RESPONSE SURFACE MODELING OF COMPRESSIVE STRENGTH OF HIGH-PERFORMANCE CONCRETE FORMULATED BY A HIGH WATER REDUCING AND SETTING ACCELERATING SUPERPLASTICIZER. BOX–BEHNKEN EXPERIMENTAL DESIGN

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ABSTRACT

This work aims to determine, on the ground of the response surface methodology of the experimental design, a model predicting the compressive strength of high-performance concrete formulated by a high water reducing and setting accelerating (SP103) superplasticizer as a function of the proportion of the constituents used. A second-degree polynomial equation is used to model the effect of the water content, the proportion of SP103 superplasticizer, and the W/C ratio keeping all other components and operating conditions unchanged.

The study has four essential parts. The first one focuses at the choice of the materials used, the method of formulation and the realization of the different mixtures. The second one presents the analysis of the effects and expected interactions between the various factors. The third part aims at the determination of the main physical, chemical, and mechanical characteristics of the materials used, while the last part describes the application of the response surface methodology defining each factor studied and validating the model advanced.

Keywords: response surface modeling, compressive strength, high-performance concrete, superplasticizer, water reducing, accelerating of setting, Box–Behnken, experimental design.

INTRODUCTION

The concrete is a composite material consisting of a mixture of a hydraulic binder (cement), aggregates (gravel and sand), water and possibly admixtures [1 - 7]. The high-performance concrete is characterized by its very high compressive strength reaching a value of 50 MPa within 28 days [8, 9]. It is difficult to understand the role of each constituent and hence to clarify its behavior because of its heterogeneity [2, 10]. The incorporation of superplasticizers in the high-performance concrete has to be done as late as possible in the course of mixing in order to minimize the amount of the mixing water and to improve the concrete properties in its fresh and hardened state [8 - 11]. The improvement of these properties and the compressive strength in particular is of a primary importance. The application of statistical techniques such as that of the experimental design makes this improvement more available [12 - 14]. They provide a minimum number of experiments [15 - 19], they factors screening as well as operating conditions optimization. This work continues our previous study on the development of new ecological and sustainable concrete based on different percentages of mineral additives and chemical admixtures [4, 8, 10]. The final product (the compressive strength) is obtained and subsequently characterized upon formulating well chosen matrices. This approach can be infinite and more expensive.

The experimental design methodology provides to structure the research and validate the hypotheses with a focus at better understanding of the phenomena studied [12, 17, 20]. Its application is interesting in the sense that it reduces the number of the tests, while varying several parameters (factors) at a time enabling the evaluation of their effects as well as their interactions. The experiments design according to the response surfaces methodology provides to solve optimization problems [21 - 23] and find the operating conditions that allow the maximization of the high-performance concrete compressive strength.

The key points of this study refer to strategic component type proportions assuming that the compressive strength depends only on the variation of three factors, i.e. the superplasticizer content, the amount of the mixing water and the W/C ratio. The studied response is followed using prismatic specimens (4 cm x 4 cm x 16 cm) preserved in water for 28 days. They are broken in the course of compression because of the effect of different forces. This model of rupture is evaluated in MPa. The iso-response curves and the response surface trace provide to determine the operational conditions for an optimal compressive strength reached within 28 days on the ground of a polynomial mathematical model of a second degree. At the end of this process, a validation test is realized to justify the quality of the chosen model and its application, while comparing the theoretical values of the compressive strength (calculated from the model) and the experimental one (obtained upon application of the model optimal conditions).

EXPERIMENTAL

Materials

Cement

CEM I 42.5 N cement from the plant of Amran in Yemen was used in this work.

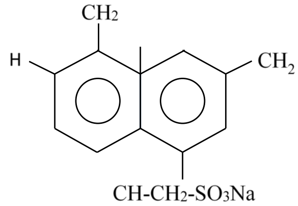

Fig. 1. A chemical structure of sulfone polynaphthalene.

High range water reducing and setting accelerating superplasticizer

The high range water reducing and setting accelerating superplasticizer (SP103) is a liquid polymer synthesized for the concrete industry. It is based on sodium or calcium salts of sulfonated polynaphthalene (Fig. 1) or sodium salt of sulfonated polymelamine [EN 934-2] (Fig. 2), [8, 9].

