Research Article

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Optimization of the synergistic effect of micro silica and fly ash on the behavior of concrete using response surface method

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Abstract: The impacts of different replacement levels of micro silica (MS), fly ash (FA), and the combined usage of MS and FA on the behavior of concrete were investigated. Design of experts has been used incorporating the response surface methodology to find the synergetic effect of FA and MS on fresh and hardened concrete properties. A total of 15 and 30% of the binder content were replaced by FA. As well as a total of 5 and 10% of the binder content were replaced by MS. Experimental results of concrete mixtures have been used to construct ANOVA models. These models have also been confirmed to expect the properties of concrete mixtures based on different fractions of FA ash and MS. All statistical models are significant because P-value is less than 5%, and the difference between predicted R^2 and adjusted R^2 is lower than 20%. The variation between confirmation tests for the optimized mixture and test results are lower than 5%.

Keywords: optimization, design of experiments, response surface method, faced central composite design, desirability

1 Introduction

Micro silica (MS) has been widely used in concrete as it decreases the required cement content for a specific target

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strength, improves the durability of hardened concrete, and increases the strength of concrete while keeping the similar mixture design variables when added in optimal amounts [1–3].

Fly ash (FA) has also been commonly used in concrete as it decreases the cost of the concrete, saves natural resources and energy and decreases environmental problems. The impacts of FA on the different properties of concrete have been stated in ACI Committee 232 "Report on the use of FA in concrete in 2002." Some of the problems are related to utilizing this material in concrete since FA has a low surface area and associated pozzolanic activity. At standard temperatures, the pozzolanic activity is slow to initiate and it does not improve to any considerable degree till some weeks after the initiation of hydration. This resulted in slow strength improvement even if the concrete may have higher strength at later ages.

Overcoming the influences of FA on the early age properties of concrete mixtures is still a challenge. MS seems to be a possible solution to this problem because of its high activity nature. MS developed considerable amounts of calcium silicate hydrates at an early age which would improve the early age strength. This work program was initiated to study the synergistic effect of MS and FA on the slump and compressive strength of concrete.

Many studies have lately been performed using a synergistic of two by-products [4,5].

The effects of synergistic of FA and silica fume on some properties of self-compacting concrete were studied by Çelik et al. [6]. FA up to 45% and silica fume up to 15% were replaced for Portland cement. The experimental results observed that the increased ratios of FA and silica fume in self compacting concrete mixtures considerably decreased the rapid chloride permeability and water absorption values of concrete at a late age.

The effect of FA and MS fume on early-age strength of concrete was studied by Breesem et al. [7]. Compressive strength at 3 and 7 days was studied. The results showed

that partial replacement of Portland cement by 30% FA and 10% MS fume have a positive impact on the early compressive strength development.

However, little information is presently known regarding the optimum percent of FA and MS together in concrete mixtures.

This investigation studies ternary blends of FA, MS, and Portland cement using a wide range of mixtures: FA from 0 to 30%, and MS from 0 to 10% by the total mass of the binder. Fresh and hardened properties, in terms of the workability and compressive strength of the ternary blend concrete, were studied.

The main objective of the investigation work stated in this study is to use the response surface methodology (RSM) optimization technique in modeling the fresh and hardened properties of ternary blend concrete to determine the optimum percent of the variables (FA and MS) that result in a maximum 28 days compressive strength and 80 mm slump.

2 Methodology

2.1 Design of experiment (DoE)

DoE is a statistical method to investigate the effect of independent variables (factors) on the experimental results. DoE has many benefits, for example, the use of minimum number of experimental runs to analyze response surfaces, assessment of the quadratic impact of considered responses, documentation of probable interrelations between independent variables, and conclusion of the optimal response [8].

In statistics, RSM explores the relationships between independent factors and one or more response variables [9,10]. As the RSM gives perfect results and the interaction among response variables can be determined by it, it has been largely used in the field of concrete technology [11–13]. So faced central composite design (FCCD) was implemented to obtain the optimum proportion of independent factors (FA and MS) and the effect of them on the slump and compressive strength of concrete.

