

3.1. Single-factor effect analysis

The single-factor experimental results were shown in Fig. 2, and the influence regularities of the 28-day compressive strength under the change of various factors could be found in these figures.

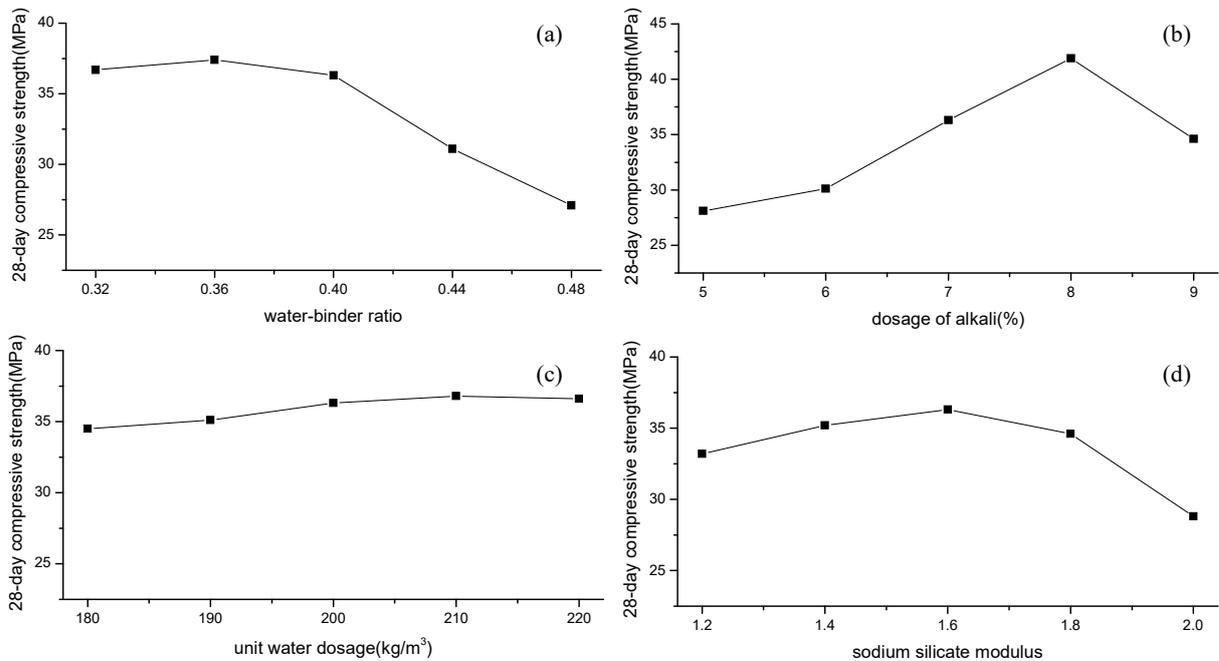


Fig. 2 - The influence regularities of the 28-day compressive strength under the change of various factors of (a) water–binder ratio, (b) alkali dosage, (c) unit water dosage, (d) sodium silicate modulus.

3.1.1 Influence of water–binder ratio

The preparation parameters are set as follows: the alkali dosage is 7%; the unit water dosage is 200 kg/m³; the sodium silicate modulus is 1.6; and the water–binder ratios are 0.32, 0.36, 0.4, 0.44, and 0.48. The regularity of the 28-day compressive strength of fly ash geopolymer concrete is shown in Figure 2 (a). Initially, the strength increases slightly and then decreases continuously with the increasing water–binder ratio. The 28-day compressive strength reaches the maximum value when the water–binder ratio is 0.36, whereas the strength decreases significantly when the water–binder ratio is more than 0.4.

If the water–binder ratio is too small, the concrete mixture will be quite dry, which causes vibration. The water will be less as a reaction medium and could provide a limited reaction scale, which will then have a negative influence on the final concrete strength. When the water–binder ratio becomes too high, the concentration of the alkaline activator declines relatively and, in turn, weakens the geopolymerization power. At the same time, the reduction and bleeding phenomena appear, and the aggregate will not be distributed well in the mixture. A significant amount of water will exist in the concrete mixture and will be sealed inside the concrete or evaporate gradually. A numbers of holes will remain after concrete hardening, which can significantly reduce the concrete strength.

