ELSEVIER

Cement & Concrete Composites 25 (2003) 737–749

Cement & Concrete Composites

www.elsevier.com/locate/cemconcomp

Influence of mix proportions on rheology of cement grouts containing limestone powder

Lucie Svermova, Mohammed Sonebi *, Peter J.M. Bartos

Department of Civil Engineering, Advanced Concrete and Masonry Centre, University of Paisley, Paisley PA1 2BE, UK

Received 27 May 2002; accepted 30 September 2002

Abstract

In this paper the parameters of cement grout affecting rheological behaviour and compressive strength are investigated. Factorial experimental design was adopted in this investigation to assess the combined effects of the following factors on fluidity, rheological properties, induced bleeding and compressive strength: water/binder ratio (W/B), dosage of superplasticiser (SP), dosage of viscosity agent (VA), and proportion of limestone powder as replacement of cement (LSP). Mini-slump test, Marsh cone, Lombardi plate cohesion meter, induced bleeding test, coaxial rotating cylinder viscometer were used to evaluate the rheology of the cement grout and the compressive strengths at 7 and 28 days were measured. A two-level fractional factorial statistical model was used to model the influence of key parameters on properties affecting the fluidity, the rheology and compressive strength. The models are valid for mixes with 0.35–0.42 W/B, 0.3–1.2% SP, 0.02–0.7% VA (percentage of binder) and 12–45% LSP as replacement of cement. The influences of W/B, SP, VA and LSP were characterised and analysed using polynomial regression which can identify the primary factors and their interactions on the measured properties. Mathematical polynomials were developed for mini-slump, plate cohesion meter, inducing bleeding, yield value, plastic viscosity and compressive strength as function of W/B, SP, VA and proportion of LSP. The statistical approach used highlighted the limestone powder effect and the dosage of SP and VA on the various rheological characteristics of cement grout.

Keywords: Compressive strength; Grout; Induced bleeding; Limestone powder; Mini-slump; Rheology; Superplasticizer; Viscosity agent; Water/binder

1. Introduction

Cement-based grouts are widely used in injection grouting of cracks in massive structures since their physical and mechanical properties can be easily controlled. The rheological behaviour of special cement grouts intended for the underwater sealing of cracks in dams, offshore structures, massive foundations, or fissures in rock can be enhanced by the incorporation of viscosity agent (VA) [1,2]. Grouts containing VA are also used for filling post-tensioning ducts, where it is important to ensure high resistance to sedimentation and bleeding, hence ensuring corrosion protection of stressed tendons [3]. Admixtures mainly affect the flow behaviour of the cement paste without altering the composition. Therefore, it seems reasonable to try to study the effect of water/binder and admixtures, chemical and mineral, by only testing the cement paste. The rheological measurements on cement paste were used to assess the fresh properties. Viscosity agents are also used in grouts for the repair of deteriorated structures by injection.

Viscosity agents are relatively new admixtures used to enhance the cohesion and stability of cement-based systems [4–10]. Such VAs are water-soluble polysaccharides that enhance the water retention capacity of paste [4,5,7–10]. The use of viscosity agent increases the yield value and plastic viscosity of cement-based grout, thus necessitating increase in water/binder or superplasticiser dosage to insure a low yield stress necessary for proper penetrability, spreading and control sedimentation [4,10]. Several researchers have related the improvement in rheological properties and the performance of cement-based grout to the addition of superplasticiser (SP) and VA [4–10]. For example, for underwater cement-based grout, mini-slump, washout resistance and residual

* Corresponding author. Tel.: +44-141-849-4216; fax: +44-141-848-3275.

E-mail address: mohammed.sonebi@paisley.ac.uk (M. Sonebi).

0958-9465/$ - see front matter © 2002 Elsevier Ltd. All rights reserved.
doi:10.1016/S0958-9465(02)00115-4

compressive strength are highly influenced by water/binder ratio (W/B), VA and SP dosages [7–9]. The washout resistance improves as VA content increases for a given W/B despite the greater dosage of SP necessary to maintain fluidity [7–9]. For a given W/B of 0.40, an increase in mini-slump due to a greater dosage of SP can increase the washout mass loss and reduce residual strength, regardless of the dosage of VA [9]. An optimisation is therefore necessary to establish a balance between the dosages of VA and SP, the W/B ratio and the proportion of mineral admixture (limestone powder, fly ash, silica fume, etc.) to ensure suitable flow and penetration and reduce the bleeding. The mix optimisation of grout often necessitates several trial batches to achieve a balance between the mineral and chemical admixtures and W/B to ensure suitable fluidity, stability and mechanical properties, some of which parameters have opposite effects. For this reason the statistical modelling approach was used in order to reduce the number of trial batches. Nehdi and co-workers [11,12] reported that the limestone microfiller replacement of cement slightly increased the yield value of cement paste and decreased its plastic viscosity, which implies better stability and flowability of the cement paste. However, increasing the limestone microfiller contents reduced the induced bleeding of cement paste only at high W/B ratios and did not seem to have a significant effect at low W/B [11,12].

The aim of this study is to evaluate the effect of the W/B, the dosages of SP and VA, and the proportion of limestone powder replacement of cement on the rheological properties and the compressive strength (f'_c) at 7 and 28 days of grouts using a statistical design approach and analysis of experiments [13]. The mini-slump test, Marsh cone, Lombardi plate cohesion meter, induced bleeding test, and coaxial rotating cylinder viscometer were used for testing the behaviour of fresh cement grouts. The compressive strengths of grouts at 7 and 28 days of age were also measured. The established models can identify parameters and the two-way interactions that have significant effect on the rheological properties and compressive strength of grouts. The models can be used to evaluate the potential influence of adjusting mix variables on grout properties required to ensure successful development of grout. Such simulation can facilitate the test protocol needed to optimize grout with a given set of performance criteria that can be tried in the laboratory.

