

STUDY ON USING MAXIMUM AMOUNT OF FLY ASH IN PRODUCING ULTRA-HIGH PERFORMANCE CONCRETE

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Abstract

In the present study, the synergic effects of cementitious materials in the ternary binder containing cement, silica fume, fly ash on the workability and compressive strength were evaluated by using the D-optimal design of Design-Expert 7. The ternary binder composed of 65 vol.-% cement, 15 vol.-% SF and 20 vol.-% FA at the W/F_v ratio of 0.50 is the optimum mixture proportions for the highest compressive strength of the UHPC. To produce the sustainable UHPC, high-volume fly ash ultra high performance concrete with a good flowability and 28-d compressive strength over 130 MPa can be produced with fly ash content up to 30 vol.-% in the binder.

Keywords: UHPC; high volume fly ash; silica fume; workability; compressive strength.

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1. Introduction

Ultra-high performance concrete (UHPC) is a new type of concrete being researched and used in many constructions [1, 2]. UHPC possesses good flowability, 28-d compressive strength over 130 MPa at the normal curing condition, very low porosity and high durability [3, 4]. To obtain these outstanding properties, UHPC commonly consists of a low water to binder ratio, high amount of Portland cement, silica fume (SF) and superplasticizer (SP) [3–5]. With high content of Portland cement and silica fume, UHPC is not only very expensive compared with normal and high performance concrete but also not environmentally friendly. In order to develop more sustainable and eco-efficient UHPC, various pozzolanic materials have been used as partial cement replacement in UHPC [6–8].

Silica fume is commonly pozzolanic material used in UHPC. It plays three main functions: 1) to fill the voids between particles to achieve a high packing density; 2) to improve the rheological properties by lubrication effects resulting from small and perfect spherical particles; and 3) to produce secondary hydration products by consumption of portlandite (the pozzolanic reaction). Hence, SF strongly influences properties of concrete [9–11]. In a Portland cement concrete with water cement ratio of 0.5, about 18.3% SF, referred to the weight of cement, is enough to totally consume $\text{Ca}(\text{OH})_2$ that is released from cement hydration [12]. However, the optimal SF content of UHPC is normally about 20-30 wt.-% of cement to improve the filler effect [13–15]. However, the high price of SF

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makes it as a non-desired material in producing UHPC. The other pozzolanic materials such as fly ash (FA), available in huge volume as a waste material with low cost and environmental problem can be used in UHPC [8, 16, 17]. When FA partially replaces Portland cement, the workability of the UHPC increases but its compressive strength decreases. When quartz powder is completely replaced by FA, the workability of the UHPC dramatically decreases with coarse FA and is constant with finer ones. The mixture containing fine FA to partially replace SF needs higher SP dosage and possesses slower compressive strength development in water at 20°C compared to the mixture containing SF.

The present study investigates synergic effects of SF and FA partially replacing cement on workability and compressive strength of UHPC at the ages of 3 and 28 days by using statistical analysis of the Design-Expert software. With the purpose of using FA as much as possible, workability and compressive strength of UHPC containing different FA and water contents were also studied in this study.

2. Materials and methods

2.1. Materials

Cementitious materials used in this study were ordinary Portland cement, fine fly ash (FA) and undensified powder of SF. Quartz sand was utilized as aggregate. Chemical compositions and physical properties of the materials are given in Table 1 and Table 2. Superplasticizer was a polycarboxylate ether type.