3.1.2. Influence of dosage of alkali

The preparation parameters are set as follows: the water–binder ratio is 0.4; the unit water dosage is 200 kg/m³, the sodium silicate modulus is 1.6; and the alkali dosages are 5%, 6%, 7%, 8%, and 9%. The regularity of the 28-day compressive strength of fly ash geopolymer concrete is shown in Figure 2 (b). The concrete strength decreases with the alkali dosage. By contrast, the concrete strength presents an obvious increase as the alkali dosage gradually increases. The strength of the samples reaches the maximum when the alkali dosage is 8%. Thereafter, the strength decreases significantly as the alkali dosage increases.

The alkaline activator, which is a combination of NaOH and sodium silicate, can provide large amounts of OH⁻, which can dissolve the mixture in strong alkaline environment and the glass-phase composition of fly ash. The fracture of the Si-O and Al-O bonds can also dissolve the active silicon–aluminum ingredient, which leads to the formation of various aluminosilicate oligomers. Polycondensation then reacts to form three-dimensional network aluminosilicate gels that combine with the coarse and fine aggregates, thus leading to the high strength of concrete after hardening. When the alkali dosage and activator concentration are low, the alkali activation force is insufficient. Thus, the polymerization degree of the

product in geopolymerization is low, resulting in the structure being not dense enough after concrete hardening. However, when the dosage of alkali is too high, excessive Na⁺ is adsorbed on the surface of fly ash particles and hinders the further formation of a 3D network structure in the late stage of geopolymerization. At the same time, the condensation rate of the geopolymer cementitious material significantly and rapidly increases. The liquidity of the concrete mixture is reduced significantly when the dosage of alkali is too high. This condition leads to a decrease in strength because of difficult pouring of the concrete mixture, excessive pores, and poor compactness of the sample.

3.1.3. Influence of unit water dosage

The preparation parameters are set as follows: the water–binder ratio is 0.4; the alkali dosage is 7%; the sodium silicate modulus is 1.6; and the unit water dosages (kg/m³) are 180, 190, 200, 210, and 220. The regularity of the 28-day compressive strength of fly ash geopolymer concrete is shown in Figure 2 (c). The concrete strength increases lightly with the unit water dosage, but the scope is generally small.

If the unit water dosage is too small, then the geopolymer cementitious material paste in concrete will be limited and could not wrap around the aggregate. The workability of the concrete mixture is poor, thus weakening the filling effect. The number of holes remain after concrete hardening, which leads to low strength. After the elevation of the unit water dosage, the concrete workability improves obviously, and the homogeneity and the degree of compaction becomes better, which could be advantageous to the improvement of the concrete strength. Overall, the influence of the change in unit water dosage on concrete workability is greater than that on strength.

3.1.4. Influence of sodium silicate modulus

The preparation parameters are set as follows: the water–binder ratio is 0.4; the alkali dosage is 7%; the unit water dosage is 200 kg/m³; and the sodium silicate moduli are 1.2, 1.4, 1.6, 1.8, and 2.0. The regularity of the 28-day compressive strength of fly ash geopolymer concrete is shown in Figure 2 (d). The concrete strength increases and reaches the maximum value when the sodium silicate modulus is 1.6, and decreases obviously as the sodium silicate modulus increases gradually.

The large number of low-silicate polymers in sodium silicate liquid plays an important role in promoting the aluminum silicate polymerization reaction. The low-silicate polymer concentration is relatively low when the sodium silicate modulus is low, which does not satisfy the requirements of

polymerization reaction; therefore, the geopolymer strength is low. The concentration of low-silicate polymers in the solution is improved after the sodium silicate modulus increases, thus promoting the occurrence of the polymerization reaction and increasing the geopolymer strength. However, if the sodium silicate modulus is too large, then the existing form of silicate in the solution is mainly high-silicate polymers, which decreases the proportion of low-silicate polymers in the solution. This case is not conducive to the polymerization reaction, and thus the strength of geopolymer concrete decreases obviously.