2. Statistical design approach

The technique of analysis used was a statistical analysis of the results obtained from a set of experiments [13]. This technique applied to cement material grout can give a lot of information from a few experiments. A 2^{4-1} fractional statistical experimental design ($2^{k-1} = 8$) was used to evaluate the influence of two different levels for each independent variable on the relevant grout properties. Four key parameters ($k = 4$) that can have significant influence on mix characteristics of cement grout were selected to derive mathematical models for evaluating relevant properties. The four variables were W/B, dosages of SP and VA, and proportion of LSP as replacement of cement. The modelled experimental region consisted of mixes ranging between coded variables of -1 to $+1$. The derived statistical models are valid for mixes made with ranges of W/B of 0.35–0.42, dosages of VA of 0.02–0.07%, by mass of binder (or 0.057–0.166% of water), SP of 0.3–1.2%, by mass of binder, and the proportion of LSP from 12 to 45% (Table 1). The model

Table 1
Mix proportion for grouts used in the two-level fractional factorial design

	Coded values					Actual values			
		W/B	SP	LSP	VA	W/B	SP (%)	LSP (%)	VA (%)
Levels of factors	1	−1	−1	−1	−1	0.35	0.3	12.0	0.02
	2	1	−1	−1	1	0.42	0.3	12.0	0.07
	3	−1	1	−1	1	0.35	1.2	12.0	0.07
	4	1	1	−1	−1	0.42	1.2	12.0	0.02
	5	−1	−1	1	1	0.35	0.3	45.0	0.07
	6	1	−1	1	−1	0.42	0.3	45.0	0.02
	7	−1	1	1	−1	0.35	1.2	45.0	0.02
	8	1	1	1	1	0.42	1.2	45.0	0.07
Centre points	9	0	0	0	0	0.385	0.75	28.5	0.045
	10	0	0	0	0	0.385	0.75	28.5	0.045
	11	0	0	0	0	0.385	0.75	28.5	0.045
	12	0	0	0	0	0.385	0.75	28.5	0.045
Points of verification	13	0.14	0.00	0.09	−0.60	0.39	0.75	30.0	0.03
	14	0.43	0.33	−0.82	−0.20	0.40	0.90	15.0	0.04
	15	0.71	0.44	0.39	1.00	0.41	0.95	35.0	0.07
	16	0.43	0.33	0.24	−0.20	0.40	0.90	32.5	0.04
	17	−0.14	−0.56	−0.52	−0.60	0.38	0.50	20.0	0.03

consisted of eight factorials points where each variable was fixed at two different levels.

Four replicate central points were prepared to estimate the degree of experimental error for the modelled responses. The central points consisted of mixes with variables corresponding to 0.385, 0.75%, 0.045%, and 28.5% for W/B, dosage of SP, VA and LSP, respectively. Finally, five random mixes were produced to establish the accuracy of the derived models. The mixes were produced and tested in random order, which is one of the requirements of factorial experimental design.

The fresh cement grouts were tested with mini-slump test, Marsh cone, Lombardi plate cohesion meter, induced bleeding test, and coaxial rotating cylinder viscometer. The compressive strengths were measured at 7 and 28 days.

The 17 mix combinations, expressed in coded and actual values, considered in the experimental design of grouts are listed in Table 1. The coded factors of variables are calculated as follows:

$$\text{Coded Factor} = (\text{Actual value} - \text{Factor mean})/(\text{Range of the factorials values}/2)$$
$$\text{Coded W/B} = (\text{Actual W/B} - 0.385)/0.035$$
$$\text{Coded VA} = (\text{Actual VA} - 0.045)/0.025$$
$$\text{Coded SP} = (\text{Actual SP} - 0.75)/0.45$$
$$\text{Coded LSP} = (\text{Actual LSP} - 28.5)/16.5$$

Table 2
Chemical and physical properties of cement and limestone powder

	Cement	Limestone powder
SiO_2	20.8	–
Al_2O_3	5.0	–
Fe_2O_3	3.2	–
CaO	63.7	–
MgO	2.6	0.2
Na_2O eq.	0.39	–
Free CaO	1.6	–
LOI	0.65	–
$CaCO_3$	–	99
Relative density	3.14	2.65
Specific surface area (m^2/kg)	385	–

Standard compressive strength (MPa)

Age (d)	Cement
7	41.5
28	57.8

3. Material proportions and testing procedures

The grouts investigated in this study were prepared using an ordinary Portland cement and limestone powder. The chemical and physical properties of cement and limestone powder are presented in Table 2. The limestone powder was produced from carboniferous limestone of a very high purity and was finer than cement. The limestone had grading of 98% < 45 and 25% < 5 μm.

A new generation of superplasticiser on the basis of modified polycarboxylates was used with a solid content of 30% and specific gravity of 1.11. The viscosity agent was the Kelco–Crete welan gum that is a high molecular weight, microbial polysaccharide. The welan gum was supplied in a powder gum.

All grout mixes were prepared in a 5 l planar-action high-shear mixer. The mixing tap water had a temperature of 16 ± 1 °C, which was measured before mixing started. The viscosity agent was mixed with cement. The superplasticiser was added to the water and mixed together. Mixing time was measured from when the limestone powder (the first solid component) was added into the mix of water and superplasticiser. Finally, the mix of cement and viscosity agent was added and all components were mixed for 7 min from the start of measuring time. The grout temperature following the end of mixing was maintained at 20 ± 2 °C.

Following the end of mixing, the properties of the fresh cement grout were measured. The following tests of the fresh cement grout were carried out (the figures in brackets show the range of times when the individual tests start after finishing of mixing): mini-slump test (1–2 min), Marsh cone (4–5 min), Lombardi plate cohesion meter (10–15 min), coaxial cylinder rotation viscometer (10–20 min), induced bleeding test (20–30 min) and unit weight. Three cylinders with 50 × 55-mm diameter and height were cast to determine 7 and 28 days compressive strength.

The mini-slump test is based on the measurement of the spread of grout placed into a cone-shaped mould. The mini-slump cone has an upper diameter of 19 mm, a lower diameter of 38.1 mm, and a height of 52.7 mm. The cone is placed in the centre of a smooth plate and the spread diameter of the grout after lifting of the cone is measured.

The Marsh cone test is based on measuring the time necessary for the flow of a particular volume of grout through a flow-cone. Nowadays, different types of flow-cone are used. A plastic funnel with a capacity of 1200 ml was used in this case. One half of the upper part of the funnel was covered with a sieve. The grout was placed in the funnel through this sieve, which prevented large particles blocking the outlet. The funnel was wetted before each test. A volume of 1100 ml of the grout was placed in the cone with the outlet sealed and then the time for the flow of each 100 ml of grout was recorded. The flow time of Marsh cone at 700 ml was evaluated.