SP103 used in the formulation of mortars and/or concrete of a high performance was delivered by CONMIX Ltd. in Sharjah, the United Arab Emirates.

Water

Tap water was used to obtain the mortar and/or concrete.

Sand

Standard sand according to EN 196-1 norm was used to make the mortar. It was delivered by the new French company of Littoral.

Software used

The software (NEMRODW): A New Efficient Methodology for Research using an Optimal Design was used to study the effect of each factor and their interactions on the compressive strength of the formulation matrix containing various percentages of SP103. This software provided the optimization of the desired process (industrial/research and development) while improving the quality of the information required ("y" response).

Methods

Experimental protocol of preparation of mortar and/or concrete in its hardened state

A mortar with and no SP103 superplasticizer was prepared. The compositions referred to the normal mortar defined by EN 196-1 standard. The water quantity was adjusted, while the paste obtained was with a standard consistency. The procedure employed included

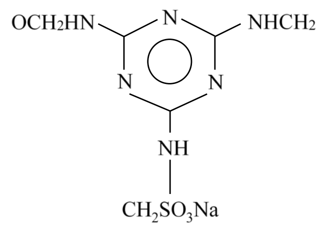

Fig. 2. A chemical structure of sulfone polymelamine.

mixing and filling a mold (4 cm x 4 cm x 16 cm). The mortar in the mold was tightened twice. Each process of tightening was followed by the application of 60 shocks using a shock device. After that the mold was leveled, covered with a glass plate and stored in a wet room. After 20 h or 24 h from the start of the mixing, the specimens were removed from the mold and stored in water at 20 ± 1°C until the time of the rupture test. The compressive strength was measured at a young age (2 days), a median age (7 days) and after 28 days using a bending test machine (Fig. 3) to break the specimen into two halves. Each of the latter was subjected to compression using a hydraulic compressive testing machine (Fig. 4). The value of the resistance referred to the average value of the crushing stress of the three test pieces (6 halves).

The compressive strength was calculated using Eq.(1):

$$\text{Compressive strength} = \frac{\text{Load , in "N"}}{\text{Area, in "mm}^2\text{"}}; \text{MPa} \qquad (1)$$

Experimental design method

The experiments designs were a part of a general process for improving the quality of the process studied.

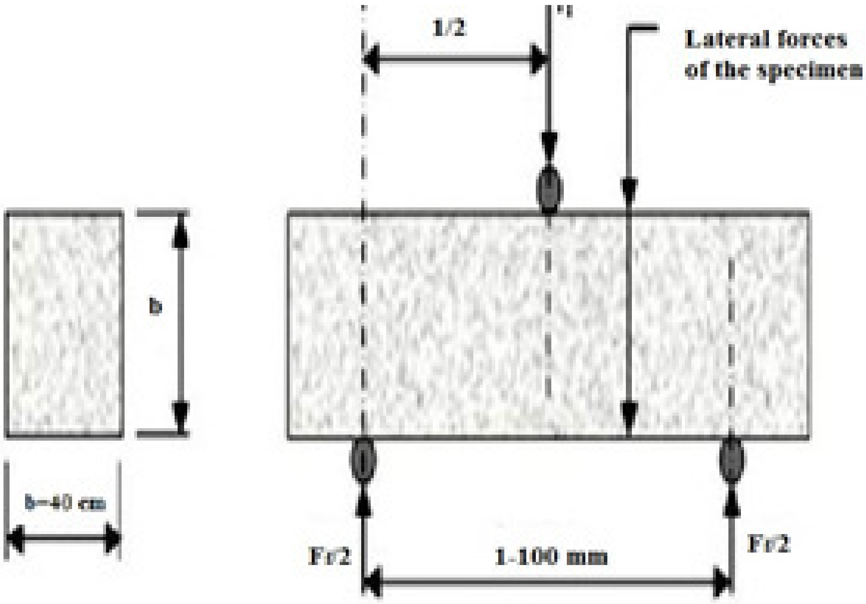

Fig. 3. Bending load device for mortar specimens.