3.2. Experimental results and analysis of RSM

In accordance with the results of single-factor analysis, the influence factors that have more influence on the compressive strength of the fly ash geopolymer concrete, such as water–binder ratio, alkali dosage, and sodium silicate modulus, are selected in the research. The three-factor–three-level response surface analysis tests are designed using the BBD method to establish the mathematical model. The response variable (Y) represents the 28-day compressive strength of concrete, and the influence factors X_1 , X_2 , and X_3 represent the water–binder ratio, alkali dosage, and sodium silicate modulus, respectively. The factors and code levels of RSM are shown in Table 2. A group of 17 BBD experiment iterations are designed, as shown in Figure 3, which include 12 factorial analysis test iterations and 5 center point repeat test iterations. The experimental design obtained by the Design-Expert software V8.0.6.1 is shown in Table 3, and the results of test are also shown in Table 3.

The proportion of cementitious material in experiment always consists of 60% fly ash, 25% slag, and 15% cement. The value of sand percentage is 33%, the unit water dosage is 200 kg/m³, and the mixing amount of superplasticizer is 1.2%.

Design-Expert software V8.0.6.1 is used to perform secondary polynomial regression analysis of the data in Table 3. The relationship between the response variable Y (representing the 28-day compressive strength of concrete) and the influence factors, such as X_1 (water–binder ratio), X_2 (dosage of alkali), and X_3 (sodium silicate modulus), is fitted using the following second-order regression equation:

$$Y = -723.125 + 1211.25X_1 + 111.638X_2 + 133.188X_3 - 16.875X_1X_2 - 28.125X_1X_3 - 3X_2X_3 - 1453.125X_1^2 - 6.35X_2^2 - 30X_3^2$$

3.3. ANOVA and analysis of interaction

It can be easily found from analysis of variance that $R^2=0.9905$, showing that the correlation between the predicted and actual values is very good and the regression model coincides well with the test data. It can also be

Table 2

Factors and code levels of RSM.

Factor	Code	Levels of code		
		-1	0	1
water-binder ratio	X_1	0.32	0.36	0.4
dosage of alkali (%)	X_2	7	8	9
sodium silicate modulus	X_3	1.4	1.6	1.8

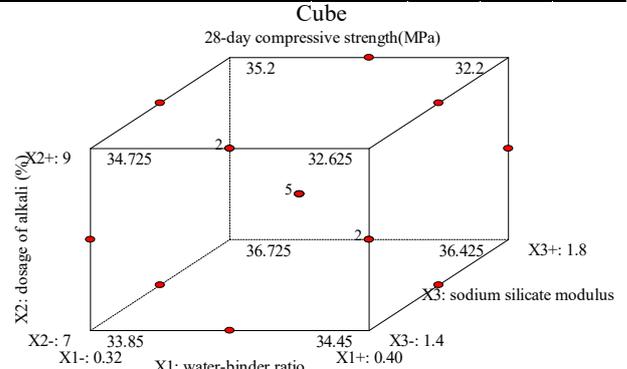


Fig. 3. Three-factor BBD experimental design points distribution.

Table 3

Experimental design of RSM and results

Test number	Design of tests			28-day compressive strength / MPa
	water-binder ratio	dosage of alkali (%)	sodium silicate modulus	
1	0.36	8	1.6	44.1
2	0.32	7	1.6	36.7
3	0.36	9	1.8	36.1
4	0.4	8	1.4	40.3
5	0.36	7	1.8	39.1
6	0.36	8	1.6	44.8
7	0.4	7	1.6	36.3
8	0.36	8	1.6	45.1
9	0.4	8	1.8	40.8
10	0.36	7	1.4	36.4
11	0.32	9	1.6	36.5
12	0.4	9	1.6	33.4
13	0.32	8	1.8	41.9
14	0.36	9	1.4	35.8
15	0.36	8	1.6	43.6
16	0.36	8	1.6	44.4
17	0.32	8	1.4	40.5

seen that adjusted $R^2=0.9783$, indicating that the model can explain response value change of 97.83%. Only 2.17% of the total variance cannot be explained by this model. Fig. 4 displays the relationship between the forecast values and the experimental values (Predicted vs. Actual) which are distributed comparatively adjacent to the straight line. It is seen that the experimental results are in good agreement with the predicted. Fig. 5 is the normal probability distribution diagram of residual, where, they lie rationally close on a straight line suggested the errors are distributed normally and no digression of the variance. Fig. 6 is the pattern of residual and forecast value (Residuals vs. Predicted), the general trend is that the plot is irregularly distributed, suggesting that the model does not show any violation of the independence or the variance is constant for every