The cohesiveness of the grout was measured with a Lombardi plate cohesion meter [15]. The apparatus consists of a thin steel plate (100 mm × 100 mm × 1 mm), on which the grout can stick, and an electronic scale. The clean dry plate was weighed and then submerged once into the grout. The plate was then withdrawn and weighed again after any dropping of grout stopped (Fig. 1).

The specific weight of the grout was measured by a mud balance. This mud balance consists of a constant-volume sample cup with lid connected to a balance arm. A reader is moved along the balance arm to indicate the scale reading. There is a knife edge attached to the arm near the balance cup and a bubble level built into this knife edge for levelling the arm. It was possible to calculate the thickness of grout on each side of the plate from the unit weight and the amount of grout sticking to the steel plate.

The resistance of the fresh grout to induced bleeding was evaluated using a pressure filter. The equipment consists of a pressure vessel, filter paper, which is placed on a sieve, and a graduated cylinder. A 200 ml grout sample is placed in the pressure vessel. After closing the cell, the graduated cylinder is placed under the outlet of the cell. The cell is pressured by compressed air to 0.55 MPa. The volume of water going out through the outlet on the bottom of the cell is recorded at 15 and 30 s, then at every minute up to 10 min, and then at every 5 min up to 30 min [16]. The results of this test are presented as area under curve response time vs. volume of water (Fig. 2).

The viscosity of cement grout is determined using a coaxial rotating cylinder viscometer (smooth cylinders, no serration) that enabled the determination of apparent viscosity at different shear rates [17]. The test is contained in the annular space between an outer cylinder (rotor) with radius of 18.415 mm and a bob with radius of 17.245 mm and height of 3.80 cm. The rotor and the bob are plunged into a cup which contains 350 ml of sample (Fig. 3) [17]. Viscosity measurements are made

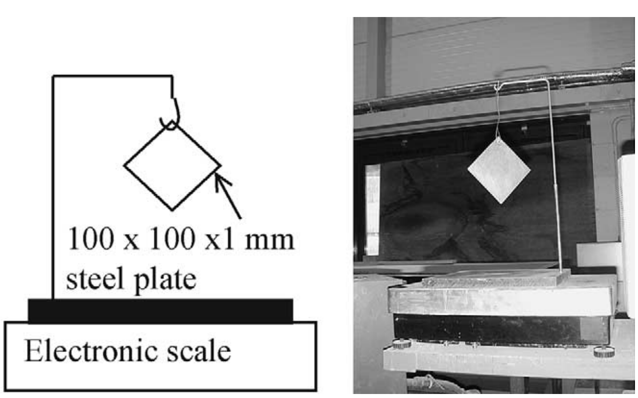

Fig. 1. Plate cohesion meter apparatus [15]. W/B = 0.35, SP = 1.2%, VA = 0.02%, LSP = 45%.

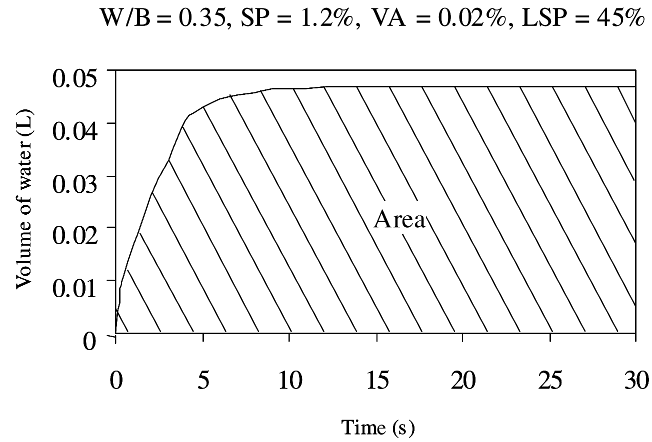

Fig. 2. Example of the results of induced bleeding test.

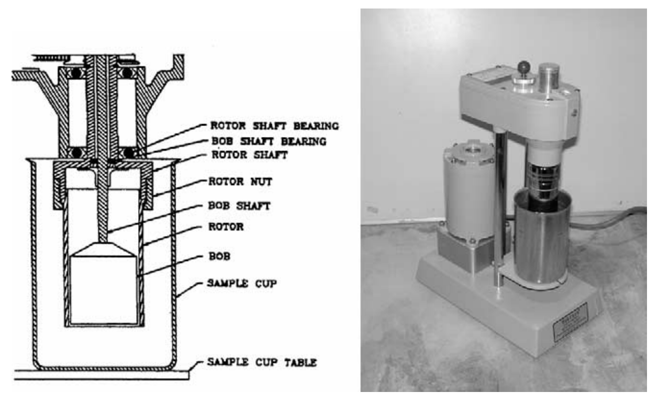

Fig. 3. Coaxial rotating cylinder viscometer [17].

when the outer cylinder, rotating at a known speed, causes a viscous drag to be exerted by the fluid. This drag creates a torque on the bob, which is transmitted to a precision spring where its deflection is measured and then compared with the test conditions and the instrument's constants. The measurement is made for 12 speeds of rotor from 0.9 to 600 rpm when the values of viscometer reading $= \theta$ are recorded. The value of shear stress $= \tau$ (Pa) is calculated by including $k_1 =$ torsion constant of spring per unit deflection (N cm/deg.), $k_2 =$ shear stress constant for the effective bob surface (cm^{-3}) and $k_3 =$ shear rate constant (s^{-1}/rpm) [17].

The speed of rotor was increased step by step from 0 to 600 rpm and reading on viscometer with increasing rotating speed was recorded. The reading of θ was taken when the needle in the viscometer was stabilised, or 30 s after the change of speed in cases when the needle has not stabilised which was caused by the thixotropy of the cement grout. The measurement of the reading by decreasing rotating speed step by step allowed the assessment of the thixotropy of grout between the ascending and descending legs of the shear stress–shear rate rheograms. The upcurve was chosen for final evaluation for better description of rheological behaviour of the grouts including a structural breakdown phenomenon of inner forces among particles [18,19].

