Pergamon

Cement and Concrete Research 31 (2001) 1553–1560

CEMENT AND CONCRETE RESEARCH

Factorial design modelling of mix proportion parameters of underwater composite cement grouts

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Received 31 January 2001; accepted 14 June 2001

Abstract

There is an increasing need to identify the rheological properties of cement grout using a simple test to determine the fluidity, and other properties of underwater applications such as washout resistance and compressive strength. This paper reviews statistical models developed using a factorial design that was carried out to model the influence of key parameters on properties affecting the performance of underwater cement grout. Such responses of fluidity included minislump and flow time measured by Marsh cone, washout resistance, unit weight, and compressive strength. The models are valid for mixes with 0.35–0.55 water-to-binder ratio (W/B), 0.053–0.141% of antiwashout admixture (AWA), by mass of water, and 0.4–1.8% (dry extract) of superplasticizer (SP), by mass of binder. Two types of underwater grout were tested: the first one made with cement and the second one made with 20% of pulverised fuel ash (PFA) replacement, by mass of binder. Also presented are the derived models that enable the identification of underlying primary factors and their interactions that influence the modelled responses of underwater cement grout. Such parameters can be useful to reduce the test protocol needed for proportioning of underwater cement grout. This paper attempts also to demonstrate the usefulness of the models to better understand tradeoffs between parameters and compare the responses obtained from the various test methods that are highlighted.

Keywords: Antiwashout admixture; Flow time; Minislump; PFA; SP; Underwater grout; Water/binder; Washout resistance

1. Introduction

Recently, there has been a growing interest in the use of high-performance cement grout containing antiwashout admixture (AWA) for repairs, injection, embedding of anchors, and post-tensioning underwater. A cement-based grout should be stable enough to reduce sedimentation, bleeding, and water dilution. Cement-based grouts are widely used in injection grouting of cracks in massive structures since their physical and mechanical properties can be easily controlled. This is assured by judicious choice of the type and fineness of cement, water-to-binder ratio (W/B), chemical, and mineral admixtures [1]. The incorporation of one or more types of supplementary cement replacement materials (CRMs) having different morphology and grain-size distribution values can improve particle size distribution and packing of solid particles, hence enhancing fluidity, stability, and permeability. Improvements in the quality and uniformity of CRMs and the attention given to admixture formulations have greatly assisted progress.

Viscosity-modifying admixtures (VMAs), also known as AWAs, are relatively new admixtures used to enhance the cohesion and stability of cement-based systems. Such AWAs are water-soluble polysaccharides that enhance the water retention capacity of paste [2,3]. Welan gums are long-chain polymers and have high molecular weight (about 2 million grams) which is produced from the fermentation of a specific microorganism under a controlled environment [4]. They are used in concrete intended for underwater repair of marine and hydraulic structures, and tremie concrete for the construction of curtain walls, as well as in cement grout for underwater crack injection of damaged concrete bridge piers and footings, and for applications underwater such as post-tensioning and embedding anchors [1,5]. The use of AWA increases the yield value and plastic viscosity of the cement-based grout, thus necessitating an

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0008-8846/01/$ – see front matter
PII: S0008-8846(01)00583-X

increase in water/binder or superplasticizer (SP) dosage to insure a low yield value necessary for proper penetrability and spreading. Increasing the W/B results in decrease in mechanical properties, impermeability, and durability [6,7].

The use of pulverised fuel ash (PFA) in cementitious grout represents a relatively mature technology, which has been utilised in a range of construction applications. Experience has shown that compared to neat Portland cement grout, PFA replacement incorporation can effect improvements in rheology and provide the full range of engineering properties normally required. Applications such as soil, rock, and oil-well grouting all require enormous amounts of cement and are therefore good examples of areas where some part of cement could be partially replaced by PFA to produce low-cost, environmentally safe, and durable grouts [8].