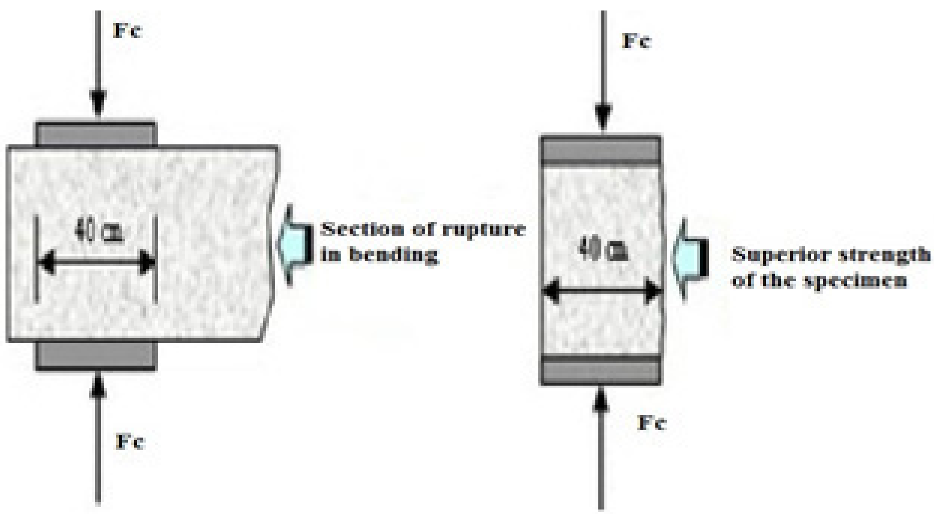

Fig. 4. Compressive load device for mortar specimens.

They provided new knowledge by controlling one or more input parameters aiming better results and validating the selected model, while minimizing the number of the tests carried out. Thus they saved time, materials and labor optimizing the studied response.

Methodology by the response surface of experiments design

The methodology of the response surfaces aimed to explore the relations between a response (y) and several factors X_1, ..., X_n by fitting a polynomial mathematical model of a second degree. It provided a spatial presentation of the model (response surface/iso-response), which in turn described the principal and secondary effect of each factor and their interactions. Thus the appropriate operating conditions for the optimization of the studied response (y) were found.

Definition of the factors and their experimental domain

A composite experiment matrix was chosen to improve the compressive strength of mortar and/or concrete based on SP103 superplasticizer. It led to obtaining an optimal quality predicting the calculated response (the compressive strength at the 28th day). It was based on three factors and led into the field of study (Table 1).

The different factors, the corresponding coded variables, and the experimental matrix are presented in Tables 2 and 3.

The empirical model definition

A second-degree polynomial model (Eq. 2) was used to improve the mechanical properties of the concrete modified by SP103 superplasticizer. It provided the determination of the main effect, the effect of the inter-

Table 1. Experimental field of factors studied and their levels.

Factors	Notation	Quantity		Unity
		Low level **(-1)**	High level **(+1)**	
Content of SP103 (X_1)	X_1	0,00	4,00	%
Quantity of water (ml) (X_2)	X_2	142,00	225,00	ml
W/C report	X_3	0,32	0,50	%

Table 2. Coded variables of the experimental design.

N° d'exp	X_1	X_2	X_3	Y
1	-1	-1	-1	51.69
2	1	-1	-1	70.70
3	-1	1	-1	52.14
4	1	1	-1	64.10
5	-1	-1	1	47.00
6	1	-1	1	70.10
7	-1	1	1	52.14
8	1	1	1	63.80
9	-1	0	0	56.30
10	1	0	0	70.10
11	0	-1	0	66.50
12	0	1	0	70.70
13	0	0	-1	68.90
14	0	0	1	68.30
15	0	0	0	67.10

Table 3. Matrix of the experimental design.