Table 4

ANOVA of the model

Source	Freedom	Sum of squares	Mean square	F value	P value
Model	9	226.57	25.17	81.30	< 0.0001
X_1	1	2.88	2.88	9.30	0.0186
X_2	1	5.61	5.61	18.12	0.0038
X_3	1	3.00	3.00	9.69	0.0170
X_1X_2	1	1.82	1.82	5.89	0.0457
X_1X_3	1	0.20	0.20	0.65	0.4453
X_2X_3	1	1.44	1.44	4.65	0.0680
X_1^2	1	22.76	22.76	73.51	< 0.0001
X_2^2	1	169.78	169.78	548.31	< 0.0001
X_3^2	1	6.06	6.06	19.58	0.0031
Residual	7	2.17	0.31	-	-
Lack of Fit	3	0.79	0.26	0.76	0.5719
Pure Error	4	1.38	0.35	-	-
Cor Total	16	228.74	-	-	-

$R^2=0.9905$, $AdjR^2=0.9783$

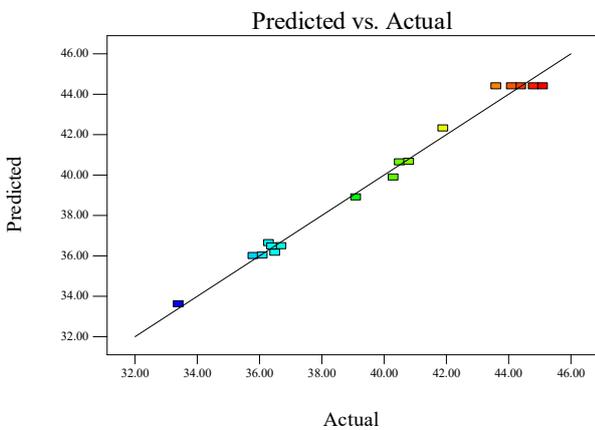


Fig. 4 - The relationship of predicted and actual values.

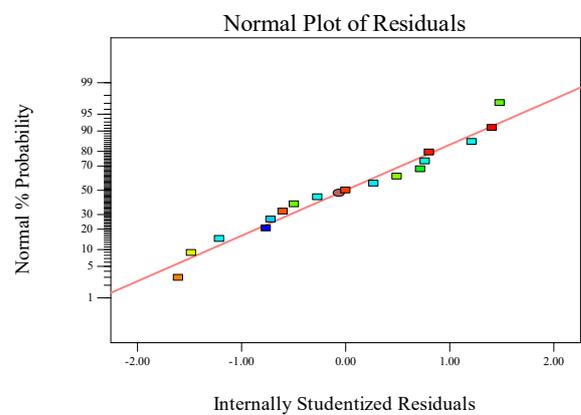


Fig. 5 - Normal probability plots of residuals.

response value.

All of above show that the presented regression equation is appropriate to be used for prediction of 28d compressive strength, and the model is efficiently functional for optimization of the preparation parameters of fly ash geopolymer concrete.

The ANOVA in Table 4 indicates that the response surface regression model reaches a highly significant level ($P < 0.0001$), and the lack of fit item is insignificant ($P = 0.5719 > 0.05$). The response surface model thus fits well with actual situation and can efficiently predict the 28-day compressive strength of concrete. Table 4 also indicates that the factors in the RSM have a significant influence on the 28-day compressive strength of concrete, and the order of the influence is X_2 (dosage of alkali) $> X_3$ (sodium silicate modulus) $> X_1$ (water-binder ratio). The interactive item X_1X_2 is significant, the interactive item X_2X_3 is generally significant, and the interactive item X_1X_3 is insignificant. The influence of the water-binder ratio and alkali dosage interaction on the 28-day compressive strength is obvious, that of the alkali dosage and sodium silicate modulus interaction is relatively obvious, and that of the water-binder ratio and sodium silicate modulus interaction is not