Several researchers have related the improvement in properties of underwater cement-based grout to the enhancement in washout resistance [1,5–7]. For example, AWA cement grout with a minislump of 13 cm was successfully used for embedding anchors underwater with 0.40 of W/B. The grout, incorporating 8% silica fume replacement and containing 0.03% of welan gum (by mass of binder), had a washout mass loss 5% following the method proposed Khayat and Yahia [6]. The mean of compressive strength at 21 days for grout cast above water and in water were 62 and 46 MPa, respectively. High-performance cement grout made with a microfine cement with 0.6 W/B and 2% of SP was used successfully for crack injection grouting of submerged structures [1]. The underwater grout was highly stable (washout mass loss = 9.7%), yet it exhibited low viscosities at high and low shear rates. The grout is shown to exhibit good balance between rheological and mechanical properties [1].

The main objective of this study is to evaluate the effect of W/B, the concentrations of SP and AWA on the rheological properties, and compressive strength at 7 days of underwater grouts using statistical design approach and analysis of experiments [9,10]. Two grouts were used, one made with 100% cement and the second one made by incorporation of 20% PFA as a replacement of cement. The established models can identify parameters and the two-way interactions that have significant effect on the rheological properties of underwater grouts. The models can be used to evaluate the potential influence of adjusting mix variables on grout properties required to ensure successful development of underwater grout. Such simulation can facilitate the test protocol needed to optimise underwater grout with a given set of performance criteria that can be tried in the laboratory.

2. Development of factorial design approach

A 2^3 statistical experimental design was used to evaluate the influence of two different levels for each variable on the relevant grout properties. Three key parameters that can have significant influence on mix characteristics of underwater cement grout were selected to derive mathematical models for evaluating relevant properties. The three variables were W/B, concentrations of SP, and AWA (Fig. 1). The modelled experimental region consisted of mixes ranging between coded variables of -1.414 and $+1.414$ (Table 1). The derived statistical models are valid for mixes made with 100% cement with ranges of W/B of 0.35–0.55, concentrations of AWA of 0.053–0.141%, by mass of water (or 0.02–0.08% of mass of binder), and SP of 0.4–1.6% (dry extract), by mass of binder. In the case of PFA grout, the same ranges of W/B and AWA were used, but the SP concentration was varied from 0.6% to 1.8%, by mass of binder. The grout responses that were modelled were the minislump, the flow time of Marsh cone at 900 ml, the washout mass loss, unit weight, and 7-day compressive strength. The 0.9-l flow was selected as a basis for comparison because of the nonlinear nature of the flow that appears beyond 0.9 l, due to an increased effect of friction, and also to the thixotropic character of the cement pastes.

Such a two-level factorial design requires a minimum number of tests for each variable. Given the fact that the expected responses do not vary in linear manner with the selected variable and to enable the quantification of the prediction of the responses, a central composite plan was selected where the response could be modelled in a quadratic manner. Since the error in predicting the responses increases with the distance from the centre of the modelled region, it is advisable to limit the use of the models to an area bound by coded values corresponding to $-\alpha$ to $+\alpha$ limits. Parameters were carefully selected to carry out composite factorial design where the effect of each factor is evaluated at five different levels in codified values of $-\alpha$, -1, 0, $+1$, and $+\alpha$. The α value is chosen so that the variance of the response predicted by the model would depend only on the distance from the centre of the modelled region.

The 40 mix combinations, expressed in coded values, considered in the experimental design for cement and PFA grouts are listed in Table 1. Six replicate central points were

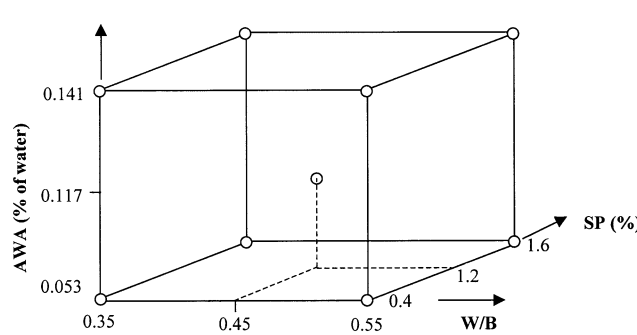

Fig. 1. Structure of the three-variable factorial plan of experiment.