Table 1. Chemical composition of cementitious materials, (%)

| N° | Materials | SiO ₂ | Fe ₂ O ₃ | Al ₂ O ₃ | CaO | Na ₂ O | K ₂ O | MgO | L.O.I |
|----|-----------|------------------|--------------------------------|--------------------------------|------|-------------------|------------------|------|-------|
| 1 | Cement | 22.6 | 3.5 | 5.3 | 64.2 | 0.14 | 0.61 | 2.3 | 0.81 |
| 2 | SF | 92.6 | 1.85 | 0.9 | 0.32 | 0.39 | 1.20 | 0.85 | 1.60 |
| 3 | FA | 58.7 | 7.3 | 22.9 | 1.0 | 0.33 | 3.6 | 0.9 | 4.41 |

Table 2. Physical properties of materials

| N° | Items | Cement | SF | FA | Quartz sand |
|----|--|--------|-------|---------|-------------|
| 1 | Specific density, (g/cm ³) | 3.1 | 2.2 | 2.24 | 2.64 |
| 2 | Mean particle size (μm) | 21.1 | 0.151 | 7.87 | 313.45 |
| 3 | Compressive strength of cement (MPa) | 3 days | 28.7 | 28 days | 47.9 |

2.2. UHPC compositions and testing methods

UHPC has two main parts which are paste and aggregate particles. Typical UHPC mixtures are given in Table 3. The paste volume is 57 vol.-% of UHPC. W/F_v is the volume of water to the volume of fine materials (cementitious materials) ratio. The pozzolanic admixtures partially replace cement in volume. Superplasticizer (SP) dosage is 1.1% in solid content of cementitious materials.

UHPC was mixed with a total mixing time of 13 minutes based on the sequence shown in Fig. 1. Mini-cone slump flow of UHPC mixtures was determined 12 minutes after water addition. The slump

Table 3. Typical mix proportions of UHPC

| N° | Mixtures | Cement | Quartz sand | [kg/m ³] | | | w/b | W/F _v |
|----|----------|--------|-------------|----------------------|-------|-------------|-------|------------------|
| | | | | SF | FA | Total Water | | |
| 1 | 75:15:10 | 855.0 | 1135.2 | | 82.4 | | 0.191 | 0.55 |
| 2 | 65:15:20 | 741.0 | | 121.4 | 164.8 | 202.3 | 0.197 | |
| 3 | 55:15:30 | 541.5 | | | 247.1 | | 0.203 | |
| 4 | 75:15:10 | 883.5 | | | 85.1 | | 0.174 | |
| 5 | 65:15:20 | 765.7 | | 125.4 | 170.2 | 190.0 | 0.179 | |
| 6 | 55:15:30 | 647.9 | | | 255.4 | | 0.185 | |
| 7 | 75:15:10 | 914.0 | | | 88.1 | | 0.156 | |
| 8 | 65:15:20 | 792.1 | | 129.7 | 176.1 | 176.9 | 0.161 | |
| 9 | 55:15:30 | 670.2 | | | 264.2 | | 0.166 | |

flow values were measured after further 2 minutes without stroking. Samples 50 x 50 x 50 mm³ were formed without vibration, kept in moulds at 27°C, 95% relative humidity (RH) for 24h and followed by storing at 27°C, 100% RH until examination. Compressive strength of samples was tested in accordance with ASTM C109.

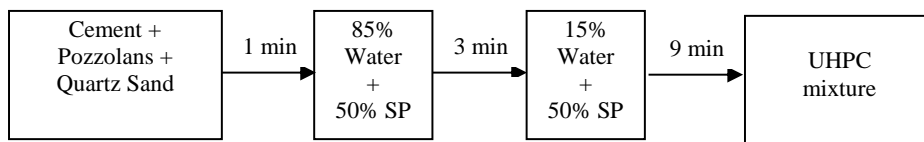


Figure 1. Mixing procedure of UHPC

2.3. Mixture design model

Concrete is a multivariate system and normally needs more than one important objective function. The classical method for optimizing mixture proportions is trial and error, or changing one ingredient and studying the effect of the ingredient on the response. It will be inefficient and costly. More importantly, they may not provide the economical mixture. Standard response surface designs, such as factorial designs or central composite design can use for optimizing concrete mixture in which the n mixture components have to be reduced to $n - 1$ independent factors by taking the ratio of two components [18]. However, changing the proportion of one ingredient immediately influences the proportion of the others because the mix proportions are limited to sum to 100%. Hence, different method is required for choosing appropriate experimental design and analyzing final results for all dependent variables of mixture. The mixture models are appropriate for these problems [18, 19]. Therefore, the mixture model with D-optimal design of Design-Expert 7 software was used in this study to evaluate the synergic effects of SF, FA and cement on workability and compressive strength at the ages of 3 and 28 days of UHPC.