N° d'exp	SP103 (%)	Water (ml)	W/C report	Rmc - 28 Days (MPa)
1	0	142.0	0.3	51.69
2	4	142.0	0.3	70.70
3	0	225.0	0.3	52.14
4	4	225.0	0.3	64.10
5	0	142.0	0.5	47.00
6	4	142.0	0.5	70.10
7	0	225.0	0.5	52.14
8	4	225.0	0.5	63.80
9	0	183.5	0.4	56.30
10	4	183.5	0.4	70.10
11	2	142.0	0.4	66.50
12	2	225.0	0.4	70.70
13	2	183.5	0.3	68.90
14	2	183.5	0.5	68.30
15	2	183.5	0.4	67.10

action between the different factors and the quadratic effect using Nemrod software. The surface model of the second-degree response can be presented in the form:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=i+1}^{k} \beta_{ij} x_i x_j + \varepsilon \quad (2)$$

The variables x in Eq. (2) were replaced with the factors investigated and the second-degree polynomial equation (Eq. 3) was obtained:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + + \beta_{12}(X_1 X_2) + \beta_{13}(X_1 X_3) + \beta_{23}(X_2 X_3) + \varepsilon \quad (3)$$

where Y was the studied response (the compressive strength at the 28-th day), β_0 was the value of the response at the center of the study area, $\beta_{1,2,3}$ were the regression coefficients of the linear effects, $\beta_{11,22,33}$ were the regression coefficients of the quadratic effects, $\beta_{12,13,23}$ were the regression coefficients of the interactions effects; $X_{1,2,3}$ and $X_{1,2,3,}^2$ were the experimental variables coded, while ε stood for the residues.

Statistics study

Mathematical analysis of the test results

The mathematical and the statistical analysis consisted essentially of identifying the p model coefficients from the results of N experiments. The application of the least squares method to the test results yielded 10 model coefficients.

Coefficients estimation and statistics

Table 4 lists all factor coefficients studied as well as their effects, the statistical values (t-student), and the observed (significant) probability.

The presented results in Table 4 showed that t-student values were used to determine the significance of the coefficients of each parameter, while the significant values were defined as the least important level. In general, the higher the t-student's magnitude, the smaller the significance and the greater the corresponding coefficient term were. The significant factor of the majority of the coefficients was found to have a value close to 5 %, which indicated that the matrix was of a good quality. In other words, β_1, β_{11} and β_{12} were statistically significant coefficients.

Results analysis

Variance analysis

The variance analysis provided the identification of the model elements of the greatest and the lowest importance. Table 5 summarizes the variance analysis results of the retained model and their significance.

Table 5 shows that the response has a significant value (0.390). In other ways, the main effect of the regression was significant since the probability of the risk significance (significant = 0.39) was less than 5 %. Thus, the model had a confidence level of 95 %.

Table 4. Statistical estimation of the model coefficients and their importance with respect to the experimental dispersion.

Coefficient	Estimated value	F.Inflation	Standard deviation	Value of t exp	Significance
β_0	69.68		1.30	53.56	< 0.01 ***
β_1	7.95	1.00	0.77	10.39	0.04 ***
β_2	-0.31	1.00	0.77	-0.41	70.00 NS
β_3	-0.62	1.00	0.77	-0.81	45.90 NS
β_{11}	-7.12	1.30	1.51	-4.72	0.59 **
β_{22}	-1.72	1.30	1.51	-1.14	30.70 NS
β_{33}	-1.72	1.30	1.51	-1.14	30.70 NS
β_{12}	-2.31	1.00	0.86	-2.70	4.24 *
β_{13}	0.47	1.00	0.86	0.55	60.70 NS
β_{23}	0.62	1.00	0.86	0.73	50.30 NS

*** *Very highly significant coefficient; ** Very significant coefficient];* Significant coefficient; NS No significant coefficient.*

Table 5. Variance analysis of the obtained model.

Source of variation	Sum of squares	Degrees of freedom	Average square	Report	Significance
Regression	946.64	9	105.18	17.96	0.39 **
Residues	29.29	5	5.86		
Total	975.93	14			

Results statistical analysis

The correlation coefficient, the multiple linear, R^2, and the adjusted coefficient multiple linear R^{2a} were calculated to estimate the quality of the obtained model. These coefficients are presented in Table 6. Table 6 shows that the obtained model coefficients are high. In other words, the obtained model has a satisfactory descriptive quality because of the correlation coefficient R^2 of 0.97 and the adjusted coefficient of determination R^{2a} of 0.92, i.e. these values are close to 1.