Table 1
Coded and absolute values for the investigated parameters

Parameter	Grout	− 1.414	− 1	Central point	+ 1	+ 1.414
W/B	Cement	0.31	0.35	0.45	0.55	0.59
	PFA	0.31	0.35	0.45	0.55	0.59
AWA (percentage of water)	Cement	0.026	0.053	0.117	0.141	0.156
	PFA	0.026	0.053	0.117	0.141	0.156
SP (%)	Cement	0.152	0.4	1.0	1.6	1.848
	PFA	0.352	0.6	1.2	1.8	2.048

prepared to estimate the degree of experimental error for the modelled responses. The coded units of variables are calculated as follows:

$$\text{Coded W/B} = (\text{absolute W/B} - 0.45)/0.1$$

$$\text{Coded AWA} = (\text{absolute AWA} - 0.117)/0.044$$

$$\text{For cement grout: Coded SP} = (\text{absolute SP} - 1)/0.6$$

$$\text{For PFA grout: Coded SP} = (\text{absolute SP} - 1.2)/0.6$$

3. Materials and mix proportions

The grout used to establish statistical models was systematically proportioned using an ordinary Portland cement with a Blaine fineness of 385 m^2/kg. The incorporated mineral admixtures included PFA (BS 3892: Part 1) with 20% cement replacement. The chemical and physical properties of cement and PFA are given in Table 2.

The AWA was the Kelco-Crete welan gum that is a high-molecular weight, microbial polysaccharide. The gum was supplied in a powder gum. A new-generation vinyl copolymer-based formaldehyde-free SP was used.

Table 2
Chemical and physical properties of cement and PFA

	Cement	PFA	Cement	
SiO_2	20.8	49.5	Vicat set time (min)	
Al_2O_3	5.0	25.9	Initial	119
Fe_2O_3	3.2	8.8	Final	164
CaO	63.7	1.6		
MgO	2.6	1.5		
Na_2O eq.	0.39	3.3		
Free CaO	1.6	–		
LOI	0.65	3.5		
Relative density	3.12	2.15		
Specific surface area (m^2/kg)	385	–		
Percentage passing the 45-μm sieve	–	87.3		

Standard compressive strength (MPa)

Age (day)	Cement
7	41.5
28	57.8

4. Testing procedures

All grout mixes were prepared in batches of 1.5 l that were mixed using a high-shear mixer. The mixing water was 13 ± 2°C to compensate for the heat generated during the mixing action. The grout temperature following the end of initial mixing was maintained at 23 ± 3°C. The mixing sequence consisted of adding all of the water and SP to the mixer along with the AWA. The cementitious materials were then introduced gradually after 1 min. The grout was mixed for 1 min, followed by 30 s of rest and another mixing period of 1 min.

Following the end of mixing, the temperature and specific gravity of the grout were measured, and the minislump, flow time, and the washout mass loss values were determined. All tests were carried out between 5 and 15 min following the initial contact of cement with water and were done always in the same sequence. Three 50-mm cubes were cast in laboratory environment (T = 20 ± 2°C) to determine the compressive strength at 7 days.

The fluidity was evaluated using minislump cone and Marsh cone having a 5-mm outlet diameter. The dimensions of minislump are presented in Fig. 2 [11]. As shown in Fig. 2, the minislump cone has an upper diameter of 19 mm, a lower diameter of 38.1 mm, and height of 57.2 mm. The test procedure involves the measurement of the spread diameter of a certain volume of grout (38 ml) placed in the cone. The cone is positioned at the centre of the horizontal Perspex base plate. After pouring the grout into the cone without causing it to overflow, the upper part of the cone is tamped lightly to bleed off any entrapped air pockets, and the cone is then gently lifted. The spread diameter of a given mix represents the mean of two diameters recorded at the end of the flow.