3. Results and discussion

3.1. Design of D-optimal for the mixture model

Three cementitious materials: cement, SF and FA in volume content as mixture components of binder of UHPC. The three binder components are designated as A, B, C, respectively. The predicted responses, namely flowability and compressive strength at the ages of 3 and 28 days are designated as R_1 , R_2 and R_3 , respectively. All the other components of UHPC, mixing procedure, casting, treatment, and test methods were kept in constant for all mixtures. Based on preliminary tests, the range of the binder components was chosen:

$$\begin{aligned} A + B + C &= 100\% \\ 47.5\% \leq A &\leq 82.5\% \\ 7.5\% \leq B &\leq 22.5\% \\ 10\% \leq C &\leq 30\% \end{aligned}$$

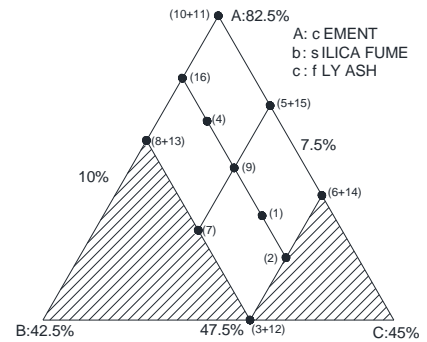


Figure 2. 16-run D-optimal design. Points with a (+) indicate replicates

The D-optimal design was chosen and assumed that a mixture quadratic model should be satisfactory to represent the effect of the mixture components on the predicted responses. The complete mixture quadratic model is in Eq. (1).

$$R = f(A, B, C) = \beta_1 A + \beta_2 B + \beta_3 C + \beta_{12} AB + \beta_{13} AC + \beta_{23} BC \quad (1)$$

where $\beta_1, \beta_2, \beta_3$ are linear coefficients; $\beta_{12}, \beta_{13}, \beta_{23}$ are cross product coefficients.

The designing experiments produced by the Design-Expert 7 are shown in Fig. 2 and Table 4. They are the actual mixture components. The complete model has 16 runs including 11 runs at different contents of the binder and 5 replicated runs to provide an estimate of error. The W/F_v ratio of 0.55 was used in 16 mixtures to make sure all the mixtures having sufficient flowability. The typical mix proportions of mixtures can be found in Table 3. Experimental results of mini-cone flow (R_1) and compressive strength at the age of 3 days (R_2) and 28 days (R_3) of 16 mixtures are also given in Table 4.

3.2. Statistical analysis

The 16 designed mixtures of UHPC in Fig. 2 and Table 4 were mixed and tested the slump flow, 3-d and 28-d compressive strength. The 16-designed run data is analyzed by Design-Expert 7. The first step in the analysis is to identify a suitable model. Even though the design selected the mixture quadratic model, other model may be suggested by the software to have a better fitness for the experimental data. With the input data, the fit summary suggests the mixture quadratic model for the responses of the slump flow and 28-d compressive strength, and the mixture special cubic model for the responses of 3-d compressive strength. The complete models are as follows:

$$R_1 \text{ (flowability)} = 52.78A - 1052.84B + 55.79C + 2392.61AB + 960.66AC + 1236.61BC \quad (2)$$

$$\begin{aligned} R_2 \text{ (3d strength)} &= -28.07A - 320.65B - 698.59C + 1173.44AB + 1619.68AC \\ &+ 4624.61BC - 10027.21ABC \end{aligned} \quad (3)$$

$$R_3 \text{ (28d strength)} = -11.0A - 911.16B - 606.19C + 1645.72AB + 1306.93AC + 2064.31BC \quad (4)$$

