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# Experiment design to evaluate interaction of high-range water-reducer and antiwashout admixture in high-performance cement grout

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**Abstract**

The injection of cement grout in water-saturated medium and the sealing of anchorages require the use of grout with high resistance to water dilution to enhance in-situ performance. This can be achieved by reducing the W/C and incorporating an antiwashout admixture (AWA) to enhance the stability. Such admixture can increase the viscosity and yield stress and necessitate higher dosage of high-range water-reducer (HRWR) to maintain the desired fluidity. Cement-based grouts with 0.30 to 0.50 W/C and different combinations of AWA and HRWR were evaluated. The study was undertaken to highlight the influence of W/C and dosage of chemical admixtures on fluidity, washout resistance, and residual compressive strength (RCS) of the underwater-cast grout. Statistical models established using a statistical design of experiments indicate that the W/C has greater effect on changes in minislump flow, washout, and RCS than the concentrations of HRWR and AWA. On the other hand, for mixtures prepared with a fixed W/C of 0.40, the models show that measured responses are highly affected by the dosages and interaction of both admixtures. Trade-off between fluidity and washout resistance and means to optimize mixture proportioning to enhance the resistance to water dilution without adversely affecting fluidity are discussed. 

*Keywords:* Anchorage; Antiwashout admixture; Cement grout; Experiment design; High-range water-reducer; Underwater casting; Repair

## 1. Introduction

Antiwashout admixtures (AWAs) typically employed in cement-based systems are water-soluble polymers that increase the viscosity of mixing water and enhance water retention and the homogeneity of suspended solid particles. The main advantage of incorporating an AWA in cement-based mixtures is to reduce bleeding, segregation, and washout loss.

The incorporation of AWA increases both the yield stress and plastic viscosity of cement-based materials [1–3]. When used in conjunction with high-range water-reducer (HRWR), a highly flowable mixture with low yield stress can be obtained with relatively high plastic viscosity to control sedimentation and water dilution. When incorporated at high dosages, the HRWR can delay setting time and increase the period during which the suspension remains in plastic state leading to greater risk of heterogeneity of the hardened material. An optimization is therefore necessary to establish balance between the HRWR and AWA contents for a given W/C to ensure suitable flow in submerged conditions. Successful placement of grout saturated medium requires the use of highly fluid yet stable mixture that can achieve the required properties to facilitate placement and ensure good performance. Because of the opposing mode of actions of HRWR and AWA on flow properties, mixture optimization often necessitates several trial batches to achieve balance between the admixtures and W/C to ensure suitable fluidity, stability, and mechanical properties.

The study presented here was undertaken to establish statistical models that can describe the relevant significance of each of the HRWR, AWA, and W/C on fluidity, stability, and residual compressive strength (RCS). Such mathematical models can be used to develop mixture proportioning guidelines given the relative weights of each mixture parameter on fluidity, washout loss, and strength.

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## 2. Model and statistical design of experiments

Current optimization procedure for developing high-performance cement-based materials is mainly based on experiments rather than on a comprehensive approach. In general, the approach consists in selecting and testing a first trial batch, evaluate the results, and then adjust the mixture proportions and test further mixtures until the required properties are achieved. In contrast, statistical experimental design methods are rigorous techniques for both achieving desired properties and establishing an optimized mixture for a given constraint while minimizing the number of trials [4–8]. Using this approach, a mathematical model describing the main influence and two-way interactions of various parameters on a given property can be established.

The objective of the first part of this study is to evaluate the influence of three independent variables (W/C, HRWR, and AWA) on fluidity, washout mass loss, and RCS using the statistical design of experiments. The modeled responses are assumed to be linearly dependent on the level of each independent variable. Central points are prepared to test the validity of the linearity assumption. Such an approach allows evaluation of the adequacy of linearity of the model, thus providing an indication of whether it is necessary or not to run a quadratic one. The two-level statistical design of experiments for three independent variables ($k=3$) consists of eight ($8=2^k$) factorial points where each variable is fixed at two different levels. Initially, the W/C is varied from 0.3 to 0.5, the HRWR dosage from 0.8% to 1.7%, and the AWA content from 0.02% to 0.06%.

The second part of this investigation consists of evaluating the effects of HRWR and AWA dosages and their interactions on fluidity, washout mass loss, and RCS for grouts prepared with a fixed W/C of 0.40. Based on the derived models, a response surface experimental plan is carried out to minimize the random error observed with the linear models, thus enhancing their adequacy. The response surface experimental plan consists of a central composite design (CCD). In case of two independent variables ($k=2$), a CCD plan consists of four ($N_f=2^k$) factorial points, four ($4=2k$) axial points at a distance $\alpha$ from the origin, and at least one central point. However, in this study five central points were considered, thus ensuring a constant variance of predicted values within the modeled region [4]. The value of $\alpha$ is fixed such as the design is rotatable, thus at an equal distance from the origin the predicted values should have the same variance [4–6]. The value of $\alpha$ is taken here as ${N_f}^{1/4}$ with $N_f$ being the number of factorial points ($\alpha=\pm 1.4$).