FCCD was used to design the number of experiments by using equation (1).

$$n = 2^k + 2k + c, \tag{1}$$

where k is the studied factors, " 2^k " is the factorial points, "2k" is the axial points, and "c" is the center point. The first-order model was fitted through the factorial points

and the axial points to fit the second-order polynomial [8]. For a nonlinear and complex system, the second-degree polynomial equation (2) is used to define the relationship between the studied factors and to expect the response.

$$Y = \alpha_0 + \sum \alpha_i X_i + \sum \alpha_{ii} X_i^2 + \sum \alpha_{ij} X_i X_j, \qquad (2)$$

where *Y* is the response, X_i is the studied factor, α_0 is a constant, and α_i (i = 1, 2, 3), α_{ij} (i = 1, 2, 3; j = 2, 3; j > i), and α_{ii} (i = 1, 2, 3) denote the linear coefficients, interaction coefficients, and quadratic regression coefficients of each studied factor, respectively.

In this study, Minitab-18 software was implemented to obtain the ANOVA output to develop prediction mathematical models using multiple regression analysis.

2.2 Factors and their levels

To construct the DoE, first, the number of levels for adopted factors must be determined. Based on the number of factors and their levels attained, the test program can be achieved. Two factors and three levels were used to evaluate the influence of FA and MS on the slump and compressive strength of concrete. The factors, factors coding, and levels are presented in Table 1.

2.3 Materials

- 1. Ordinary Portland cement Type I following ASTM C150-17 [14].
- 2. FA class F following ASTM C618-14 [15].
- Powdered solid MS was used in some binders. The chemical and physical properties of ordinary Portland cement, MS and FA are presented in Table 2.
- 4. A high-range water-reducing admixture following ASTM C494-17 [16], Type F, was used.
- 5. Natural sand was used as a fine aggregate.

Table 1: Coding and experimental values of adopted variables

Factor	Coded level			
	-1	0	+1	
FA (kg/m ³) MS (kg/m ³)	0 0	67.5 22.5	135 45	

Table 2: Chemical and physical properties of binder materials

Chemical composition	Cement	FA	MS
SiO ₂ (%)	21.2	40.19	91.8
Al ₂ O ₃ (%)	4.8	28.77	1.2
Fe ₂ O ₃ (%)	3.3	15.75	1.3
CaO (%)	62.9	9.15	1.6
MgO (%)	1.9	1.89	0.9
SO ₃ (%)	2.4	1.91	0.3
Loss on ignition (LOI)	2.0	_	2
Insoluble residue (IR)	1.1	_	_
Physical properties			
Specific gravity	3.15	2.1	2.2

6. Natural gravel was used as a coarse aggregate. The grading of fine and coarse aggregates followed ASTM C 33-16 [17].

2.4 Preparation and testing of specimens

Batching and mixing were conducted following ASTM C192-16 [18]. Nine mixtures were prepared to investigate the two key factors adopted in Table 3. Each mixture ID starts with a number that denotes the partial replacement of the succeeding letter. A total of 15 and 30% of the binder content (67.5 and 135 kg/m³, respectively) were replaced by FA. As well as a total of 5 and 10% of the binder content (22.5 and 45 kg/m³, respectively) were replaced by MS.

Slump tests were performed by following ASTM C143-15 [19]. The compressive strength test was determined according to BS EN 12390-3 [20]. For compressive strength, cube molds of $100 \text{ mm} \times 100 \text{ mm} \times 100 \text{ mm}$ dimensions were cast. After being de-molded, all the concrete specimens were cured underwater at 23 ± 2°C until the age of 7 and 28 days.

Table 3: Mix proportion for a cubic meter mixture

No. MS (kg) Mix ID **Coding notation** FA (kg) Cement (kg) Water (L) SP (L) Fine Coarse FA, MS agg. (kg) agg. (kg) -1, -11 Control 0 450 190 4.5 770 950 15FA5MS 22.5 360 4.5 770 2 0,0 67.5 190 950 190 30FA10MS 270 3 1, 1 135 45 4.5 770 950 4 15FA10MS 0,1 67.5 45 337.5 190 4.5 770 950 5 10MS 0 45 405 190 4.5 950 -1, 1770 6 15FA 0, -167.5 382.5 190 4.5 770 950 7 5MS -1, 00 22.5 427.5 190 4.5 770 950 8 30FA5MS 1, 0 135 22.5 292.5 190 4.5 770 950 30FA 1. -1135 315 190 4.5 770 950

3 Discussion of experimental results

3.1 Slump of concrete

The slump measurement for all mixtures is displayed in Table 4 and Figure 1. From Figure 1, it can be detected that the inclusion of MS significantly decreased the slump of the concrete, whereas the inclusion of FA considerably increased the slump of the concrete mixtures.