The Marsh cone test measures the flow time of a given volume of a grout through a cone of a standard size. The funnel Marsh cone used in this study has a capacity of 1200 ml and an internal orifice diameter of 5 mm (Fig. 2). The time needed for a grout sample to flow through the cone is proportional to the viscosity of the grout. The flow time increases with the increase in viscosity, and thereby it becomes an index of fluidity. The flow time measurement is performed by taking a representative sample of 1100 ml of

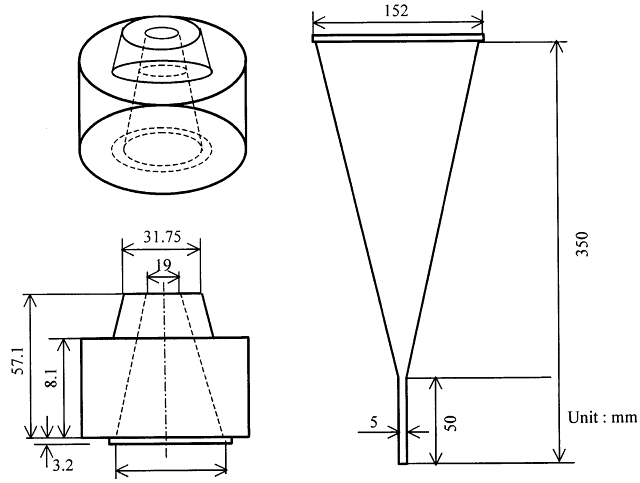

Fig. 2. Minislump and Marsh cone tests.

grout, plugging the lower orifice of the cone, then pouring the sample through a fine sieve that can retain any clumps of cement particles. Once the grout is allowed to flow out of the funnel and into a graduated cylinder, the time of flow is recorded at set intervals of 100 ml up to 1000 ml.

The washout resistance of the grouts was evaluated by measuring the washout mass loss using a 500-ml grout sample that was poured through a funnel and into a similar quantity of water from a predetermined height [6]. The bottom of the funnel is positioned at 10 mm above the water level of the lower beaker. As the grout falls freely into the beaker, it becomes partially diluted by the water and partially washes away with the displaced water. The mass loss was calculated as the difference between the mass of the original 500-ml sample and the sample dropped through water. This difference in mass is due to the loss of cementitious materials diluted in water and displaced with it.

5. Results and discussion

5.1. Derived statistical models

The derived statistical models for 100% C and PFA grouts are summarised in Table 3 with correlation coefficients and probability (Prob.) > $|t|$ values. The estimates for each parameter refer to the coefficients of the model found by a least-square approach. The Prob. > $|t|$ term is the probability of getting an even greater t statistic, in absolute value, that tests whether the true parameter is zero. Probabilities less than .05 are often considered as significant evidence that the parameter is not zero, i.e., that the contribution of the proposed parameter has a highly significant influence on the measured response.

The presentation in Table 3 enables the comparison of various parameters as well as the interactions of the modelled responses. For the majority of the parameters, the probabilities that the derived coefficients of the various parameters influence each response are limited to 5%. This signifies that there is less than 5% chance, or 95% confidence limit, that the contribution of a given parameter to the tested response exceeds the value of the specified coefficient. A negative estimate signifies that an increase of the given parameter results in a reduction of the measured response. For example, an increase in W/B increases the minislump and reduces the flow time, i.e., increases the flowability. For any given measured response, the presence of interaction with coupled terms indicates that the influence of the parameter on that response is quadratic. The models in Table 3 give an indication of the relative significance of various parameters on each response. The majority of the models had high correlation coefficients (R^2).