The values of yield stress and plastic viscosity are obtained from modified Bingham model [20], which is described by the equation:

$$\tau = \tau_0 + \mu_p \dot{\gamma} + c\dot{\gamma}^2$$

where τ_0 = yield stress (Pa), μ_p = plastic viscosity (Pa s), $\dot{\gamma}$ = shear rate (s^{-1}), c = constant.

The value of the constant c is mostly about 10^{-3} and less, which is much smaller than the value of plastic viscosity μ_p and yield stress τ_0, and for this reason this constant is considered to equal zero.

The compressive strength was determined on 55 × 50-mm cylinders (diameter × height). The specimens were demoulded one day after casting and were stored in water until testing at 7 and 28 days.

4. Test results and discussion

4.1. Derived statistical models

The test results for mixes investigated in this study are given in Table 3. The various responses which resulted from the designed experimental programme were analysed and plotted using a statistical software package [14]. The derived statistical models for all these tests results with correlation coefficient, Prob. > $|t|$ values and prospective transformation were shown in Table 4. The estimates for each parameter refer to the coefficients of the model found by a least-squares approach. The Prob. > $|t|$ is the probability of getting an event greater t statistic, in absolute value, that tests whether the true parameter is zero. Probabilities less than 0.05 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 4 enables the comparison of various parameters as well as the interactions of the modelled responses. For the majority of 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.

Transformation was used for stabilising of the model in two cases. For example, the transformation of natural logarithm was used for plate cohesion meter and plastic viscosity. In order to illustrate the method, assume that responses Y_1 and Y_2 are functions of W/B, dosages of SP and VA, and the proportion of LSP, then

Linear model:

$$Y_1 = a_0 + a_1\,\mathrm{W/B} + a_2\,\mathrm{SP} + a_3\,\mathrm{LSP} + a_4\,\mathrm{VA} + a_5\,\mathrm{W/B\,SP} + a_6\,\mathrm{W/B\,LSP} + a_7\,\mathrm{W/B\,VA} + \varepsilon$$

Natural logarithm:

$$\ln Y_2 = a_0 + a_1\,\mathrm{W/B} + a_2\,\mathrm{SP} + a_3\,\mathrm{LSP} + a_4\,\mathrm{VA} + a_5\,\mathrm{W/B\,SP} + a_6\,\mathrm{W/B\,LSP} + a_7\,\mathrm{W/B\,VA} + \varepsilon$$

Table 3
Results of testing methods used for individual mixes

Mix	Mini-slump (mm)	Flow time (s)	Cohesion meter (mm)	Viscometer		Induced bleeding (L min)	f'_c 7 days (MPa)	f'_c 28 days (MPa)
				Yield value (Pa)	Plastic viscosity (Pa s)			
1	77.0	NM	1.350	11.18	0.52	1.09	40.3	47.5
2	61.5	NM	1.409	8.40	0.40	1.22	26.7	34.7
3	77.0	NM	1.068	10.77	1.17	0.93	38.3	39.2
4	179.5	81.4	0.082	1.81	0.11	0.51	29.7	33.5
5	64.5	NM	1.742	4.43	0.79	0.92	23.0	31.4
6	118.0	184.3	0.570	11.26	0.17	1.29	17.0	23.7
7	142.0	174.3	0.079	2.55	0.17	1.29	25.5	26.8
8	172.5	197.5	0.164	5.46	0.13	1.40	17.7	20.0
9	113.0	321.7	0.445	10.98	0.35	1.24	35.0	37.9
10	126.0	295.9	0.362	10.18	0.33	1.30	29.1	39.2
11	116.0	474.2	0.318	9.81	0.27	1.25	32.3	34.1
12	111.5	566.9	0.271	9.09	0.29	1.25	27.8	33.5
13	150.5	61.0	0.144	6.15	0.19	1.28	29.9	40.0
14	104.5	NM	0.430	10.25	0.24	1.52	34.8	45.3
15	134.0	>20 min	0.221	9.77	0.20	1.01	24.2	28.9
16	142.0	242.4	0.153	6.47	0.16	1.32	30.0	33.8
17	117.0	702.4	0.296	9.00	0.31	1.35	34.4	42.5

NM: not measurable.

Table 4
Parameter estimates of seven statistical models

		Mini-slump (mm)		Plate cohesion meter (mm)		Yield value (Pa)		Plastic viscosity (Pa s)		Induced bleeding (L min)		f_c' 7 days (MPa)		f_c' 28 days (MPa)	
		$R^2 = 0.99$		$R^2 = 0.97$		$R^2 = 0.97$		$R^2 = 0.97$		$R^2 = 0.99$		$R^2 = 0.91$		$R^2 = 0.93$	
		Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$	Estimate	Prob. > $\|t\|$
	Transform	none		natural log		none		natural log		none		none		none	
a_1	Intercept	111.50		−0.77		6.98		−1.18		1.08		27.28		32.10	
a_2	W/B	21.37	0.00	−0.37	0.01	−0.25	0.37	−0.56	0.00	0.024	0.08	−4.50	0.00	−4.13	0.00
a_3	SP	31.25	0.00	−0.93	0.00	−1.84	0.00	−0.28	0.01	−0.048	0.01	–	–	−2.23	0.03
a_4	LSP	12.75	0.00	−0.32	0.02	−1.06	0.01	−0.28	0.01	0.14	0.00	−6.48	0.00	−6.63	0.00
a_5	VA	−17.63	0.00	0.56	0.00	0.28	0.31	0.42	0.00	0.038	0.02	–	–	–	–
a_6	W/B SP	11.88	0.00	–	–	−1.26	0.01	–	–	−0.1	0.00	–	–	–	–
a_7	W/B LSP	–	–	0.27	0.04	2.68	0.00	–	–	0.1	0.00	–	–	–	–
a_8	W/B VA	–	–	–	–	–	–	−0.16	0.06	0.17	0.00	–	–	–	–

where a_0 denotes the overall mean; coefficients a_n represent model constants (contribution of independent variables on the response), and ε is the random error term representing the effects of uncontrolled variables. The third order interaction is usually neglected.