Table 4. 16-run D-optimal design with data

| Run | Cement | SF | FA | Experimental | | |
|-----|--------|-------|-------|--------------|-------------|-------------|
| | A [%] | B [%] | C [%] | R_1 [mm] | R_2 [MPa] | R_3 [MPa] |
| 1 | 60.0 | 15.0 | 25.0 | 292 | 56.2 | 110.3 |
| 2 | 55.0 | 15.0 | 30.0 | 305 | 52.3 | 102.1 |
| 3 | 47.5 | 22.5 | 30.0 | 283 | 51.4 | 92.3 |
| 4 | 70.0 | 15.0 | 15.0 | 262 | 66.1 | 109.5 |
| 5 | 72.5 | 7.5 | 20.0 | 260 | 76.5 | 101.5 |
| 6 | 62.5 | 7.5 | 30.0 | 290 | 70.7 | 95.9 |
| 7 | 57.5 | 22.5 | 20.0 | 280 | 57.7 | 109.2 |
| 8 | 67.5 | 22.5 | 10.0 | 263 | 79.1 | 104.8 |
| 9 | 65.0 | 15.0 | 20.0 | 273 | 63.2 | 120.5 |
| 10 | 82.5 | 7.5 | 10.0 | 200 | 61.3 | 79.5 |
| 11 | 82.5 | 7.5 | 10.0 | 205 | 62.5 | 81.5 |
| 12 | 47.5 | 22.5 | 30.0 | 280 | 52.1 | 94.8 |
| 13 | 67.5 | 22.5 | 10.0 | 257 | 78.8 | 106.9 |
| 14 | 62.5 | 7.5 | 30.0 | 285 | 69.3 | 93.8 |
| 15 | 72.5 | 7.5 | 20.0 | 272 | 74.2 | 103.8 |
| 16 | 75.0 | 15.0 | 10.0 | 258 | 69.9 | 104.8 |

The adequacy of the complete regression models (Eqs. (2), (3) and (4)) is assessed by using some standards. Firstly, the analysis of variance (ANOVA) is used to check the significance of the models. All of the models are significant. Their lacks of fit are not significant (Table 5, 6 and 7). The

Table 5. ANOVA for the complete mixture quadratic model of the workability

| Source | Sum of Squares | Df | Mean Square | F Value | p-value Prob \geq F | |
|-----------------------|----------------|----------------|-------------|---------|--------------------------|-----------------|
| Model | 11604.88 | 5 | 2320.98 | 40.50 | \leq 0.0001 | significant |
| <i>Linear Mixture</i> | 8466.01 | 2 | 4233 | 73.87 | \leq 0.0001 | |
| <i>AB</i> | 584.73 | 1 | 584.73 | 10.20 | 0,0096 | |
| <i>AC</i> | 277.63 | 1 | 277.63 | 4.84 | 0.0523 | |
| <i>BC</i> | 121.57 | 1 | 121.57 | 2.12 | 0.1759 | |
| Residual | 573.06 | 10 | 57.31 | | | |
| <i>Lack of Fit</i> | 453.56 | 5 | 90.71 | 3.8 | 0.0848 | Not significant |
| <i>Pure Error</i> | 119.50 | 5 | 23.90 | | SD | 7.57 |
| Cor total | 12177.94 | 15 | | | Mean | 266.56 |
| R-Squared | 0.9529 | Adj R-Squared | 0.9294 | C.V% | 2.84 | |
| Pred R-Squared | 0.8885 | Adeq Precision | 19.987 | PRESS | 1357.34 | |