Fig. 5 presents the experimental (observed) values as a function of the predicted (calculated) one from the model of the studied response (the compressive strength). It is evident from Fig. 5 that the experimental values of the compressive strength relate to those calculated by the model. In other words, the correlation coefficient R^2 has a satisfactory descriptive quality.

RESULTS AND DISCUSSION

Characterization of the materials used

Cement

The chemical and mineralogical compositions of clinker, gypsum, and cement determined by XRF are presented in the Tables 7 and 8.

The cement physical and mechanical characteristics are presented in Table 9.

Table 6. Estimates and statistics of the postulated model coefficients.

Standard deviation of response	2.42
R^2	0.97
R^{2a}	0.92
R^2 pred	0.75
*PRESS	245.84
Number of degrees of freedom	5
R^{2a}: Adjustment coefficient R^2 pred: Predictive squares coefficient PRESS: Predicted Residual Sum of Squares	

Sand

The particle size analysis of the sand used is illustrated in Fig. 6. The particle size analysis presented in Fig. 6 shows that the grains of the sand used are distributed in a systematic way according to the specifications of EN 196-1 standard.

Mixing water

The main characteristics of the water used are summarized in Table 10.

SP103 superplasticizer

The physical properties of SP103 superplasticizer are listed in Table 11.

Graphical analysis of the model

It is necessary to find the optimal setting conditions

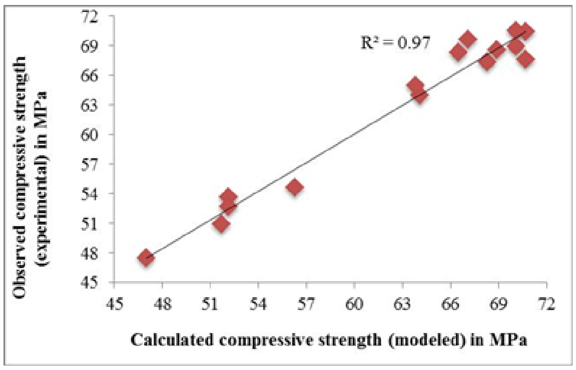

Fig. 5. Observed values as a function of the predicted one in case of the compressive strength rupture model.

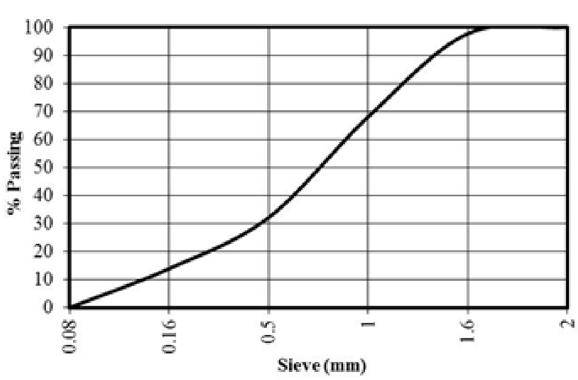

Fig. 6. Sand granulometric curve.

Table 7. Elementary chemical compositions of clinker, gypsum, and cement in atomic weight.

Chemical name	Chemical formula	Cement nomenclature	Clinker	Gypsum	Cement
Lime	CaO	C	62.76	33.40	61.29
Silica	SiO_2	S	21.00	0.70	19.99
Alumina	Al_2O_3	A	5.84	0.36	5.57
Ferrite	Fe_2O_3	F	3.00	0.09	2.85
Magnesia	MgO	M	1.96	0.63	1.89
Sulfur trioxide	SO_3	Ś	0.90	47.20	3.22
Potassium oxide	K_2O	K	1.21	0.03	1.15
Sodium oxide	Na_2O	N	0.20	0.10	0.20
Chloride Ion	Cl^-	Cl	0.02	0.01	0.02

that provide maximization of the compressive strength while reducing the amount of water used through the iso-response curves and the response surfaces.