For example, mini-slump, plate cohesion meter, yield value, plastic viscosity, induced bleeding and f_c' at 28 days are given in Eqs. (1)–(6), respectively.

$$\text{Mini-slump (mm)} = 111.5 + 31.3\,\text{SP} + 21.4\,\text{W/B} - 17.6\,\text{VA} + 12.75\,\text{LSP} + 11.9\,\text{W/B}\,\text{SP} \quad (1)$$

$$\ln \text{Plate cohesion meter (mm)} = -0.77 - 0.93\,\text{SP} + 0.56\,\text{VA} - 0.37\,\text{W/B} - 0.32\,\text{LSP} + 0.27\,\text{W/B}\,\text{LSP} \quad (2)$$

$$\text{Yield value (Pa)} = 7 + 2.7\,\text{W/B}\,\text{LSP} - 1.8\,\text{SP} - 1.3\,\text{W/B}\,\text{SP} - 1.1\,\text{LSP} + 0.3\,\text{VA} - 0.3\,\text{W/B} \quad (3)$$

$$\ln \text{Plastic viscosity (Pa s)} = -1.2 - 0.56\,\text{W/B} + 0.42\,\text{VA} - 0.28\,\text{SP} - 0.28\,\text{LSP} - 0.16\,\text{W/B}\,\text{VA} \quad (4)$$

$$\text{Induced bleeding (L min)} = 1.1 + 0.17\,\text{W/B}\,\text{VA} + 0.14\,\text{LSP} - 0.1\,\text{W/B}\,\text{SP} + 0.1\,\text{W/B}\,\text{LSP} + 0.05\,\text{SP} - 0.04\,\text{VA} + 0.03\,\text{W/B} \quad (5)$$

$$f_C' \text{ at 28 days (MPa)} = 32.1 - 6.6\,\text{LSP} - 4.1\,\text{W/B} - 2.2\,\text{SP} \quad (6)$$

The correlation coefficients of the proposed models for mini-slump test, plate cohesion meter, yield value, plastic viscosity, induced bleeding and compressive strength at 7 and 28 days are 0.99, 0.97, 0.97, 0.97, 0.99, 0.91 and 0.93, respectively. The high correlation coefficient of most responses demonstrates excellent correlation where it can be considered that at least 95% of the measured values can be accounted for with the proposed models.

Table 5 indicates the average measured response of the four replicate grouts, coefficient of variation, estimated error with 95% confidence limit, as well as relative error for each of the measured properties. The estimated error of cement grout for mini-slump, plate cohesion meter, yield value, plastic viscosity, induced bleeding, and compressive strengths at 7 and 28 days were ±6.6 mm, ±0.08 mm, ±0.80 Pa, ±0.04 Pa s, ±0.026 L min, ±3.3, and ±2.9 MPa, respectively.

The relative experimental errors for mini-slump, yield value, plastic viscosity and compressive strength are shown to be limited to 2–12%. On the other hand, the relative error for the plate cohesion meter was 22%. This value is expected to decrease with the increase in relative cohesion plate value, since the mean of cohesion plate value of the grout corresponding to the central points was slightly low.

The flow time model is not given as the results of the Marsh cone could not be used for final evaluation because the grouts with high level of viscosity agent (0.07%) had such high viscosity that they were unable to flow through the 5-mm-outlet of the funnel. In this case, an outlet bigger than 5 mm is recommended.

4.2. Accuracy of the proposed models

The accuracy of the statistical models was determined by comparing average values of predicted/measured ratio for mixes of grout which were produced for verification of two-level fractional factorial design. The aver-

Table 5
Repeatability of test parameters

	Mini-slump	Plate cohesion meter	Viscometer		Induced bleeding in 30 min	f'_c 7 days	f'_c 28 days
			Yield value	Plastic viscosity			
Mean ($n = 4$)	116.6 mm	0.35 mm	10.0 Pa	0.31 Pa s	1.26 L min	31.1 MPa	36.2 MPa
Coefficient of variation (%)	5.6	21.3	7.9	12.3	2.0	10.4	7.8
Estimated error (95% confidence limit)	6.6 mm	0.08 mm	0.80 Pa	0.04 Pa s	0.026 L min	3.3 MPa	2.9 MPa
Relative error (%)	5.7	21.7	8.0	12.5	2.1	10.6	7.9

age predicted/measured ratios for mini-slump test, plate cohesion meter, yield value, plastic viscosity, induced bleeding test, and compressive strengths at 7 and 28 days are summarised in Table 6.

The ratios between predicted and measured properties of cement grout ranged between 0.83 and 1.05, thus indicating good accuracy for established models to predict the mini-slump test, yield value, plastic viscosity, induced bleeding, and compressive strength at 7 and 28 days. In general, the proposed models to predict mini-slump, yield value, plastic viscosity, induced bleeding and compressive strength appear to be satistifactory in predicting the fluidity, rheology, induced bleeding and compressive strength. On the other hand, the average value of predicted/measured ratio of plate cohesion meter was slightly higher (1.45).

4.3. Isoresponses of the proposed models of the key variables

4.3.1. Mini-slump

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. As shown in Table 4, the mini-slump is influenced, in order of significance, by the dosage of SP, W/B, the dosage of VA, the proportion of LSP and the interaction effect of W/B SP. The dosage of SP is shown to exhibit the greatest effect on the mini-slump. The increase in SP has approximately 1.8 and 1.5 times greater influence on increasing mini-slump than the decrease in the dosage of VA and the increase in W/B, respectively (31.3 vs. −17.6 and 21.4). For example, the effect of increasing W/B ratio on mini-slump vs. the proportion of LSP, when dosages of SP and VA were fixed at 0.75% and 0.05%, respectively, or vs. dosage of SP of 0.75% and the proportion of LSP of 28.5%, respectively, or vs. the dosage of VA of 0.05% and the proportion of LSP of 28.5%, is shown in Fig. 4. For any given W/B ratio and dosages of SP and VA fixed at 0.75% and 0.05%, respectively, the mini-slump increased significantly when the proportion of LSP increased (Fig. 4(a)). Similarly, the mini-slump increased when the dosage of SP increased while the dosage of VA and the proportion of LSP were fixed (Fig. 4(c)). On the other hand, the

Table 6
Predicted/measured ratios for mixes of established models

Testing method	Mini-slump	Cohesion meter	Viscometer		Induced bleeding	f'_c 7 days	f'_c 28 days
			Yield value	Plastic viscosity			
Verification points	0.97	1.45	0.85	1.05	0.87	0.87	0.83