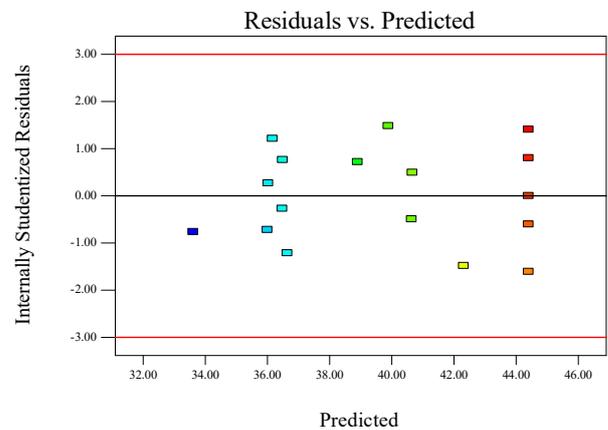


Fig. 6 - Plot of residuals against predicted response.

obvious. These results are reflected intuitively in Figures 7 to 9. The shape of the 3D response surface and contour can reflect the degree of interaction. The 3D response surface in Figures 7 and 9 is steep, and the contour is elliptical. These figures represent a relatively significant interaction between the two factors. The shape of the 3D response surface in Figure 8 is gentle, and the contour is circular, indicating the insignificant interaction between the two factors. This finding is consistent with the results of ANOVA in Table 4.

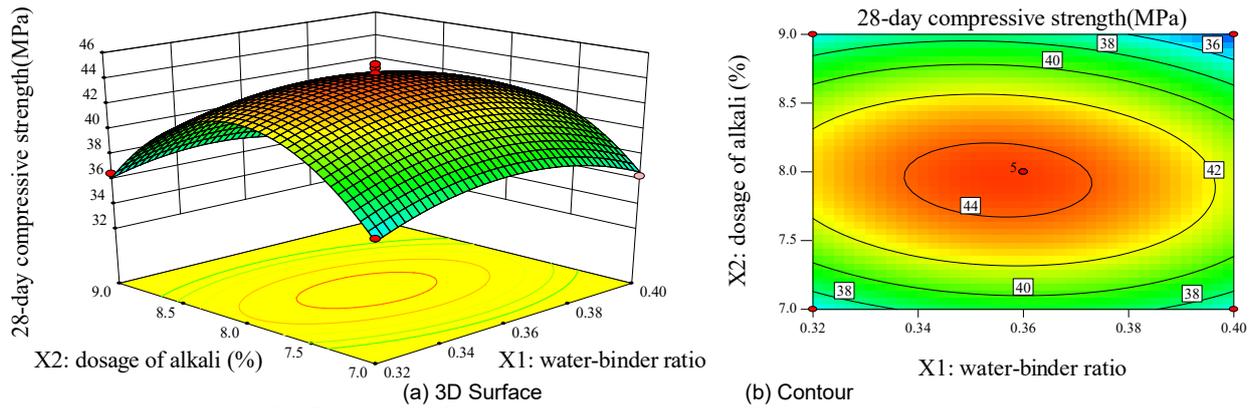


Fig. 7- Influence of X_1 , X_2 and their interaction on 28d compressive strength

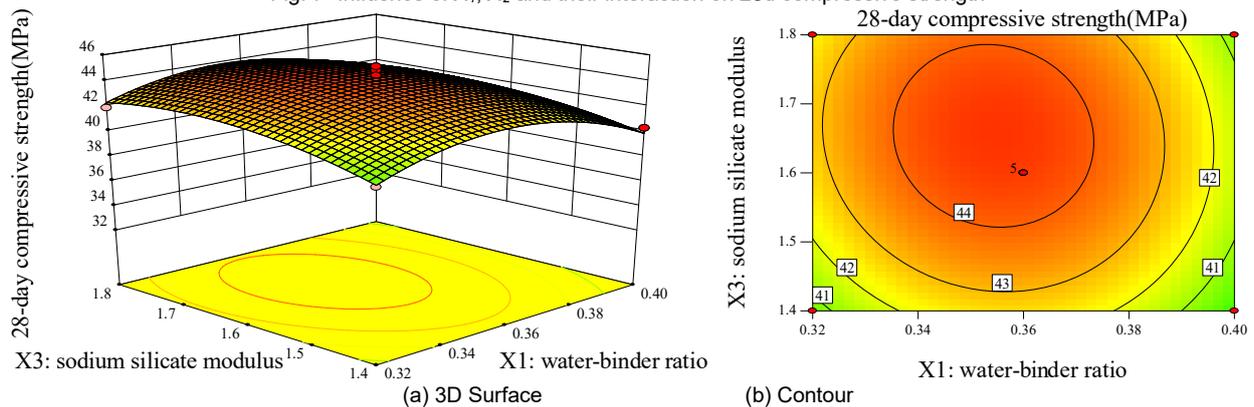


Fig. 8 - Influence of X_1 , X_3 and their interaction on 28d compressive strength