Table 6. ANOVA for the complete mixture special cubic model of the 3d strength

| Source | Sum of Squares | Df | Mean Square | F Value | p-value Prob > F | |
|-----------------------|----------------|----|----------------|---------|---------------------|-----------------|
| Model | 1335.83 | 6 | 222.64 | 154.39 | < 0.0001 | significant |
| <i>Linear Mixture</i> | 435.53 | 2 | 217.76 | 151.01 | < 0.0001 | |
| <i>AB</i> | 1.69 | 1 | 1.69 | 1.17 | 0.3075 | |
| <i>AC</i> | 136.86 | 1 | 136.86 | 94.91 | < 0.0001 | |
| <i>BC</i> | 0.63 | 1 | 0.63 | 0.44 | 0.5248 | |
| <i>ABC</i> | 175.90 | 1 | 175.90 | 121.98 | < 0.0001 | |
| Residual | 12.98 | 9 | 1.44 | | | |
| <i>Lack of Fit</i> | 8.34 | 4 | 2.09 | 2.25 | 0.1987 | Not significant |
| <i>Pure Error</i> | 4.64 | 5 | 0.93 | | SD | 1.20 |
| Cor total | 1348.80 | 15 | | | Mean | 65.08 |
| R-Squared | 0.9904 | | Adj R-Squared | 0.9840 | C.V% | 1.85 |
| Pred R-Squared | 0.9643 | | Adeq Precision | 34.599 | PRESS | 48.13 |

adjusted R-squared and the predicted R-squared of the responses are suitable. Hence, these models are adequate. Some of the coefficients in the complete models (Eqs. (2), (3) and (4)) are insignificant and could be eliminated. In this case, there is no advantage to the reduced models because the adjusted R-squared is only slightly changed. Moreover, the interactions should not be removed in the mixture model, especially with the mixture quadratic model [18, 19]. Therefore, the complete models in the Eqs. (2), (3) and (4) should be used for further navigations.

Table 7. ANOVA for the complete mixture quadratic model of the 28d strength

| Source | Sum of Squares | Df | Mean Square | F Value | p-value Prob > F | |
|-----------------------|----------------|----|----------------|---------|---------------------|-----------------|
| Model | 1642.45 | 5 | 328.49 | 47.06 | < 0.0001 | significant |
| <i>Linear Mixture</i> | 254.23 | 2 | 127.12 | 18.21 | 0.0005 | |
| <i>AB</i> | 276.65 | 1 | 276.65 | 39.63 | < 0.0001 | |
| <i>AC</i> | 513.84 | 1 | 513.84 | 73.61 | < 0.0001 | |
| <i>BC</i> | 338.78 | 1 | 338.78 | 48.53 | < 0.0001 | |
| Residual | 69.81 | 10 | 6.98 | | | |
| <i>Lack of Fit</i> | 57.63 | 5 | 11.53 | 4.73 | 0.0566 | Not significant |
| <i>Pure Error</i> | 12.18 | 5 | 2.44 | | SD | 2.64 |
| Cor total | 1712.26 | 15 | | | Mean | 100.70 |
| R-Squared | 0.9592 | | Adj R-Squared | 0.9388 | C.V% | 2.62 |
| Pred R-Squared | 0.9061 | | Adeq Precision | 21.322 | PRESS | 160.81 |

3.3. Influence of cementitious materials on the flowability of UHPC

To interpret the influence of the cementitious materials on the mini-cone slump flow of UHPC, 3D response surface and contour plots of the flowability response in dependence of cement, SF and FA contents have been plot in Fig. 3.

Results in Fig. 3 show that increasing the FA content improves the workability of UHPC at all levels of SF. At the low FA contents, the flowability of UHPC strongly increases when SF content increases. But at the higher contents of FA, the flowability of UHPC increases initially and then decreases when the SF content increases. Therefore, with the aim to obtain the maximum slump flow of UHPC, it needs adjusting the variables to a high content of FA with an optimum content of SF (Fig. 3).