Initial levels of AWA and HRWR concentrations were varied from 0% to 0.03% and 0.6% to 1%, respectively. It must be noted that the actual variable ranges are transformed to dimensionless coded values to facilitate calculation and analysis. The coded values are expressed as follows [Eq. (1)]:

$$\text{Codified value} = \frac{\text{absolute value} - \text{central value}}{\frac{1}{2}\text{range between maximum and minimum values}} \quad (1)$$

For example, a dosage of 0.02% AWA corresponds to a codified value of $-1$ in the case of the two-level statistical design run on three independent variables.

## 3. Material properties and test procedures

A Type 10 Canadian cement (complying with Canadian Standards CSA-CAN A5) similar to ASTM C150 Type I cement was used. Welan gum, a high molecular-weight polysaccharide produced by a special biofermentation, was used for the AWA. The gum was prehydrated with water at 1% concentration at high shear rate for 15 min. A naphthalene–sulfonic acid formaldehyde condensate was used for the HRWR. The HRWR has a solid content of 42% and a specific gravity of 1.21.

All paste mixtures, referred here as grout mixtures (neat cement grout), were prepared in batches of 4 l and mixed using a high-speed mixer at approximately 3000 rpm. The temperature of the mixing water was set at $14\pm2$°C to compensate for heating during mixing. All grouts had constant temperature of $22\pm2$°C at the end of mixing. The batching sequence consisted of adding water and HRWR to the mixer along with the prehydrated AWA. The cement was then introduced gradually over 60 s. The grout was mixed for 60 s, and after a rest period of 30 s, the mixing was resumed for an additional 60 s.

The fluidity was evaluated using the minislump test determined immediately after the end of mixing, corresponding to 5 min from initial contact of cement with water. The washout test consists of measuring the mass loss of a 500-ml grout sample cast in water. The grout is introduced into a funnel positioned 10 mm above a 500-ml beaker full with water. The grout flows freely in water displacing an equivalent volume of water along with diluted cement particles. The washout mass loss is evaluated as the difference of mass before and after flow and displacement of water [1,9].

The RCS of the grout was evaluated by testing $50\times100$-mm cylinders cast above- and underwater. The latter was determined on cylinders filled with water and cast by pouring the grout from the top surface. After $24\pm1$ h, the specimens were demolded and stored in lime-saturated water until the age of testing of 28 days. Each of the reported strength values corresponds to the mean of three cylinders. RCS is evaluated as the ratio between compressive strength of samples cast underwater and in air.

## 4. Test results and discussions

Table 1 summarizes the mixture proportions and test results for mixtures investigated in Phase 1 and used to establish statistical models for the minislump, washout mass loss, and the 28-day RCS responses. The coefficients of variation for compressive strength measurements were less than 10%. The values of initial and expanded parameters and mixtures properties considered in the CCD plan are summarized in Table 2.

### 4.1. Empirical linear models for minislump, washout, and RCS responses (modeling the effect of HRWR, AWA, and W/C)

The linear model associated with a two-level statistical design in case of three independent variables (HRWR, AWA, and W/C) is expressed as follow:

$$Y = a_0 + a_1\mathrm{HRWR} + a_2\mathrm{AWA} + a_3\mathrm{W/C} + a_4\mathrm{HRWR{\cdot}AWA} + a_5\mathrm{HRWR{\cdot}W/C} + a_6\mathrm{AWA{\cdot}W/C} + E \quad (2)$$

The third order interaction is usually neglected. In Eq. (2), coefficients ($a_i$) represent model constants (contribution of independent variables on the response), and $E$ is the random error term representing the effects of uncontrolled variables, i.e., not included in the model. The model constants are determined by multilinear regression analysis and are assumed to be normally distributed. The error is assumed to be random and normally distributed, so the residual terms, which represent the difference between observed and predicted values, should exhibit similar properties [5,6]. Analysis of variance is used to test the significance of regression, and $t$ tests are performed to identify the nonsignificant (NS) terms, which are then eliminated from the model. The derived models for the minislump, washout mass, and RCS responses as well as $t$ ratio for each term are summarized in Table 3. A negative estimate of a given parameter indicates that the response decreases with the increase in the value of the parameter.

The derived coefficients were obtained with 90% confidence interval. The significance of each variable on a given response is investigated using $t$ test values based on Student's distribution. Indeed, for each response, the $t$ ratio is compared to ($t_{\beta/2,\ \nu}$) Student's variable, where $(1-\beta)$ is the confidence interval, $\nu$ is the degree of freedom (DF) equal to $(n-k)$, $n$ is the number of experiments and $k$ is the number of unknown coefficients in the model. An independent variable is stated to have a significant effect on a given response when the $t$ ratio associated to that variable is greater than ($t_{\beta/2,\ \nu}$) Student's variable. For example, considering the minislump model, the test of significance of each term included in the model is carried out by comparing the absolute value of $t$ ratio with ($t_{0.05,\ 1}$) value corresponding to 6.3. Step-by-step elimination of parameter with the lowest effect is adopted. All terms are then recalculated, and test of significance for each term is repeated. The elimination of the nonsignificant interactions corresponding to HRWR·AWA and AWA·W/C interactions leads to a new Student's variable ($t_{0.05,\ 3}$) of 2.4.

The derived models for the minislump, washout mass loss, and RCS are then given in Table 4. As can be observed, all $t$ ratios are greater than the critical value of 2.4 for the first two models and 2.13 for the RCS model. The analysis of variance for the established models is given in Table 5.