For example, the derived statistical models for minislump and washout mass loss of cement and PFA

Table 3
Parameter estimates of the five derived models

Cement grout (100% C)

	Minislump (R^2 = .92)		Flow time (R^2 = .96)		Washout (R^2 = .88)		Unit weight (R^2 = .96)		7-Day f_c' (R^2 = .85)	
	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$
Intercept	9.69		35.64		8.11		1910.6		38.41	
W/B	2.85	0	– 27.22	0	4.52	0	– 119.9	0	– 19.15	0
SP	1.20	.0142	– 24.84	0	2.23	.0035	–	–	–	–
AWA	– 1.90	.0005	–	–	– 1.83	.0125	–	–	–	–
AWA·W/B	–	–	21.73	.0008	–	–	–	–	–	–

PFA grout (20% FA + 80% C)

	Minislump (R^2 = .92)		Flow time (R^2 = .96)		Washout (R^2 = .89)		Unit weight (R^2 = .98)		7-Day f_c' (R^2 = .85)	
	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$
Intercept	11.90		21.94		10.04		1862.9		17.06	
W/B	2.21	0	– 17.58	0	3.71	0	– 113.42	0	– 8.50	0
SP	1.0	.0241	–	–	1.66	.0065	–	–	–	–
AWA	– 2.06	.001	11.79	.002	– 1.83	.0033	–	–	–	–
AWA·W/B		–	– 16.76	.0006		–	–	–	–	–

Table 4
Repeatability of test results

	Minislump (cm)	Flow time (s)	Washout mass loss (%)	Unit weight (kg/m^3)	7-Day f_c' (MPa)
Cement grout					
Mean (n=6)	9.64	9.76	9.62	1896.8	40.7
V (%)	2.60	3.34	6.39	0.23	11.3
Relative error, 95% confidence limit (%)	2.1	2.8	5.3	0.2	9.3
PFA grout					
Mean (n=6)	12.02	10.06	12.97	1840.3	18.0
V (%)	3.54	3.45	3.19	0.49	11.3
Relative error, 95% confidence limit (%)	2.9	2.8	2.6	0.4	9.3

V: Coefficient of variation (%)

grouts are given in Eqs. (1) and (2) and Eqs. (3) and Eqs. (4), respectively.

Cement grout:

$$\text{Minislump (cm)} = 9.69 + 2.85\ \text{W/B} - 1.9\ \text{AWA} + 1.2\ \text{SP} \tag{1}$$

$$\text{Washout loss (\%)} = 8.11 + 4.52\ \text{W/B} + 2.23\ \text{SP} - 1.83\ \text{AWA} \tag{2}$$

PFA grout:

$$\text{Minislump (cm)} = 11.9 + 2.21\ \text{W/B} - 2.06\ \text{AWA} + \text{SP} \tag{3}$$

$$\text{Washout loss (\%)} = 10.04 + 3.71\ \text{W/B} - 1.83\ \text{AWA} + 1.66\ \text{SP} \tag{4}$$

Table 4 indicates the average measured responses of the six replicate grouts made with cement and PFA, coefficients of variation (V), as well as the relative errors with 95% confidence limit for each of the measured properties. The estimated errors of cement grout for the minislump, washout mass loss, unit weight, and 7-day f_c' were ± 0.3 cm, ± 0.6%, ± 4 kg/m^3, and ± 5 MPa, respectively. The values for PFA grout were 0.4 mm, 0.4%, 9 kg/m^3, and 2 MPa, respectively. The relative experimental errors for minislump, flow time, and washout are shown to be limited to 2–5%. On the other hand, the relative error for the 7-day f_c' was approximately 9% for the two grout mixes made with cement and PFA.

5.2. Accuracy of the proposed models

The accuracy of each of the proposed models was determined by comparing predicted-to-measured values obtained with mixes prepared at the centre of the experimental domain. The predicted-to-measured ratio for minislump, washout mass loss, unit weight, and compressive strength at 7 days are summarised in Table 5.

The ratio between predicted and various measured properties of grout ranges between 0.77 and 1.01, thus indicating good accuracy for the established models to predict the minislump, washout, and compressive strength. In general, the proposed models for minislump, washout, and the compressive strength appear to be satisfactory in predicting the fluidity and strength with low scattering between the measured and predicted values.