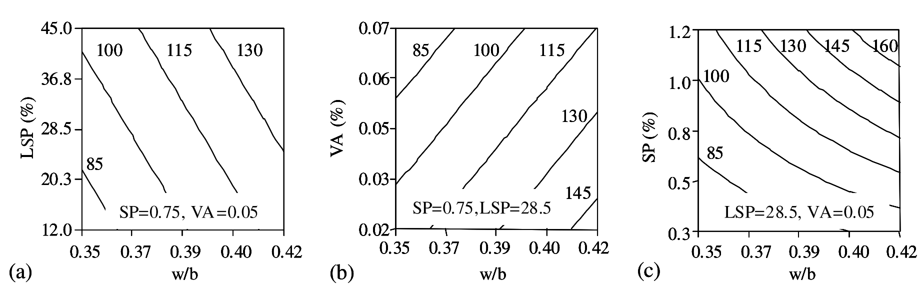

Fig. 4. Isoresponse curve for mini-slump test with W/B vs. LSP (%) or VA (%) or SP (%).

mini-slump reduced when the dosage of VA increased while the dosage of SP and the proportion of LSP were kept constant (Fig. 4(b)).

4.3.2. Plate cohesion meter

As shown in Table 4, plate cohesion meter is influenced, in the order of significance, by the dosages of SP and VA, the W/B and the proportion of LSP. The increase in W/B has a fairly similar influence on the plate cohesion meter as the increase in the proportion of LSP (−0.37 vs. −0.32). By comparing the effect of SP and VA dosages on the plate cohesion meter, the increase of dosage of SP can then be interpreted to have approximately 1.7 times greater influence on the reduction of the plate cohesion value than the increase in VA, given that the W/B and the proportion of LSP are held constant. Fig. 5 shows an example of isopresponse curves of the plate cohesion meter vs. W/B for fixed values of SP and VA or SP and LSP or LSP and VA. For fixed values of SP and VA at 0.75% and 0.045%, respectively, the plate cohesion meter decreased when the W/B increased or the proportion of LSP increased (Fig. 5(a)). The increase of the dosage of SP, for any given W/B and fixed LSP proportion and VA dosage, led to a reduction in the plate cohesion meter (Fig. 5(c)).

4.3.3. Yield value

As shown in Table 4, the quadratic effect of W/B LSP is shown to exhibit the greatest effect on the yield value following by the dosage of SP (2.7 and −1.8). The increase of W/B and the proportion of LSP reduced the yield value. The model (Eq. (3)) shows that the increase of the dosage of SP is more efficient in reducing yield value than an increase in the proportion of LSP (−1.8 vs. −1.1). For example, the effect of increasing W/B ratio on yield value vs. the proportion of LSP, when dosages of SP and VA were fixed at 0.75% and 0.05%, respectively, or vs. dosage of SP of 0.75% and the proportion of LSP of 28.5%, respectively, or vs. the dosage of VA of 0.05% and the proportion of LSP of 28.5%, is shown in Fig. 6. The yield value seemed to decrease with increased W/B up 0.40, then tended to increase beyond this threshold value (Fig. 6(a)). For fixed dosages of SP and VA, the yield value increased significantly up to about 0.40 of W/B and higher proportion of LSP (up to 29%). At lower proportions of LSP, however, a decrease of yield value was observed with an increase in W/B (Fig. 6(a)).

4.3.4. Plastic viscosity

Plastic viscosity is influenced, in order of significance, by the W/B, the dosage of VA, the dosage of SP and the proportion of LSP. The W/B is shown to have the greatest effect on the plastic viscosity (Eq. (4)). The increase of W/B has approximately 1.3 times greater influence on reducing the plastic viscosity than the decrease in the dosage of VA (−0.56 vs. 0.42). The model (Eq. (4)) shows that the effect of changing the dosage of SP on the plastic viscosity is similar to that of the proportion of LSP (−0.28 vs. 0.28). By comparing the effects of SP and VA on the plastic viscosity, it can be observed

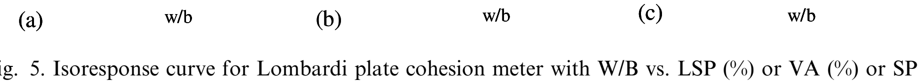

Fig. 5. Isoresponse curve for Lombardi plate cohesion meter with W/B vs. LSP (%) or VA (%) or SP (%).

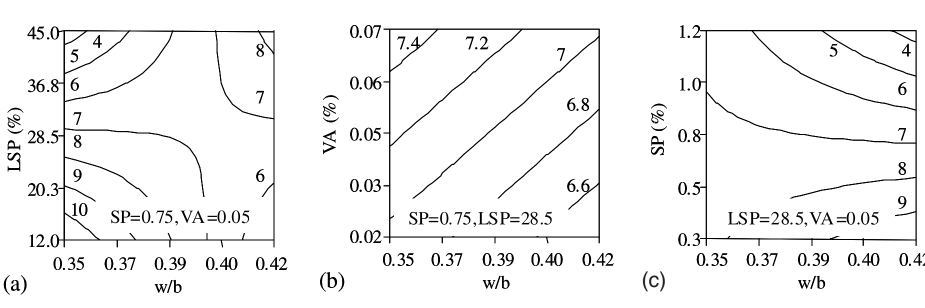

Fig. 6. Isoresponse curve for yield value with W/B vs. LSP (%) or VA (%) or SP (%).

that the effect of VA is higher than that of SP (0.42 vs. −0.28). For example, the effect of increasing W/B ratio on plastic viscosity vs. the proportion of LSP, when dosages of SP and VA were fixed at 0.75% and 0.05% respectively, or vs. dosage of SP of 0.75% and the proportion of LSP of 28.5%, respectively, or vs. the dosage of VA of 0.05% and the proportion of LSP of 28.5%, is shown in Fig. 7. The increase in W/B and/or the proportion of LSP led to a reduction in plastic viscosity (Fig. 7(a)).