Generally, the increases in FA replacement percent by 15 and 30% considerably increased the slump values. For example, the incorporation of 30% FA in the control mix resulted in an 80% increase in slump value, while adding 15% FA to control mix increased the slump by only 30%. This behavior could be ascribed to the morphological impact of FA grains and their features [21].

Compared with the control concrete mixture, the MS concrete mixtures displayed greater decreases in slump values. For instance, adding 10% MS to the control mixture reduced the slump by 50%. While adding 5% MS to the control mixture reduced the slump by only 30%. These values are in agreement with those detected in previous studies [22-24]. They concluded that the inclusion of MS reduces the quantity of lubricating water available, which causes an increase in the yield stress and plastic viscosity of concrete.

These effects of FA and MS on slump are similar to the results stated by Brooks et al. [25] and Nochaiya et al. [3].

The synergistic effect of FA and MS has a positive effect on the slump measurement. For instance, compared with MS mixtures, the mixtures with a combined usage of FA and MS exhibited lower decreases in slump values. For instance, incorporating 30% FA to a mixture of 10MS to produce 30FA10MS increased the slump by

Table 4: Experimental tests results

No.	Mix ID	Coding notation FA, MS	Slump (mm)	CS at 7 days	CS at 28 days
1	Control	-1, -1	100	27.3	30.2
2	15FA5MS	0, 0	90	27.8	34.8
3	30FA10MS	1, 1	110	34.9	42.4
4	15FA10MS	0, 1	80	31.5	38.2
5	10MS	-1, 1	50	31.4	36.9
6	15FA	0, -1	130	25.6	29.7
7	5MS	-1, 0	70	28.4	32.6
8	30FA5MS	1, 0	150	28.1	34.7
9	30FA	1, -1	180	23.7	27.7

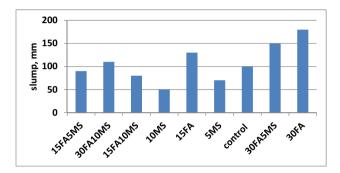


Figure 1: Slump results for all mixtures.

120%. While adding 15% FA to a mix 10MS to produce 15FA10MS increased the slump by only 60%. Similarly, adding 30% FA to a mix of 5MS to produce 30FA5MS increased the slump by 114%. While adding 15% FA to a mix of 5MS to produce 15FA5MS increased the slump by only 30%.

Briefly, among the different independent variables studied in this investigation, the incorporation of 30% FA and the use of a low level of MS appear to have pronounced impacts in increasing the slump value.

3.2 Compressive strength

The 7 and 28 days compressive strengths and relative strengths are presented in Figures 2 and 3, respectively. The compressive strength of these mixtures at the two ages studied was determined to decrease with the increase in FA fraction. In general, FA is well-known to reduce the strength of concrete at an early age [26,27]. Helmuth [28] also stated that the bonding of FA particles to the matrix at an early age is very weak. At the age of 7 and 28 days, the FA concrete mixtures without MS content were found to

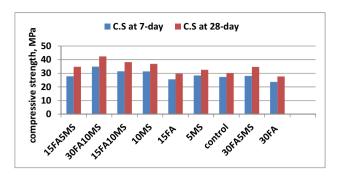


Figure 2: Compressive strength of all concrete mixtures.

have lower strengths than the control mixture. Whereas, the FA mixtures with MS were found to achieve greater strengths as shown in Figure 3. The 30FA10MS mixture was determined to increase significantly in compressive strengths 34.9 and 42.4 MPa at 7 and 28 days of age, respectively.

The relative strength results at 28 days were also observed to have high strength results of 140.4 and 126.5%, respectively, for mixtures 30FA10MS and 15FA10MS concerning PC, as shown in Figure 3.

The effect of MS on the compressive strength of blended concrete can be found by comparing the relative strengths of the mixtures with and without MS. The results are presented in Figure 4. Results for concrete mixtures with MS were higher at the two ages considered than for those mixtures without MS (for the same FA fraction).

The high compressive strength of ternary binder mixtures was owing to both the filler effect and the pozzolanic reaction of MS giving the matrix a denser microstructure, thus resulting in a substantial improvement in strength.

The maximum relative strength values achieved by the mixture with cement replacement of 30% FA and 10% MS were 147.3 and 153% after 7 and 28 days, respectively.