Response surfaces and the iso-response

Fig. 7 shows the variation of the response in the plane: SP103 superplasticizers (X_1)/amount of water (X_2) while fixing the third factor W/C ratio (X_3) to 0.41.

(a) An iso-response curve

(b) A response surface curve.

Fig. 7 shows that the response "compressive strength at the 28-th day" increases with increase of the percentage of the superplasticizers (Factor X_1) and decrease of the amount of water (factor X_2). In other words, the

Table 8. Mineralogical composition of clinker.

Chemical name	Mineral name	Chemical formula	Cement nomenclature	Content
Tricalcium silicate	Alite	Ca_3SiO_5	C_3S	47.70
Dicalcium silicate	Balite	Ca_2SiO_4	C_2S	25.10
Aluminate tricalcium	Aluminate	$Ca_3Al_2O_6$	C_3A	10.40
Tetracalcium Aluminoferrite	Ferrite	$Ca_4Al_2Fe_2O_{10}$	C_4AF	9.10

Table 9. Physical properties of cement used.

Designations	Values	Units
Absolute Density	3.14	$g.cm^{-3}$
Refusal of the sieve 45 µm	12.50	%
Refusal of the sieve 90 µm	1.50	%
Specific surface Blaine	3240.00	$cm^2 \cdot g^{-1}$

Table 10. Main features of the mixing water.

Components	Units	Values
pH	-	7.00
Turbidity	(mg/l)	450.00
CO_3^{-2}	(mg/l)	216.00
HCO_3^-	(mg/l)	0.00
Ca^{+2}	(mg/l)	56.40
Mg^{+2}	(mg/l)	52.40
Conductivity	µS/cm	692.00

Table 11. Physical properties of superplasticizer (SP103).

Name	Nature	Color	Density ($g.cm^{-3}$)	Area training (%)	Chloride content
SP103	Liquide	Brown	1,20	0,50 – 1,00	Nil

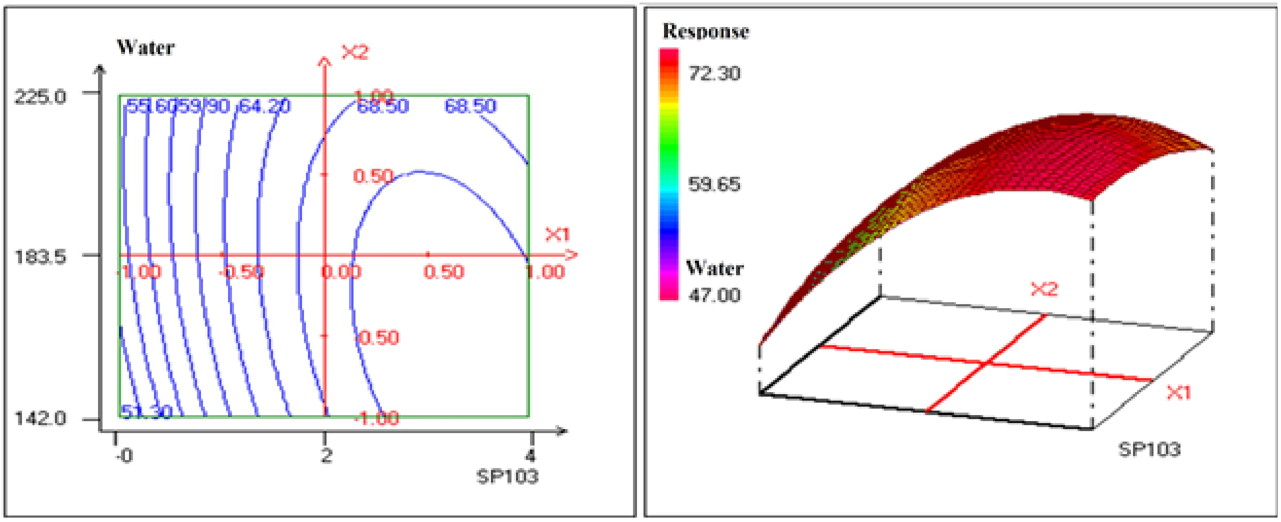

Fig. 7. Variation of the response plane: SP103 superplasticizers (X_1)/amount of water (X_2) while fixing the third factor W/C ratio (X_3) to 0.41.

response increases when factor X1 (SP103) approaches the mean level, while factor X_2 (quantity of water) approaches the upper-level in case the third factor X_2 (W/C ratio) is fixed at a value of 0.41.