The derived linear models for the minislump, washout, and RCS can be expressed as follows:

$$\mathrm{Minislump} = 129.4 + 21.9\mathrm{W/C} + 11.9\mathrm{HRWR} - 11.9\mathrm{AWA} - 5.6\mathrm{HRWR{\cdot}W/C} \quad (3)$$

$$\mathrm{Washout} = 10.3 + 3.3\mathrm{W/C} - 2\mathrm{AWA} + 1.8\mathrm{HRWR} - 0.8\mathrm{HRWR{\cdot}W/C} \quad (4)$$

$$\mathrm{RCS} = 68.4 - 5.4\mathrm{W/C} + 3.8\mathrm{AWA} - 3.2\mathrm{HRWR} \quad (5)$$

The significance of the derived models can be evaluated by comparing the $F$ value to $F_{\beta,\ k,\ n-k}$ tabulated in Fisher–Snedecor distribution. The derived regression is considered to be significant, i.e., the fluctuation due to the independent variables is mainly explained by the model, if $F$ value is greater than $F_{\beta,\ k,\ n-k-1}$. For a 90% confidence interval, or

Table 1
Mixture proportions and results for grouts used in the two-level factorial design

| | Mix proportions | | | | Test results | | |
|---|---|---|---|---|---|---|---|
| Mix | AWA, % (g/l)[a] | HRWR, % (g/l)[a] | Cement (kg/l) | W/C | Minislump (mm) | Washout (%) | 28-day RCS (%) |
| − − − | 0.02 (0.32) | 0.8 (12.8) | 1.6 | 0.3 | 100 | 7.0 | 71.4 |
| − − + | 0.02 (0.24) | 0.8 (9.6) | 1.2 | 0.5 | 160 | 14.0 | 63.6 |
| + − − | 0.06 (0.96) | 0.8 (12.8) | 1.6 | 0.3 | 80 | 2.0 | 85.5 |
| + − + | 0.06 (0.72) | 0.8 (9.6) | 1.2 | 0.5 | 130 | 11.0 | 66.7 |
| − + − | 0.02 (0.32) | 1.7 (27.2) | 1.6 | 0.3 | 140 | 12.0 | 65.6 |
| − + + | 0.02 (0.24) | 1.7 (20.4) | 1.2 | 0.5 | 165 | 16.0 | 60.0 |
| + + − | 0.06 (0.96) | 1.7 (27.2) | 1.6 | 0.3 | 110 | 7.0 | 72.6 |
| + + + | 0.06 (0.72) | 1.7 (20.4) | 1.2 | 0.5 | 150 | 13.0 | 63.6 |
| 000 | 0.04 (0.56) | 1.25 (17.5) | 1.4 | 0.4 | 125 | 10.5 | 67.9 |
| 000 | 0.04 (0.56) | 1.25 (17.5) | 1.4 | 0.4 | 130 | 11.0 | 69.1 |
| 000 | 0.04 (0.56) | 1.25 (17.5) | 1.4 | 0.4 | 135 | 11.0 | 70.4 |

[a] Percentage by mass of cement.

Table 2
Mixture composition and test results of grout used to establish two-level composite design models (W/C = 0.40, cement = 1.4 kg/l)

| | Mix proportions | | | | Test results | | |
|---|---|---|---|---|---|---|---|
| | AWA, % (g/l)[a] | HRWR, % (g/l)[a] | Coded AWA | Coded HRWR | Minislump (mm) | Washout (%) | 28-day RCS (%) |
| Initial points | 0 (0) | 0.6 (8.4) | − 1 | − 1 | 120 | 11.6 | 64.8 |
| | 0 (0) | 1 (14.0) | − 1 | 1 | 145 | 15.0 | 61.5 |
| | 0.03 (0.42) | 0.6 (8.4) | 1 | − 1 | 85 | 2.3 | 85.5 |
| | 0.03 (0.42) | 1 (14.0) | 1 | 1 | 125 | 12.0 | 67.9 |
| Axial points | 0 (0) | 0.8 (11.2) | − 1.4 | 0 | 140 | 15.0 | 61.1 |
| | 0.036 (0.50) | 0.8 (11.2) | 1.4 | 0 | 110 | 7.0 | 75.5 |
| | 0.015 (0.21) | 0.52 (7.28) | 0 | − 1.4 | 80 | 4.0 | 77.8 |
| | 0.015 (0.21) | 1.08 ((15.1) | 0 | 1.4 | 145 | 16.0 | 59.6 |
| Central points | 0.015 (0.21) | 0.8 (11.2) | 0 | 0 | 130 | 12.5 | 68.5 |
| | 0.015 (0.21) | 0.8 (11.2) | 0 | 0 | 130 | 12.7 | 66.0 |
| | 0.015 (0.21) | 0.8 (11.2) | 0 | 0 | 135 | 13.0 | 64.2 |
| | 0.015 (0.21) | 0.8 (11.2) | 0 | 0 | 135 | 12.6 | 67.9 |
| | 0.015 (0.21) | 0.8 (11.2) | 0 | 0 | 130 | 12.0 | 66.0 |

[a] Percentage by mass of cement.