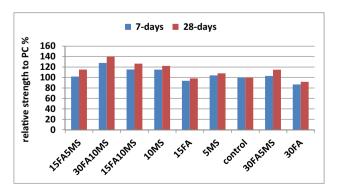


Figure 3: Relative strength of control concrete mixture.

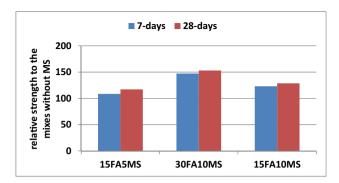


Figure 4: Relative strength of the mixes without MS of concrete.

These results show that the inclusion of MS together with FA can significantly enhance compressive strength.

4 RSM modeling

4.1 Discussion on derivative statistical model

To determine the effect of independent factors comprising FA and MS on the slump and compressive strength of concrete mixtures and to calculate the slump and compressive strength for different factors, the FCCD method was adopted in this investigation. To attain this, nine experiments were prepared for each response. The achieved FCCD responses were stated in the form of second-degree polynomial equations. The fit regression models for slump and 7 and 28 days compressive strength are given in equations (3)–(5).

Slump =
$$96.67 + 36.67 \text{ FA} - 28.33 \text{ MS}$$
, (3)

C.S at
$$7 - \text{day} = 27.656 + 3.533 \text{ MS} + 1.775 \text{ FA*MS},$$
 (4)

C.S at
$$28 - \text{day} = 34.133 + 4.983 \text{ MS} + 2.000 \text{ FA*MS}$$
. (5)

The ANOVA results of the studied responses are presented in Table 5. The statistical significance of every term was tested, and any factor with a P-value > 0.05 was eliminated from the derived model. A P-value > 0.05 shows an inconsequential role and indicates that the factor has no impact on the response.

As can be noticed from Table 5, the *P*-value for all models were <0.05, indicating that the response models were very valid.

As presented in Table 5, the linear term of FA and MS were significant for slump and the P-value was lower than 0.05. But the quadratic and the interactive term were inconsequential and the P-values were > 0.05. As the linear term of FA and MS were significant, the slump of the concrete mix was considerably affected by FA and MS.

The role of each coefficient in the model's equations can be recognized by comparing their values, which reveal their influence on the response. However, the above expressions are limited by the upper and lower boundaries of the investigated factors (i.e., +1 and -1 levels) presented in Table 1. From Table 5, it can be observed that the linear effect of FA and MS inversely affect the slump measurement of concrete mixtures.

The derived slump model presented by equation (3) showed that FA and MS content had significant effects on the slump model response. FA had a comparable inverse influence on the slump relative to MS, 36.67 vs –28.33.

Since the *P*-value of the linear term of MS was <0.05, the 7 days and 28 days compressive strength of the concrete mixtures were considerably affected by the MS.

Table 5: ANOVA results

Source	Slump (mm)		Significance	CS at 7 days		Significance	CS at 28 days		Significance
	Coeff.	<i>P</i> -Value		Coeff.	<i>P</i> -Value		Coeff.	<i>P</i> -Value	
Model		0.005	Yes		0.004	Yes		0.007	Yes
Constant	96.67	0.000	Yes	27.656	0.000	Yes	34.133	0.000	Yes
Linear		0.001	Yes		0.002	Yes		0.002	Yes
FA	36.67	0.001	Yes	-0.067	0.804	No	0.850	0.117	No
MS	-28.33	0.003	Yes	3.533	0.001	Yes	4.983	0.001	Yes
Square		0.253	No		0.151	No		0.953	No
FA ²	10.00	0.154	No	0.667	0.216	No	-0.150	0.838	No
MS ²	5.00	0.413	No	0.967	0.108	No	0.150	0.838	No
Two-way interaction		0.272	No		0.010	Yes		0.025	Yes
FAMS	-5.00	0.272	No	1.775	0.010	Yes	2.000	0.025	Yes

Bold factors refer to significant values.

Subsequently, the P-value of the linear term and quadratic term of FA were >0.05, the 7 and 28 days compressive strength of the concrete mixtures were insignificantly affected by FA.

The derived CS at 7 and 28 days model presented by equations (4) and (5) showed that MS content had significant effects on the compressive strength, while the effect of FA on the CS at 7 and 28 days was negligible.