The proposed statistical models can therefore be used to evaluate the effect of a group of variables on the properties affecting the quality of cement grout. This permitted the calculation of the isoresponse curves from the parameters under study over the experimental domain and the optimisation of their effects. For example, the effect of increasing the W/B vs. the dosage of SP on minislump and washout mass loss is shown in Figs. 3 and 4, respectively, for grout cement with medium AWA concentration of 0.117% and without AWA. The minislump and washout mass loss increased significantly when W/B increased, and tended to increase with increased dosages of SP (Figs. 3 and 4). For any W/B and SP dosage, the cement grout made with AWA concentration of 0.117% had clearly greater washout resistance than the non-AWA mix.

5.3. Tradeoff between W/B ratio, SP dosage, and fly ash incorporation

When in the absence of PFA or in its presence, the minislump for all the cement grouts decreased with decreased W/B (Fig. 5). As shown in Fig. 5, for a given W/B and AWA concentration, a greater fluidity can be secured when 20% of cement was replaced with PFA. For example, for W/B of 0.50 and SP of 0.60%, the replacement of 20% of cement by PFA

Table 5
Predicted-to-measured ratios of established models

	Predicted/measured				
	Grout	Minislump	Washout	Unit weight	7-Day f_c'
Central points	Cement	1.00	0.84	1.01	0.94
Central points	PFA	0.99	0.77	1.01	0.95

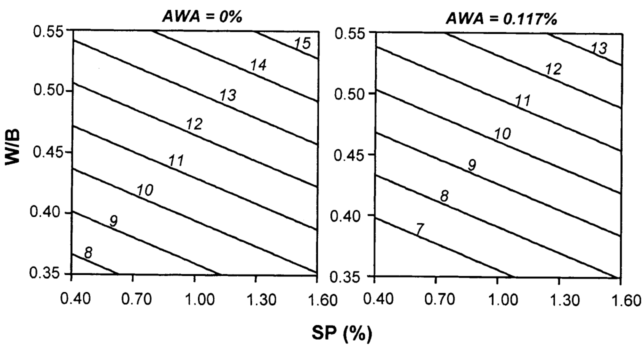

Fig. 3. Isoresponse curves for minislump of cement grout with AWA = 0% and 0.117% (percentage of water).

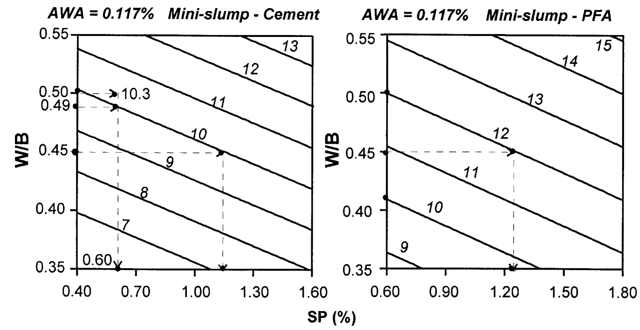

Fig. 5. Comparison of isoresponse curves for minislump of grouts made with cement and PFA (AWA = 0.117% of water).

resulted in higher minislump value, which increased from 10.3 to 12 cm. For cement grout made with 0.50 of W/B, the dosage of SP can be expected to increase from 0.40% to 1.15% when reducing the W/B from 0.50 to 0.45 to maintain an approximate minislump of 10 cm. The minislump of 10 cm with PFA grout can be made with combination of W/B of 0.41 and SP of 0.60%. On the other hand, for PFA grout, the SP dosage can increase from 0.60% to 1.25% to maintain minislump of 12 cm as the W/B is reduced from 0.50 to 0.45. It can be seen that many combinations of W/B and SP can be used to ensure a given minislump.