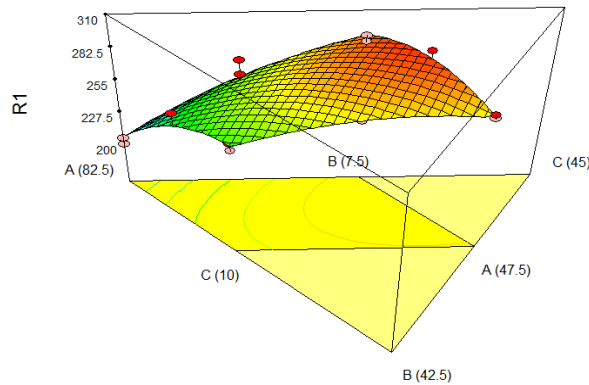


Figure 3. Response surface and contour plots of flowability of UHPC

3.4. Influence of cementitious materials on compressive strength of UHPC

Similar to the flowability response, 3D response surface and contour plots of the 3-day and 28-day compressive strength responses in dependence of cement, SF and FA contents have been present in Figs. 4 and 5, respectively.

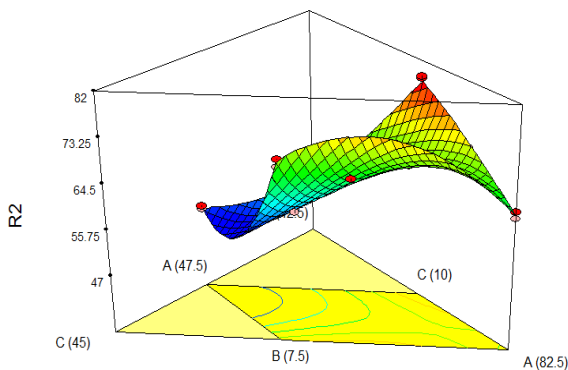


Figure 4. Response surface and contour plots of 3-d compressive strength of UHPC

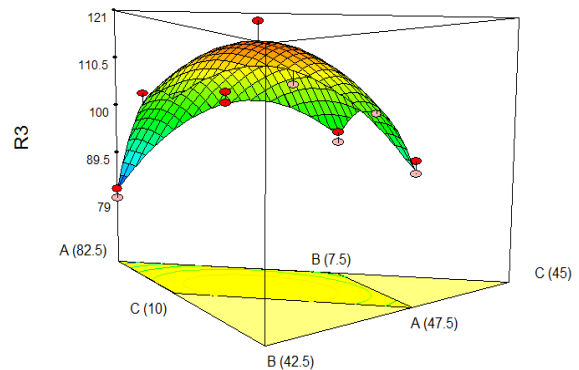


Figure 5. Response surface and contour plots of 28-d compressive strength of UHPC

The results in Fig. 4 illustrate that at the low content of FA, i.e $C = 10\%$, the compressive strength at the age of 3 days of UHPC increases when the content of SF increases. Meanwhile, at

the SF content of 7,5%, the 3-d strength of UHPC initially increases and then decreases during the increase of the content of FA. But at high contents of SF or FA, the increase of the other mineral admixture will induce low 3-d compressive strength of UHPC (Fig. 4).

3D response surface and contour plots of the 28-day compressive strength response in Fig. 5 show that at any content of FA, compressive strength of UHPC initially increases and then decreases when the content of SF increases. And at any content of SF, there is an optimized content of FA which enables UHPC containing SF to obtain the highest compressive strength at the age of 28 days. It means that the highest compressive strength comes from a ternary binder composed of cement, SF and FA (Fig. 5).

3.5. Optimization of mix proportions of UHPC containing SF and FA

The optimization tool of the Design-Expert 7 software is inducted to find the optimal proportions of UHPC containing SF and FA. The input criteria are present in Table 8. The program offers some solutions. The best solution is chosen in terms of the highest compressive strength (Table 8).