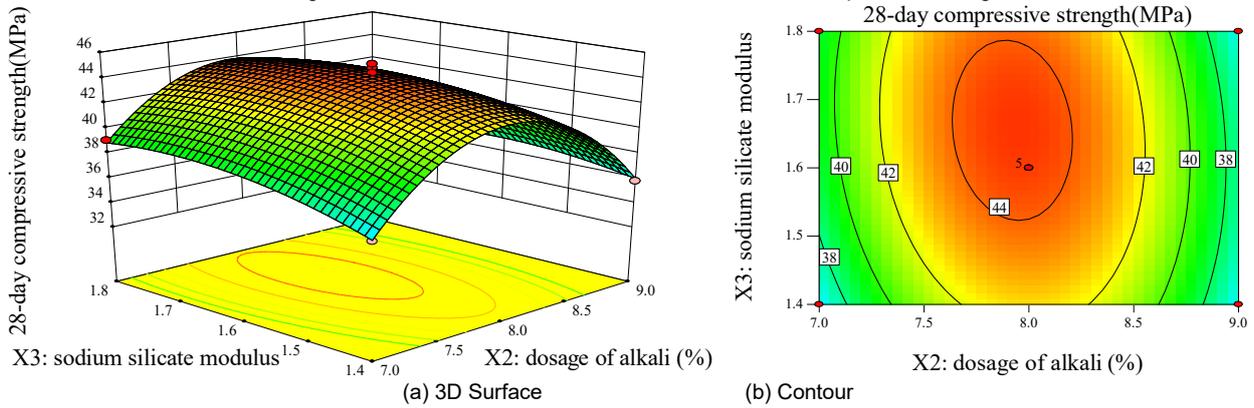


Fig. 9 - Influence of X_2 , X_3 and their interaction on 28d compressive strength

3.4. Determination of the optimal preparation parameters

The automatic optimization function of Design-Expert software V8.0.6.1 indicates that the optimal values of the factors for the highest concrete strength are as follows: a water–binder ratio of 0.35, an alkali dosage of 7.9%, and a sodium silicate modulus of 1.66. The optimum parameters are predicted to yield a maximum strength of 44.6 MPa. This prediction is verified by three validation experiments under the optimum parameters. The mean value of the obtained 28-day compressive strength is 45.6 MPa, and the relative error in comparison with the predictive value is 2.24%. Hence, the response surface model has high accuracy. It can predict the compressive strength of fly ash

geopolymer concrete and optimize the preparation parameters better.

4. Conclusions

The influence of the regularities of water–binder ratio, alkali dosage, unit water dosage, and sodium silicate modulus on the 28-day compressive strength of fly ash geopolymer concrete were studied.

The experiments were designed using the BBD method of RSM, and the preparation parameters of fly ash geopolymer concrete were analyzed comprehensively. The results showed that the order of the factors in terms of their influence on concrete strength is alkali dosage, sodium silicate modulus, and water–binder ratio, and the unit water dosage has minimal influence.

The 28-day compressive strength prediction model of concrete was established based on the RSM and the experimental results.

The preparation parameters were optimized to improve the 28-day compressive strength of fly ash geopolymer concrete through the response surface model. The optimum preparation parameters are: a water–binder ratio of 0.35, an alkali dosage of 7.9%, and a sodium silicate modulus of 1.66. The maximum strength is predicted to be 44.6 MPa based on the optimum preparation parameters. This prediction was verified by validation experiments. In addition, the fly ash geopolymer concrete with 45.6 MPa was prepared, which exhibited a relative error of 2.24%. Therefore, the optimization results of the response surface model is valid.

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