10% risk, $F_{0.1,\ 4,\ 3}$ and $F_{0.1,\ 3,\ 4}$ values are 4.19 and 5.34, respectively. The $F$ values for minislump and washout models presented in Table 5 are greater than 4.19 and $F$ value for RCS is greater than 5.34. Therefore, the derived model can be considered to be significant in describing the effect of HRWR, AWA, and W/C on the modeled responses.

The W/C is shown to exhibit the greatest effect on all three measured responses. The increase in W/C has approximately twice greater influence on increasing minislump than the increase in HRWR concentration or the decrease in AWA dosage (21.9 vs. 11.9 or − 11.9). The models [Eqs. (3)–(5)] show that a decrease in W/C is more efficient in lowering washout loss than an increase in the AWA dosage. A considerable increase in HRWR concentration would then be required to compensate for loss of fluidity as W/C greatly affects minislump consistency.

By comparing the effects of HRWR and AWA on the modeled responses, it can be observed that the effect of AWA on minislump is compensated for by the addition of HRWR ( − 11.9 vs. 11.9). Indeed, the decrease of AWA dosage has the same influence as that of increasing HRWR content on minislump. On the other hand, the increase of AWA dosage has a slightly greater effect on enhancing washout and residual strength than the decrease in HRWR dosage ( − 2 vs. 1.8 and − 3.2 vs. 3.8). This suggests that the interaction between AWA and HRWR has no significant influence on modeled responses. This result may be due to the effect of the uncontrolled variable, which is the free water content in the system that varies with W/C. The effect of HRWR–AWA combination cannot be accurately evaluated. Indeed, the AWA and HRWR dosages are expressed in terms of cement mass, which is varying from one mixture to another according to the W/C value. Therefore, their effects seem to be more dominated by the W/C that directly governs the free water content. The nonsignificant HRWR–AWA interaction can be, on the other hand, due to the relatively wide range of AWA and HRWR admixtures used in the modeled region. Varying the parameters over a wide range (W/C from 0.30 to 0.50, etc.) may not permit locating a region where the variables show a significant effect [4]. For example, the influence of HRWR is limited when used with a high W/C mixture. On the other hand, the significant effect of variables cannot be adequately evaluated using a limited modeled region where the free water content is not significant. Further information regarding the interaction between AWA and HRWR can be established by varying the AWA and HRWR dosages with the W/C fixed to secure a given strength.

### 4.2. Accuracy and limitation of linear models

The accuracy of the derived models is assessed by comparing the residual standard deviation and the standard deviation

Table 3
Estimates and $t$ test values for each term included in linear models

| | Minislump (mm) | | Washout mass (%) | | RCS (%) | |
|---|---|---|---|---|---|---|
| Term | Estimate | $t$ ratio | Estimate | $t$ ratio | Estimate | $t$ ratio |
| Intercept | 129.4 | 41.4 | 10.3 | 35.5 | 68.4 | 99.4 |
| HRWR | 11.9 | 3.8 | 1.8 | 6.1 | − 3.2 | − 4.3 |
| AWA | − 11.9 | − 3.8 | − 2 | − 6.9 | 3.8 | 5.2 |
| W/C | 21.9 | 7.0 | 3.3 | 11.3 | − 5.4 | − 7.4 |
| HRWR·AWA | 0.6 | 0.2 | 0 | – | − 0.5 | − 0.7 |
| HRWR·W/C | − 5.6 | − 1.8 | − 0.8 | − 2.6 | 1.6 | 2.2 |
| AWA·W/C | 0.6 | 0.2 | 0 | – | − 1.5 | − 2.0 |

Table 4
Significant estimates and $t$ test values for each term included in linear models

| | Minislump (mm) ($R^2$=.97) | | Washout mass (%) ($R^2$=.98) | | RCS (%) ($R^2$=.91) | |
|---|---|---|---|---|---|---|
| Term | Estimate | $t$ ratio | Estimate | $t$ ratio | Estimate | $t$ ratio |
| Intercept | 129.4 | 69.0 | 10.3 | 35.5 | 68.4 | 58.8 |
| HRWR | 11.9 | 6.3 | 1.8 | 6.1 | − 3.2 | − 2.7 |
| AWA | − 11.9 | − 6.3 | − 2 | − 6.9 | 3.8 | 3.3 |
| W/C | 21.9 | 11.7 | 3.3 | 11.3 | − 5.4 | − 4.6 |
| HRWR·AWA | NS | – | 0 | – | NS | – |
| HRWR·W/C | − 5.6 | − 3.0 | − 0.8 | − 2.6 | NS | – |
| AWA·W/C | NS | – | 0 | – | NS | – |

Table 5
Analysis of variance of minislump, washout, and RCS models

| | Minislump ($R^2$=.97)[a] | | | Washout mass ($R^2$=.98)[a] | | | RCS ($R^2$=.91)[a] | | |
|---|---|---|---|---|---|---|---|---|---|
| | DF | SS | $F$ ratio | DF | SS | $F$ ratio | DF | SS | $F$ ratio |
| Model | 4 | 6337.5 | 56.3 | 4 | 145.5 | 54.6 | 3 | 426.0 | 13.1 |
| Error | 3 | 84.4 | | 3 | 2.0 | | 4 | 43.3 | |
| Total | 7 | 6421.9 | | 7 | 147.5 | | 7 | 469.3 | |

[a] $R^2$ is calculated as the ratio between $SS_{model}$ and $SS_{total}$.

calculated from the central points. These standard deviation values should be close to each to other. The residuals are the differences between measured and predicted values. Furthermore, the predicted error sum of square (PESS), i.e., the sum of residual squares divided by the residual DF is considered. An error sum of square close to the standard deviation measured on the central points indicates a good fitting.