The results of the slump flow, compressive strength of the experimental mixture and Design-Expert's mixture in Table 8 are similar. Thus, UHPC with the binder containing 15 vol.-% SF and 20 vol.-% FA is selected as the optimal mix proportions.

Table 8. Experimental proportions versus optimized proportions

| N° | Material | Variable | Goal | Constrains | Unit | The mix proportions having the highest strength | |
|----|-----------------------|----------|----------|------------|----------|---|--------------|
| | | | | | | Design-Expert | Experimental |
| 1 | Cement | A | | 47.5-82.5 | | 63.4 | 65 |
| 2 | SF | B | In range | 7.5-22.5 | [vol.-%] | 17.3 | 15 |
| 3 | FA | C | | 10.0-30.0 | | 19.3 | 20 |
| 4 | Slump flow | | In range | 200-305 | [mm] | 283 | 273 |
| 5 | Comp. strength at 3d | | In range | 51.4-79.1 | | 61.0 | 63.2 |
| 6 | Comp. strength at 28d | | Maximum | 79.5-120.5 | [MPa] | 116.8 | 120.5 |

3.6. High-volume fly ash UHPC

The compressive strength at the age of 28 days of the selected UHPC in section 3.3 is still lower than 130 MPa. This mixture has a W/F_v of 0.55 with very high mini-cone slump flow. With the purpose of producing UHPC containing high volume of FA, workability and compressive strength of UHPC containing 15% SF with different contents of FA and W/F_v ratios are shown in Table 9 and Fig. 6.

The results in Table 9 and Fig. 6(a), (b), (c) show that at the same water content, the more the FA content, the higher the flowability and the lower the compressive strength at the ages of 3 and 7 days. At the W/F_v ratios of 0.55 and 0.50, UHPC possesses the highest 28-d compressive strength at the FA content of 20%. Meanwhile, the 28-d strength of mixture with the W/F_v ratio of 0.45 still increases when the FA content increases (Table 9 and Fig. 6(d)). Normally, when the water content decreases, the workability of the mixture reduces. The flowability of the mixtures dramatically decreases at the W/F_v ratio of 0.45. With the same cementitious content, UHPC has the maximum strength at

Table 9. Workability and compressive strength of UHPC at different FA and water contents

| N° | Mixture | W/Fv | Workability, mm | Compressive strength, MPa | | |
|----|----------|------|-----------------|---------------------------|--------|---------|
| | | | | 3 days | 7 days | 28 days |
| 1 | 75:15:10 | 0.55 | 258 | 69.9 | 92.6 | 104.8 |
| 2 | 65:15:20 | | 273 | 63.2 | 83.5 | 120.5 |
| 3 | 55:15:30 | | 305 | 52.3 | 75.8 | 102.1 |
| 4 | 75:15:10 | 0.50 | 245 | 79.1 | 118.7 | 132.8 |
| 5 | 65:15:20 | | 270 | 73.7 | 104.0 | 142.7 |
| 6 | 55:15:30 | | 295 | 68.5 | 96.8 | 135.5 |
| 7 | 75:15:10 | 0.45 | 190 | 74.8 | 101.6 | 107.2 |
| 8 | 65:15:20 | | 235 | 68.3 | 80.5 | 115.3 |
| 9 | 55:15:30 | | 245 | 55.9 | 72.3 | 121.3 |