4.2 Model checking

The fitting of the statistical models can be confirmed through the coefficient of determination (R^2) and the difference between the adjusted R^2 and the predicted R^2 should not be > 0.2 [8]. Table 6 displays the R^2 , adjusted R^2 , predicted R^2 , and the difference between adjusted R^2 and predicted R^2 . As it can be observed from Table 6, the

Table 6: Results for the statistical models

Responses	R ² (%)	Adj- <i>R</i> ² (%)	Pred- <i>R</i> ² (%)	Difference between adjusted R^2 and predicted R^2 (%)
Slump	98.76	96.68	88.38	8.30
CS-7 days	98.81	96.82	85.72	11.10
CS-28 days	98.41	95.77	83.40	12.37

 R^2 value of the responses slump, CS at 7 days, and CS at 28 days were 98.76, 98.81, and 98.41%, respectively, which indicate that 1.24, 1.19, and 1.59% of variation only can be discovered by the adopted model.

In addition, the difference between the predicted R^2 and the adjusted R^2 of all responses (slump, CS at 7 days, and CS at 28 days) were lower than 0.2, indicating that the statistical model was very realistic.

The normal probability plots of the residual fit for every response are observed in Figure 5. It can be observed

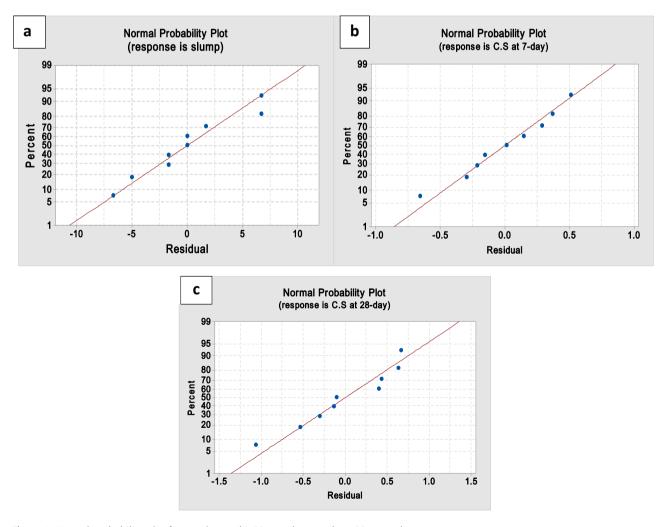


Figure 5: Normal probability plot for (a) slump, (b) CS at 7 days, and (c) CS at 28 days.

from Figure 5, that the residual of all the responses was displayed as a straight line and falling very adjacent to the straight line, representing that the errors were equally distributed. Moreover, Figure 5 confirms the suitability of the least-squares fit.

From Figure 6, it can be observed that the predicted values agreed with the results of the experimental tests revealing that the adopted model can be used to predict (slump, CS at 7 days, and CS at 28 days) within the limits of the variables studied.

4.3 Surface plot analysis

To distinguish the effect trend of the adopted factors on the studied responses, 3D surface plots were plotted and displayed in Figure 7. From Figure 7(a) it can be understood that the inclusion of FA improved significantly the slump of the concrete mixture. Whereas, the inclusion of MS considerably decreased the slump of concrete. However, the influence of FA was very significant than the MS.

From Figure 7(b) and (c), it can be observed that the inclusion of FA reduced the compressive strength of the concrete at the two ages studied. Whereas the inclusion of MS significantly increased the compressive strength of concrete. However, the effect of MS was very substantial than the FA.

4.4 Optimization of process variables

The optimized slump and 28 days compressive strength of the concrete mixtures are observed in Figure 8. The

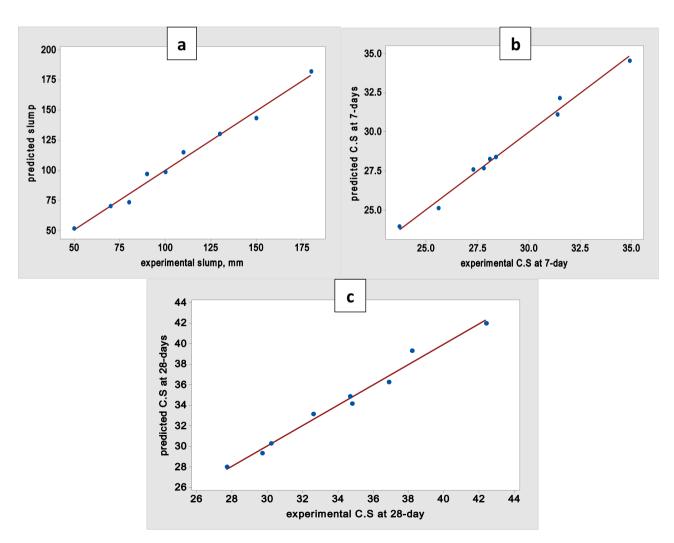


Figure 6: Comparison between experimental and predicted responses. (a) Slump, (b) CS at 7 days, (c) CS at 28 days.