In general, the residual deviation and standard deviation measured on central points for the minislump model lie close to each to other (Table 6). On the other hand, the predicted error of sum of square lies close to the standard deviation measured on central points. However, for the washout and RCS models the predicted error of sum of square is almost three times higher than that obtained from central points. In addition, the standard deviation measured on central points is higher than the residual standard deviation. Thus, it can be expected that the prediction of minislump will be acceptable, but that of the washout mass and RCS responses may result in a considerable error.

An additional quantitative evaluation of the accuracy is investigated on other mixtures prepared with a fixed W/C of 0.40 to compare predicted-to-measured responses. This W/C represents the center of the modeled W/C region and is selected to minimize the error due to variations in W/C. Indeed, the error due to a given variable on the modeled response increases with the distance from the central point. The dosages of the HRWR and AWA parameters were selected to cover a wide area within the modeled region (Table 7).

The mean ratio ($\sigma$) and coefficient of variation (COV) of the predicted-to-measured values for the investigated responses are as follows:

- Minislump model: $\sigma = 1.03$ and COV = 3.3%
- Washout model: $\sigma = 1.3$ and COV = 18.6%
- RCS model: $\sigma = 0.99$ and COV = 2%

Table 6
Statistical measures on minislump, washout, and RCS predictions

| | Minislump (mm) | Washout (%) | RCS (%) |
|---|---|---|---|
| Residual standard deviation | 3.47 | 0.54 | 2.87 |
| Standard deviation (central points) | 5.0 | 0.29 | 1.25 |
| Predicted error of sum of square (PESS) | 5.3 | 0.8 | 3.8 |

Table 7
Mixture proportions and results of grouts used for evaluating accuracy of linear models

| | W/C | AWA (%) | HRWR (%) | Minislump (mm) | Washout (%) |
|---|---|---|---|---|---|
| Selected points | 0.40 | 0.025 | 1.0 | 130 | 10.6 |
| | | 0.04 | 1.0 | 115 | 6.4 |
| | | 0.04 | 1.25 | 125 | 6.7 |
| | | 0.04 | 1.5 | 130 | 7.2 |
| | | 0.046 | 1.25 | 125 | 8.0 |
| | | 0.05 | 1.0 | 110 | 6.2 |
| | | 0.05 | 1.0 | 110 | 6.0 |
| | | 0.05 | 1.25 | 115 | 6.4 |
| | | 0.05 | 1.5 | 130 | 9.0 |
| | | 0.05 | 1.5 | 125 | 9.0 |
| Central points | 0.40 | 0.04 | 1.25 | 125 | 10.5 |
| | | 0.04 | 1.25 | 130 | 11.0 |
| | | 0.04 | 1.25 | 135 | 11.0 |

The results of measured minislump flow are close to predicted values. On the other hand, the washout model systematically leads to higher prediction than measured values. This can be partially attributed to the dominant influence of W/C on washout. For a given W/C, the importance of AWA–HRWR interaction on rheological properties of neat cement paste was shown experimentally [1]. The unusual result observed in this study may be due to the modeled domain, which is too large to adequately evaluate the effect of the investigated variables, especially in the case of the AWA and HRWR parameters. A decrease in water content increases the efficiency of the HRWR and can eliminate the ability of AWA to reduce washout loss. This result highlights the difficulties of evaluating the interaction among such admixtures with various water contents. The relatively high scatter between predicted and measured values of washout loss can be in part due to the precision of the washout test itself, especially for more viscous mixtures where a relative error on the order of 13% (95% confidence limit) can be obtained [9].

Although the above models yielded high coefficients of correlation, the variability between measured and predicted values is unacceptable, especially in the case of the washout model. This is can be due to the fact that the effect of the investigated variables on modeled responses is not linear. A nonlinear model can, therefore, provide more accurate prediction. Therefore, a quadratic model was attempted to evaluate the effect of AWA–HRWR combination on properties of mixtures with a fixed W/C of 0.40.

### 4.3. Modeling the effect of HRWR–AWA combinations for grout with 0.40 W/C (quadratic models)

A CCD was carried out to model the effect of HRWR–AWA combination on minislump, washout loss, and RCS for grout with 0.40 W/C. This ratio was selected to secure sound mechanical performance. The associated quadratic model for a CCD plan and two independent variables (HRWR and AWA) is expressed as follows:

$$Y = a_0 + a_1\mathrm{HRWR} + a_2\mathrm{AWA} + a_3\mathrm{HRWR{\cdot}HRWR} + a_4\mathrm{AWA{\cdot}AWA} + a_5\mathrm{HRWR{\cdot}AWA} + E \quad (6)$$

For any given response, the presence of interaction with coupled terms (such as AWA·AWA) indicates that the influence of the parameter on that response is quadratic. The initial HRWR and AWA dosages were varied from 0.6% to 1% and 0% to 0.03%, by mass of cement, respectively. Test results of the 13 mixtures presented in Table 2 were fitted to Eq. (6) to determine the unknown constants. The estimates as well as the *t* ratios for each term are summarized in Table 8.