It is necessary to determine the optimal conditions providing the improvement of the compressive strength at the 28-th day. These conditions can be extracted from Fig. 7. Table 12 summarizes the optimal field of the formulation.

Search for optimum

The mathematical model adapted to the response is presented by Eq. (4):

$$\hat{Y} = 69.70 + 7.95\,X_1 - 0.31\,X_2 - 0.62\,X_3 - 7.12 * X_1^2 - 1.72 * X_2^2 - 1.72 * X_3^2 - 2.31\,(X_1 * X_2) + 0.47\,(X_1 * X_3) + 0.62\,(X_2 * X_3) \quad (4)$$

Validation of the model obtained

Retained mathematical model

The estimated value ($\hat{Y}$) and the corresponding residuals can be estimated on the ground of Eq. (4). $E = Yi - \hat{Y}$.

Table 4 shows that the only main effect is that of SP103 (X_1), while the quadratic effects referring to the interactions SP103 (X_1-X_1) and those between SP103 and the water amounts (X_1-X_2) are meaningless. Then the best setting ($\hat{Y}$) is then easily presented in the form:

$$\hat{Y} = 69.70 + 7.95X_1 - 7.12\,X_1^2 - 2.31\,X_1\,X_2 \quad (5)$$

Model validation test

Validation tests of the selected model are required at the end of this optimization. They compare the values of the response predicted by the model and that obtained

Table 12. Optimal field of formulation.

Factor	Superplasticizers SP103 (X_1)	Quantity of water (X_2)	W /C report (X_3)
value	2.5%	208	0.41

Table 13. Predicted and experimental test point values of the compressive strength at 28 days.

Factor	Real value	Coded value	Predicted response (MPa)	Experimental response (MPa)
Content of SP103 (X_1)	2.5	0.6	70.78	71.90
Quantity of water (ml) (X_2)	208	0.82		

experimentally when the optimal conditions retained by the same model are applied. The operative coordinates are as follows: $X_1 = 2.5$ %; and $X_2 = 208$.

Table 13 shows the values of the predicted and the experimentally obtained compression strength (under the experimental conditions given by the model).

According to Table 13, the predicted and the experimental values of the compressive strength are similar. So, there is no significant difference between the experimental response and that predicted. Then, the operational and optimal conditions providing to predict and improve the mechanical resistance to compression are successfully identified. Furthermore, the number of the tests as well as the mixing water quantity is decreased.

CONCLUSIONS

The objective of this study is to improve the mechanical performance in respect to compressive strength of high-performance concrete containing high water reducing and setting accelerating (SP103) superplasticizer. The response surface methodology (RSM) by the experimental design is used to determine the operating conditions of the studied factors while improving the mechanical resistance to compression and minimizing the number of tests, the cost and the quantity of mixing water used. The percentage of SP103 superplasticizer, the amount of the water used and 'W/C' ratio are optimized. In order to improve the compressive strength of this type of concrete and to obtain a mathematical model that predicts this compressive strength, the methodology of the experimental design was applied. The mathematical model advanced shows that the compressive strength depends on the linear term (β_1), relative to SP103 content, to the quadratic interactions (β_{11}) and to the proportions of SP103 and the quadratic terms (β_{12}) connected with the amounts of the mixing water. The validation of the model indicates that the optimal conditions to maximize the compressive strength refer to SP103 content $X_1 = 2.5$ %, mixing water amount $X_2 = 208$ and a fixed W/C ratio $X_3 = 0.41$. These results enabled to improve the compressive strength of the high-performance concrete formulated by SP103 superplasticizer on one hand, while on the other hand it is of a great economic and environmental interest due to: (i) production costs reduction; (ii) minimization of the number of tests required; (iii) decrease the mixing water quantity.

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