5.4. Effect of AWA and SP concentrations on fluidity and washout resistance

The derived models can be employed to generate contour diagrams showing the influence of various parameters on key properties affecting the quality of underwater grout. The influence of the concentrations of AWA and SP on minislump and washout mass loss for cement grout made with fixed W/B of 0.45 and 0.55 are plotted in Fig. 6. As expected, the contour diagrams of Fig. 6 indicate that the increase in SP dosage (for a given AWA and W/B) increases minislump along with washout mass loss. For a given SP dosage, the increase in AWA concentration reduces the minislump and washout mass loss. The increase in W/B from 0.45 to 0.55 resulted in an increase in minislump and washout mass loss for any given SP and AWA dosages. For example, as can be seen in Fig. 6, cement grout made with 0.45 of W/B and 1.03% of SP can exhibit a drop in washout mass loss from 10% to 7% when the AWA dosage is increased from 0.053% to 0.133%. Such reduction can be 14.6–11.6% for cement grout made with 0.55 of W/B and similar SP and AWA concentrations.

As the W/B decreases and the dosage of AWA increases, both fluidity and washout mass loss decrease. The prediction models show that the reduction in W/B has a greater influence on reducing washout mass loss and minislump than the increase in AWA concentration. Relationships between predicted minislump and washout mass loss are plotted in Fig. 7 for cement grouts containing various concentrations of AWA and W/B. The minimum and maximum of SP dosages that were used for each AWA and W/B combination are given in Fig. 7. With the increase in SP dosage, both minislump and washout mass loss values increase. For non-AWA cement grout with 0.50 W/B, the predicted minislump of mix made without any SP is 12.2 cm. The reduction of W/B from 0.50 to 0.40 of the non-AWA grout is shown to cause a substantial decrease in washout mass loss, which required a greater SP dosage to maintain a minislump of 12.2 cm.

The incorporation of a relatively low dosage of AWA corresponding to 0.141% of water (0.08% of binder) in

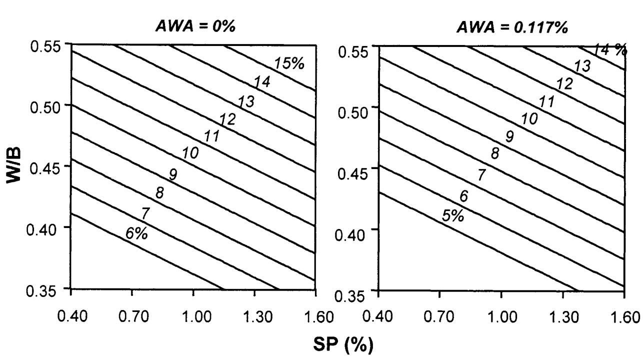

Fig. 4. Isoresponse curves for washout mass loss of cement grout with AWA = 0% and 0.117% (percentage of water).

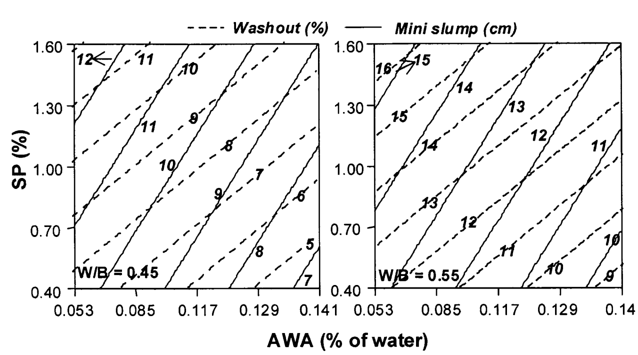

Fig. 6. Isoresponse curves for minislump and washout mass loss — cement grout (W/B = 0.45 and 0.55).

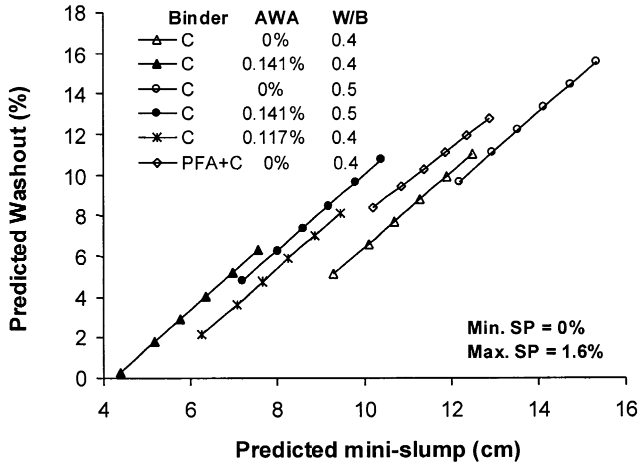

Fig. 7. Effect of AWA dosage and W/B on minislump and washout mass loss.