Similarly to the linear models, the derived coefficients were obtained with 90% confidence limits based on Student's test. The significance of each variable on a given response is investigated by using the same statistical criteria of decision adopted with the linear models. The critical value ($t_{0.05,\ 13,\ 7}$), where $n$ is the number of experiments and $k$ is the number of unknown parameters, is equal to 1.89. An independent variable is stated to have a significant effect on a given response when the *t* ratio associated to that variable is greater than 1.89. After the elimination of the nonsignificant (NS) terms, the derived quadratic models for minislump, washout, and RCS are given in Table 9, and the results of the analysis of variance are summarized in Table 10. The quadratic models can be expressed as follows [Eqs. (7)–(9)]:

$$\mathrm{Minislump} = 132 + 19.6\mathrm{HRWR} - 12.2\mathrm{AWA} - 9.8\mathrm{HRWR{\cdot}HRWR} - 3.5\mathrm{AWA{\cdot}AWA} \quad (7)$$

$$\mathrm{Washout} = 12.6 + 3.8\mathrm{HRWR} - 3\mathrm{AWA} + 1.6\mathrm{HRWR{\cdot}AWA} - 1.4\mathrm{HRWR{\cdot}HRWR} - 0.8\mathrm{AWA{\cdot}AWA} \quad (8)$$

$$\mathrm{RCS} = 68.2 + 5.9\mathrm{AWA} - 5.8\mathrm{HRWR} - 3.4\mathrm{HRWR{\cdot}AWA} \quad (9)$$

The significance of the derived models is evaluated by comparing the $F$ value to the $F_{\beta,\ k,\ n-k}$ variable based on Fisher–Snedecor distribution. For a 90% confidence limit, $F_{0.1,\ 4,\ 8}$, $F_{0.1,\ 5,\ 7}$, and $F_{0.1,\ 3,\ 9}$ are 2.81, 2.88, and 2.81, respectively. As can be observed, the $F$ values for the minislump, washout, and RCS models are greater than the respective critical values. Thus, it can be stated that the derived quadratic models are significant in describing the coupled effect of HRWR and AWA on the modeled responses. It is important to note the high significant effect of the second order interaction between AWA and HRWR on washout and residual strength.

Table 8
Estimates and *t* ratio obtained with CCD plan

| | Minislump (mm) | | Washout mass (%) | | RCS (%) | |
|---|---|---|---|---|---|---|
| Term | Estimate | *t* ratio | Estimate | *t* ratio | Estimate | *t* ratio |
| Intercept | 132 | 65.9 | 12.6 | 45.4 | 66.5 | 79.0 |
| HRWR | 19.6 | 12.4 | 3.8 | 17.2 | − 5.8 | − 8.7 |
| AWA | − 12.2 | − 7.7 | − 3.0 | − 13.5 | 5.9 | 8.9 |
| HRWR·HRWR | − 9.8 | − 5.7 | − 1.4 | − 5.8 | 1.4 | 2.0 |
| AWA·AWA | − 3.5 | − 2.1 | − 0.8 | − 3.6 | 1.2 | 1.7 |
| HRWR·AWA | 3.8 | 1.7 | 1.6 | 5.1 | − 3.6 | − 3.8 |

Table 9
Significant estimates and *t* ratio obtained with CCD plan

| | Minislump (mm) ($R^2$=.97) | | Washout mass (%) ($R^2$=.98) | | RCS (%) ($R^2$=.91) | |
|---|---|---|---|---|---|---|
| Term | Estimate | *t* ratio | Estimate | *t* ratio | Estimate | *t* ratio |
| Intercept | 132 | 59.5 | 12.6 | 45.4 | 68.2 | 88.6 |
| HRWR | 19.6 | 11.2 | 3.8 | 17.2 | − 5.8 | − 7.8 |
| AWA | − 12.2 | − 7.0 | − 3.0 | − 13.5 | 5.9 | 7.9 |
| HRWR·HRWR | − 9.8 | − 5.2 | − 1.4 | − 5.8 | NS | – |
| AWA·AWA | − 3.5 | − 1.9 | − 0.8 | − 3.6 | NS | – |
| HRWR·AWA | NS | – | 1.6 | 5.1 | − 3.4 | − 3.4 |

## 4.4. Accuracy of quadratic models

The accuracy of the quadratic models is evaluated by comparing the standard deviation obtained on five central points to the residual standard deviation (Table 11). The quadratic models offer better prediction than the first order linear models. Indeed, by comparing PESS values in Tables 6 and 7, the error obtained on predicted values using quadratic models are shown to be lower than those obtained with the linear models. The PESS values are generally closer to the standard deviation obtained from the central points.

The accuracy of the minislump and washout models are further evaluated using the five central points and six supplementary mixtures (Table 12) to compare predicted-to-measured values. The selected mixtures cover a wide range of the modeled region, again with the W/C being constant at 0.40.