4.3.5. Induced bleeding

The influences of the proportion of LSP and the dosages of SP and VA are highly significant on the induced bleeding according to the ANOVA. The proportion of LSP is shown to exhibit the greatest effect as a primary variable on the induced bleeding compared to the dosages of SP and VA (0.14 vs. −0.05 or 0.04). However, the ANOVA shows that the two-factor interaction of W/B VA is highly significant and has the greatest effect on induced bleeding. The interactions between W/B SP and W/B LSP are significant and have opposite effect (−0.1 vs. 0.1). The increase in SP dosage has a greater influence on reducing the induced bleeding than the increase in VA dosage (−0.05 vs. 0.04). For example, the effect of increasing W/B ratio on induced bleeding vs. the proportion of LSP, when dosages of SP and VA were fixed at 0.75% and 0.05%, respectively, or vs. dosage of SP of 0.75% and the proportion of LSP of 28.5%, respectively, or vs. the dosage of VA of 0.05% and the proportion of LSP of 28.5%, is shown in Fig. 8. For fixed dosage of VA and the proportion of LSP, the increase in SP led to an increase in induced bleeding for lower W/B up to 0.38. However, for higher W/B (between 0.38 and 0.42), the increase of the dosage of SP resulted in a reduction in the induced bleeding (Fig. 8(c)). This is due to the improved dispersion and packing of cement grains associated with greater SP dosage. The resulting increase in fluidity (Fig. 4) and particle packing (versus flocculated cement grains that have a lower packing density) can substantially reduce the tendency of water to percolate among cement grains under a given head, which reflects the permeability of the fresh grout. These results concur with the findings of other researchers [3,4]. For lower W/B (lower than 0.38), the increase in VA dosage exhibited a reduction in the induced bleeding, however for higher W/B beyond 0.39, the induced bleeding seemed to increase as the dosage of VA increased (Fig. 8(b)).

4.3.6. Compressive strength

The ANOVAs given in Table 4 show that the effect of W/B is highly significant on compressive strength at 7 and 28 days and has the greatest effect on strength. The compressive strength decreased, as W/B increased. The proportion of LSP and the dosage of SP are also highly significant on compressive strength. The increase of the proportion of LSP or the dosage of SP led to a reduction in compressive strength at 28 days. The effect of the proportion of LSP on compressive strength was greater than that of W/B and SP dosage.

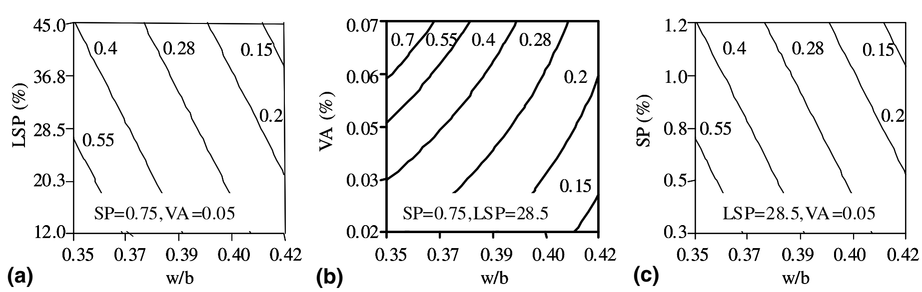

Fig. 7. Isoresponse curve for plastic viscosity with W/B vs. LSP (%) or VA (%) or SP(%).

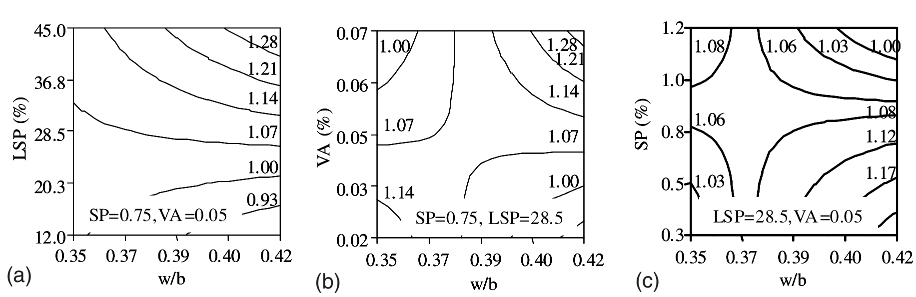

Fig. 8. Isoresponse curve for induced bleeding test with W/B vs. LSP (%) or VA (%) or SP (%).

4.4. Trade-off between SP and VA

Contour responses showing the influence of SP and VA dosages on mini-slump and plate cohesion meter, and mini-slump and yield value for grouts made with fixed W/B of 0.35 and 12% of LSP are presented in Fig. 9. As expected, for a given SP dosage, the contour diagrams of Fig. 9(a) indicate that the increase in VA dosage reduces the mini-slump while the plate cohesion meter increases. For example, for mini-slump of 90 mm, a mix grout with 0.6% of SP and 0.02% of VA can ensure the fluidity of 90 mm. The increase in VA dosage to 0.042% resulted in a reduction of mini-slump to 75 mm. However, by increasing the SP from 0.6% to 0.97%, the mini-slump can be re-established to 90 mm. For the same mix, with 0.6% of SP and 0.02% of VA, the increase in VA dosage to 0.042% increased the plate cohesion meter from 0.9 to 1.4 mm. The plate cohesion meter can be re-established to 0.9 mm by increasing SP dosage to 0.84%.

For a given VA dosage, the results from Fig. 9(b) indicate that the increase in SP led to an increase in mini-slump and a reduction in the yield value. For example, a grout made with 0.05% of VA and 0.3% of SP had mini-slump of 60 mm and yield value of 11.5 Pa. The increase of SP to 1% resulted in an increase of mini-slump to 90 mm and a reduction of yield value to 10.6 Pa. However, by reducing the VA from 0.05% to 0.02% for the same SP of 0.3%, the mini-slump increased to 75 mm and the yield value decreased to 11.25 Pa.