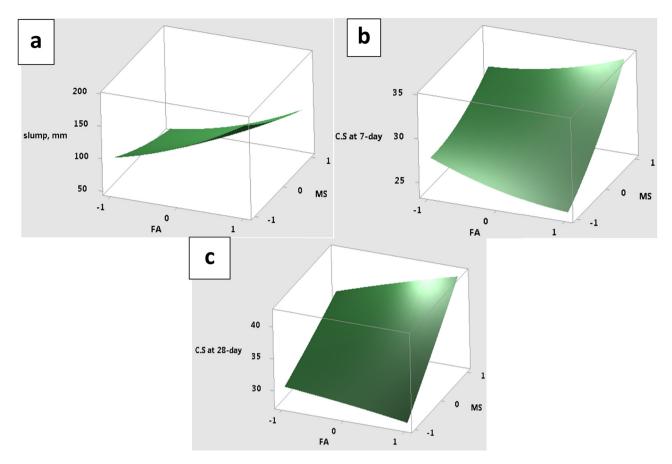


Figure 7: Surface Plot of (a) slump, mm, vs MS and FA (b) CS, MPa, at 7 days vs MS and FA (c) CS, MPa, at 28 days vs MS and FA.

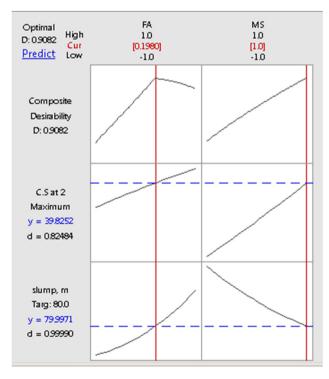


Figure 8: Optimization plots for 80 mm slump and maximum CS at 28 days.

notation "d" plotted in Figure 8 shows the desirability of the studied factors ranging from 0 to 1, where "1" denotes the ideal mode and "0" shows the undesirable combination [8]. As the slump value of 80 mm was considered as a desired slump for structural concrete, this value was selected as a target value to optimize the slump.

To confirm the results of RSM, validation tests were done and these results with the percent of error are presented in Table 7. From Figure 8, it can be observed that the optimal values of FA and MS were 5.94 and 10%, respectively, as a partial replacement of Portland cement to attain the maximum 28 days compressive strength and 80 mm slump. The experimental validation was conducted for the optimized mixture, and the results were attained with variation lower than 5% as presented in Table 7.

5 Conclusion

RSM optimization technique was adopted for modeling the fresh and hardened properties of ternary blend

Table 7: Validation test results and the percentage of error

Test description	FA%		MS%		Predicted results using RSM	Confirmation test results	Percentage of error (%)	
	Coded	Real	Coded	Real				
Slump	0.198	5.94	1	10	79.9971	81.4	1.75	
CS at 28 days	0.198	5.94	1	10	39.8252	40.5	1.69	

concrete to achieve the optimum percent of FA and MS together in concrete mixtures. In this model, three responses (slump and compressive strength at 7 and 28 days) were considered. Based on the ANOVA analysis, it was discovered that the statistical models for slump and compressive strength at 7 and 28 days are highly significant. As lack-of-fit test results and high values of coefficients of determinations (R^2) confirmed the accurateness of the second-degree polynomial model to expect the required properties of concrete concerning 7 and 28 days compressive strengths, in addition to the slump value.

Optimization of two responses was also performed to achieve a mixture with 80 mm slump and maximum 28 days compressive strength. The achieved results revealed that the optimum values of studied factors were 5.94 and 10% of FA and MS, respectively.

Author contributions: Iman Kattoof Harith: Experimental program, Investigation, analysis, Validation, Writing – original draft, Editing, Visualization. Mahdi J. Hussein, Mahmood S. Hashim: review and editing.

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