grout made with 0.50 W/B is shown to be less effective in reducing washout mass loss for a given fluidity than decreasing W/B from 0.50 to 0.40 in a non-AWA cement grout. The use of AWA necessitates an increase in dosage of SP to maintain the fluidity. For example, the required SP dosages to secure a minislump of 10.1 cm (surface = 102 cm^2) for a non-AWA cement grout made with 0.40 of W/B and another one incorporating 0.141% and with 0.50 W/B are shown to be 0.4% and 1.4%, respectively, with washout mass losses of 6.5% and 10%, respectively.

5.5. Relationship between various fluidity and washout mass losses

Correlations between the results of the test methods used to evaluate fluidity of the grout and their washout mass losses are plotted in Figs. 8 and 9. The coefficient of correlation R^2 between the minislump and washout mass loss, and the flow time are .85 and .82, respectively. These good relationships can facilitate the prediction of washout mass loss of underwater cement grout. These simple test methods of minislump and flow time can be used to predict

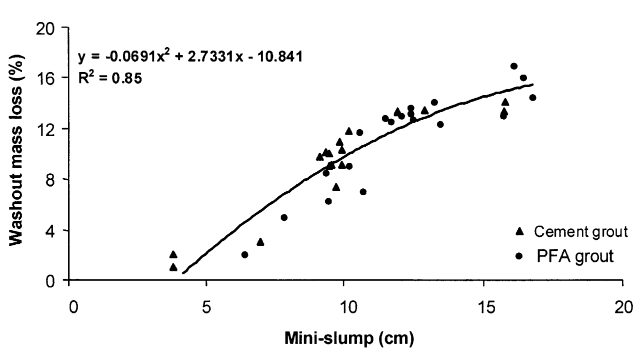

Fig. 8. Relation between washout mass loss and minislump for all mixes.

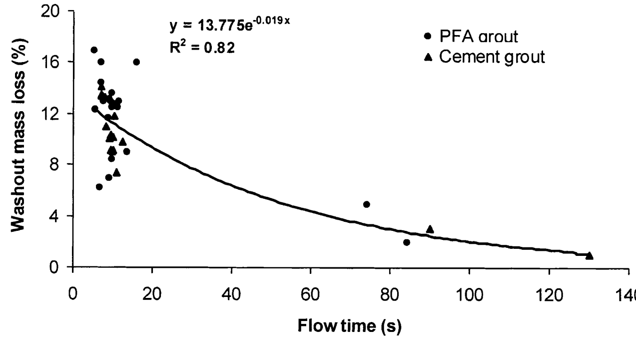

Fig. 9. Relation between washout mass loss and flow time for all mixes.

the washout resistance for quality control in the field or for mix optimisation.

6. Conclusions

The effects of W/B, the concentrations of SP, and AWA on cement and PFA grouts on the rheological properties and compressive strength at 7 days were investigated. The PFA grout was made with 20% of PFA as replacement of cement. The proposed statistical models can simplify the test protocol required to optimise a given mix underwater grout by reducing the number of trial batches needed to achieve a balance among mix variables. This is due to the use of the models in conducting simulations of various variables to secure adequate fluidity, washout resistance, and compressive strength. The models established using a factorial design approach are valid for a wide range of mix proportioning and provide an efficient means to determine the influence of key variables on grout properties. The derived models indicate that the measured properties of underwater grout are highly influenced by W/B, AWA, and SP concentrations and the replacement of cement by PFA, and several coupled effects of these parameters. These models can also provide relationships between the foregoing results for mix optimisation and quality control. The modelling and prediction of the response of other points in the experimental domain were therefore possible. Although the modellings are based on a given set of materials, they can be easily used to generate other future results using other materials.

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