The mean ratio (σ) and COV of predicted-to-measured values obtained for the minislump and washout responses are as follows:

- Minislump model: σ = 1.02 and COV = 2.9%
- Washout model: σ = 1.1 and COV = 15%

Table 10
Analysis of variance of quadratic minislump, washout, and RCS models

| | Minislump ($R^2$ = .97)[a] | | | Washout mass ($R^2$ = .98)[a] | | | RCS ($R^2$ = .91)[a] | | |
|---|---|---|---|---|---|---|---|---|---|
| Term | DF | SS | *F* ratio | DF | SS | *F* ratio | DF | SS | *F* ratio |
| Model | 4 | 4961.1 | 50.5 | 5 | 208.5 | 109.0 | 3 | 601.1 | 38.3 |
| Error | 8 | 196.6 | | 7 | 2.7 | | 9 | 47.1 | |
| Total | 12 | 5157.7 | | 12 | 211.2 | | 12 | 648.2 | |

[a] $R^2$ is calculated as the ratio between $SS_{model}$ and $SS_{total}$.

Table 11
Statistical measures on minislump, washout, and RCS

| | Minislump (mm) | Washout (%) | RCS (%) |
|---|---|---|---|
| Residual standard deviation | 4.1 | 0.5 | 2.0 |
| Standard deviation (central points) | 3.0 | 0.4 | 1.8 |
| Predicted error of sum of square (PESS) | 5.0 | 0.6 | 2.3 |

The degree of prediction of responses was improved with the composite design models compared to the first order models (two-level factorial models). In general, with the composite models, the measured minislump and washout mass loss results lie close to predicted values, regardless of the fluidity of the grout. The deviations between predicted and measured values are mostly within the standard errors measured on the modeled responses and discussed elsewhere [9]. The new established models can then predict minislump spread and washout loss within 5 mm and 1.5%, respectively, with confidence limit of 90%.

### 4.5. Trade-off between HRWR and AWA dosages

Contour response diagrams showing the influence of HRWR and AWA contents on minislump and washout loss are presented in Fig. 1. These values are established using the quadratic models. Contour diagrams of washout loss and RCS are also plotted in Fig. 2. For a given HRWR dosage, it can be seen that as the dosage of AWA increases, the minislump and washout values decrease. The minislump can then be reestablished by increasing the dosage of HRWR. For example, for a minislump consistency of 130 mm, a mixture with 0.7% HRWR and no AWA can yield such a consistency. The increase in AWA dosage to 0.02% would result in a drop of minislump to 115 mm. However, by increasing the HRWR content from 0.7% to 0.85%, the minislump of 130 mm can be reestablished.

When dealing with high fluidity grout (minislump greater than 130 mm), the HRWR demand can be significantly high to compensate for losses of fluidity associated with increasing AWA content. For example, the HRWR dosage must be increased from 0.8% to 1.0% to maintain a minislump of 140 mm when the AWA increases from 0% to 0.018%. At high fluidity the increase in HRWR is shown to cause no significant increase in minislump, even at low AWA dosage. This can be due to the high dosage of HRWR that approaches the saturation point of the admixture.

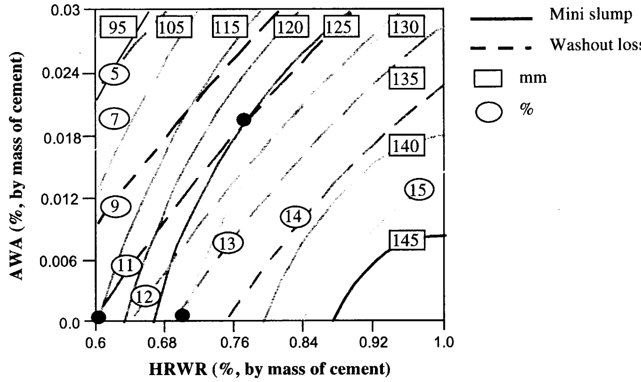


Fig. 1. Contours diagram of minislump and washout loss values.

The washout resistance is directly affected by the level of consistency of the grout. The increase in minislump due to high additions of HRWR leads to increase in washout loss, accompanied by a decrease in RCS, regardless of the AWA content. For a given minislump value, a lower washout loss was obtained when using high AWA content with higher resulting residual strength. However, in such case the HRWR demand necessary to maintain the fluidity becomes greater than that in grout with low AWA dosage. For example, for a minislump spread of 115 mm, the washout mass loss can be on the order of 11% for grout mixture made with 0.6% HRWR and no AWA. The resulting RCS of such mixture can be 66%. The increase of AWA dosage to 0.02% can provide a washout loss of 9% and an RCS of 72%. The higher AWA necessitates an increase in HRWR dosage from 0.6% to 0.7% to maintain a minislump consistency of 115 mm.