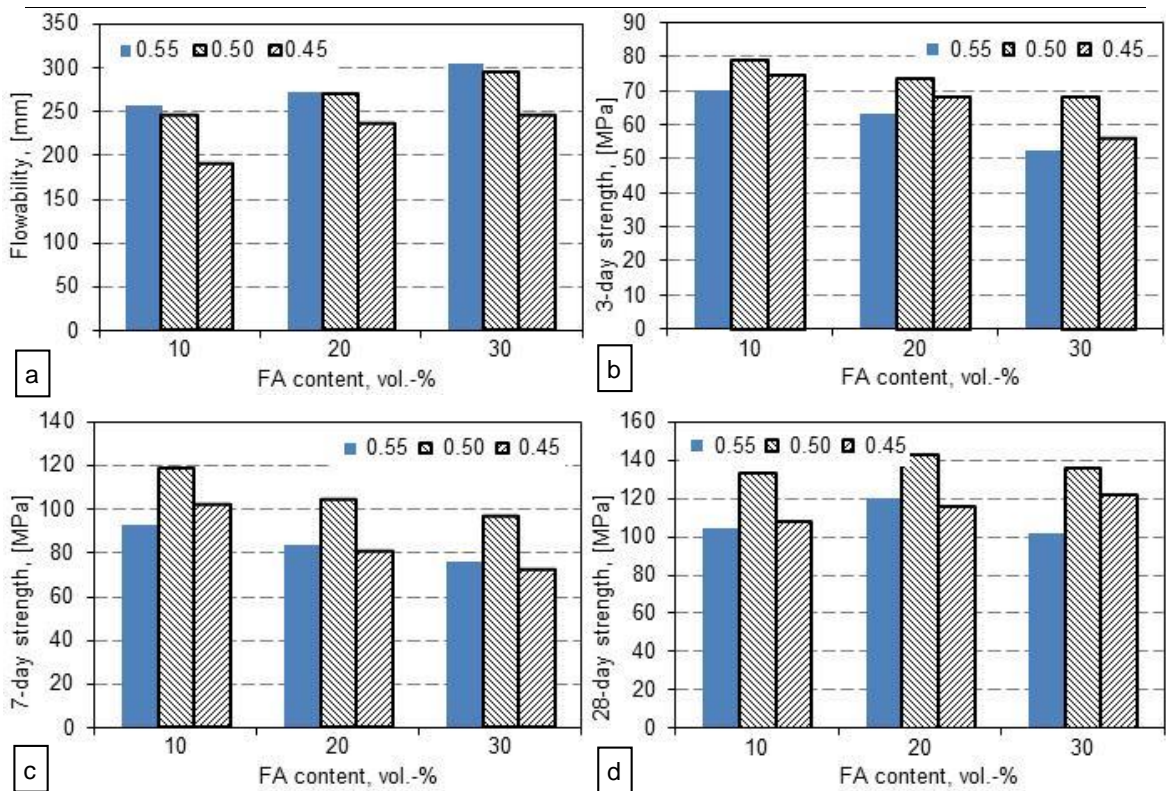


Figure 6. Effect of FA content and W/Fv on: a) Flowability; b) 3d strength; c) 7d strength and d) 28d strength

the W/Fv ratio of 0.50. At the W/Fv ratio of 0.50, the 28-d compressive strength of the mixture containing 20%FA obtains over 140 MPa and the mixture containing 30% FA has the strength of

135.5 MPa. Therefore, the high-volume fly ash ultra-high performance concrete can be produced from a ternary binder containing 15 vol.-% SF and 30 vol.-% FA at the W/F_v ratio of 0.50.

4. Conclusions

The following conclusions can be drawn from the results of this study:

- The mixture models of flowability and compressive strength of UHPC with the binder containing three mixture components of cement, fly ash and silica fume using D-optimal design of Design-Expert 7 fitted well with the experimental data. It can be analyzed the influence of the variables on the workability and compressive strength of UHPC by using 3D response surface and contour plots.

- Fly ash improves flowability and reduces compressive strength of UHPC at the early age of 3 days. At the age of 28 days, the ternary binder composed of 65 vol.-% cement, 15 vol.-% SF and 20 vol.-% FA at the W/F_v ratio of 0.50 is the optimum mixture proportions for the highest compressive strength of the UHPC in this study.

- With the purpose of using as much as FA in UHPC, high-volume fly ash ultra high performance concrete with a good flowability and 28-d compressive strength over 130 MPa can be produced with fly ash content up to 30 vol.-% in the binder.

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