As shown in Figs. 1 and 2, a highly fluid neat cement grout with minislump greater than 130 mm had a relatively high washout loss of 13%. Such fluidity and washout can be

Table 12
Mixture proportions and results of mixtures used to establish accuracy of quadratic models

| | AWA (%) | HRWR (%) | Minislump (mm) | Washout (%) |
|---|---|---|---|---|
| Selected points | 0.0 | 0.8 | 140 | 13.8 |
| | 0.02 | 0.8 | 125 | 10.0 |
| | 0.025 | 0.8 | 120 | 10.2 |
| | 0.025 | 1.0 | 130 | 10.6 |
| | 0.03 | 1.0 | 120 | 8.6 |
| Central points | 0.015 | 0.8 | 130 | 12.5 |
| | 0.015 | 0.8 | 130 | 12.7 |
| | 0.015 | 0.8 | 135 | 13.0 |
| | 0.015 | 0.8 | 135 | 12.6 |
| | 0.015 | 0.8 | 130 | 12.0 |

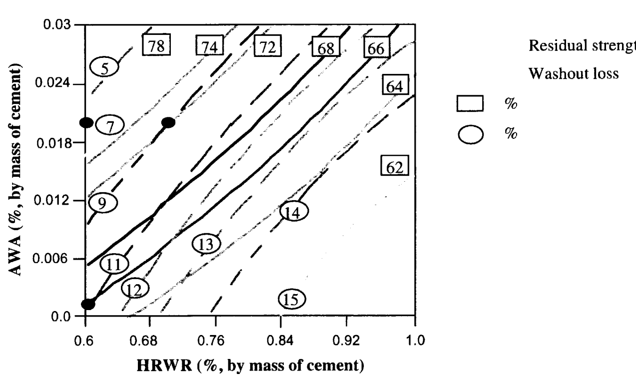


Fig. 2. Contours diagram of washout loss and RCS.

obtained in the case of grout with HRWR and AWA corresponding to 0.70% and 0%, respectively. With the increase in HRWR and AWA to 0.78% and 0.02%, respectively, a slightly lower minislump of 125 mm and a lower washout of 11% can be obtained. The reduction in washout loss due to greater concentration of AWA or lower HRWR content enhances the RCS. For a washout of 11% obtained with grouts incorporating 0.001% AWA and low HRWR content of 0.6%, an RCS of 66% can be obtained. Increasing the AWA dosage to 0.02% and HRWR to 0.7% can maintain the fluidity and reduce the washout loss to 9% and increase the RCS to 72%.

## 5. Discussion and use of derived models

Statistical design of experiments can be a useful tool to highlight the influence of key parameters affecting fluidity, stability, and RCS of cement grout intended for underwater applications. The above results show that accurate modeling of the effect of W/C, AWA, and HRWR on fluidity, washout loss, and RCS depends on the efficiency of selecting the modeled domain, the range of each parameter, and the number of variables modeled. The use of a wide domain may not be appropriate to adequately estimate the effect of HRWR and AWA parameters on the modeled responses. For example, when dealing with a wide range of W/C values, it is shown that the HRWR can result in an insignificant effect on consistency with limited benefit of using AWA to reduce washout.

The first-order models established using the two-level statistical design showed that the W/C has the greatest effect on fluidity, washout loss, and RCS. These first order models lead to relatively poor prediction of the modeled responses, especially in the case of washout loss. This is due to the dominant effect of W/C on the modeled responses and the absence of a significant interaction between AWA and HRWR in the linear model. More accurate estimates of the responses can be obtained by using a CCD carried out with mixtures of a fixed W/C of 0.40. Using a CCD design, the quadratic terms are taken into account for the curvature of the response surface, which is present when a response is near an optimum value in the experimental domain. By comparing the mean ratio of predicted-to-measured values for residual strengths obtained with central points, it can be stated that both the first-order and quadratic models lead to the same degree of accuracy.

The above models can be used to increase the efficiency in selecting the optimum mix proportions for high-performance grout. Trade-offs between the effects of AWA and HRWR and W/C on performance can be established through a simple analytical simulation. Given a W/C, cost, and washout resistance, a minimum level of residual strength can be identified. An increase in residual strength requires lower washout loss possible with an increase in AWA along with HRWR to maintain a given consistency. Another approach to achieve the required residual strength is to reduce the W/C and increase the HRWR content. The use of such models can then enable the comparison of the cost of different optimized grout mixtures.

With changes in materials properties, such as cement source, the models can still be used to predict fluidity, washout, and residual strength; the scattering between the predicted and measured values will then indicate the effect of the new cement on the accuracy of the models. In the case of unacceptable variability, limited testing protocol can be undertaken to adjust mixture parameters to secure the required properties since the relative influence of each parameter on key responses should not significantly change with changes in material properties.

## 6. Conclusions

Based on the results presented in this paper, the following conclusions can be warranted:

1. The interaction between AWA and HRWR can be adequately evaluated using quadratic models derived from a CCD run on a set of grout mixtures with a constant W/C of 0.40.
2. Accurate prediction of the combined effects of AWA and HRWR on minislump consistency, washout resistance, and RCS of grouts with 0.40 W/C can be ensured with the second order quadratic models. First-order linear models can provide adequate prediction of RCS of underwater-cast grout, regardless of the W/C.
3. The minislump consistency, washout loss, and residual strength of cement-based grouts are dominated primarily by the W/C.
4. The washout resistance improves as AWA content increases for a given W/C despite the greater dosage of HRWR necessary to maintain fluidity.
5. For a given W/C of 0.40, an increase in minislump due to a greater HRWR dosage can increase the washout loss and reduce residual strength, regardless of the AWA dosage.

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