Contour responses showing the influence of SP and VA dosages on plastic viscosity and induced bleeding with mix made with 0.35 of W/B and LSP of 12% and 0.42 of W/B and LSP = 45%, are presented in Fig. 10(a) and (b), respectively. For given VA and SP dosages, the increase of W/B and the proportion of LSP resulted in an increase in induced bleeding and a reduction in plastic viscosity. For example, for grout made with 0.42 W/B and 45% of LSP, plastic viscosity and induced bleeding of 0.145 Pa s and 1.40 L min are obtained with 0.66% of SP and 0.052% of VA. The increase of SP to 0.97% resulted in a reduction of plastic viscosity and induced bleeding to 0.12 Pa s and 1.30 L min, respectively. With 0.050% and 0.3% of VA and SP, a grout made with 0.35 of W/B and 12% of LSP had a plastic viscosity and induced bleeding of approximately 0.9 Pa s

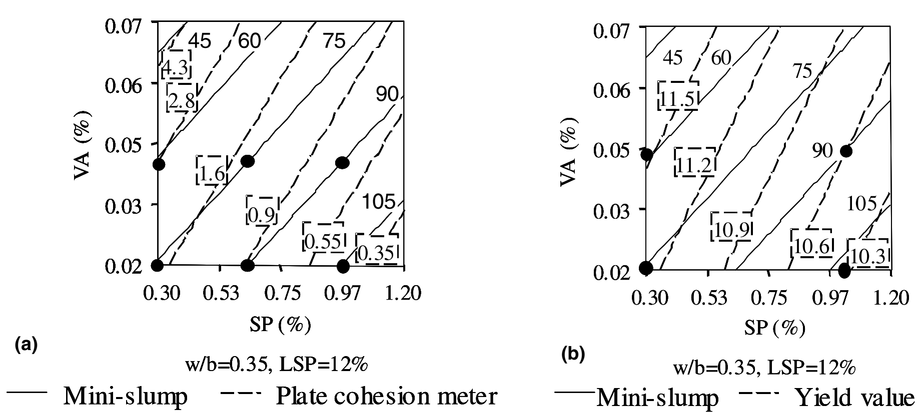

Fig. 9. Contour diagram of mini-slump and Lombardi plate cohesion meter and yield value (W/B = 0.35 and LSP = 12%).

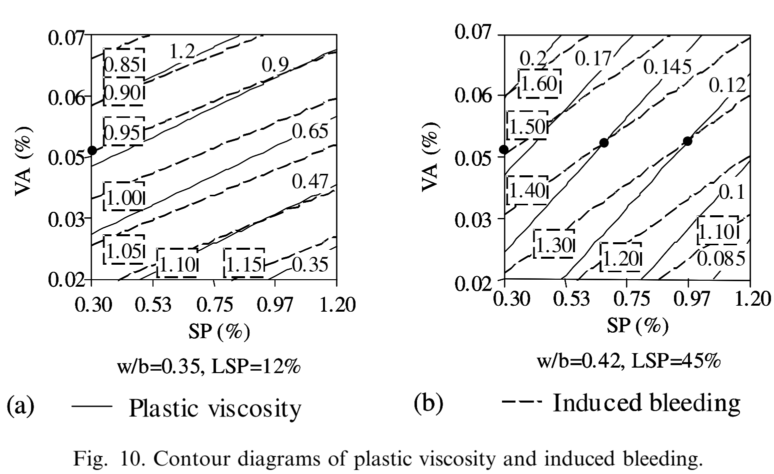

Fig. 10. Contour diagrams of plastic viscosity and induced bleeding.

and 0.95 L min, respectively. The increase of W/B and LSP to 0.42 and 45%, respectively, would result in a drop of plastic viscosity to 0.17 Pa s and an increase in induced bleeding to 1.50 L min.

4.5. Correlation of testing methods

The flow of grout is very sensitive to its shear history. The above tests were carried out with extreme care in order to keep the shear history, the experimental procedures and their timing as constant as possible. Hence, it is interesting to assess the various possible correlations between the different tests carried out.

Fig. 11 shows the relationship between three rheological values which are characteristic of the grout at low shear rates: mini-slump, plate cohesion meter and yield value. The coefficient of correlation R^2 between mini-slump and plate cohesion meter, and the mini-slump and yield value are 0.95 and 0.56, respectively. The relationship seemed to follow polynomial second-order model and showed that when the mini-slump increases the plate cohesion meter and the yield value decrease. The relationship between mini-slump and the plate cohesion meter was very good. Fig. 12(a) and (b) illustrate the correlations between rheological characteristics of grouts at low and high shear rates: the plate cohesion meter and plastic viscosity, mini-slump and plastic viscosity. The coefficients of correlation between plate cohesion meter and plastic viscosity, and the mini-slump and plastic viscosity are 0.80 and 0.75, respectively ($R^2 = 0.80$ was obtained without the result of mix 3). Fig. 12 shows that the increase in plastic viscosity led to an increase in plate cohesion meter and a reduction in mini-slump.

In Fig. 13(a) and (b), the relationship between induced bleeding and mini-slump, and induced bleeding and plastic viscosity are presented. It seems that there is no correlation between mini-slump and induced bleeding. However, Fig. 13(b) shows a good relationship between induced bleeding and plastic viscosity ($R^2 = 0.85$, without taking account of the results from mixes 3, 4 and 15). The induced bleeding was inversely

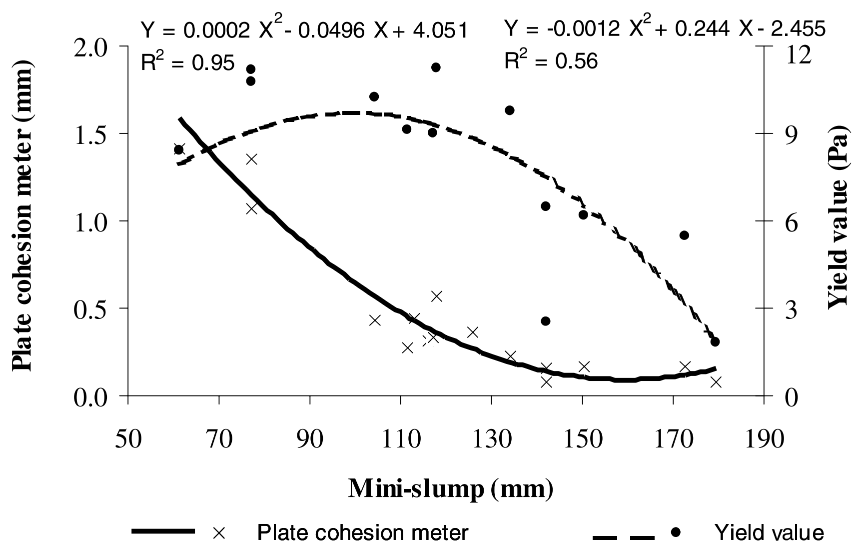

Fig. 11. Correlations between mini-slump, plate cohesion meter and yield value.

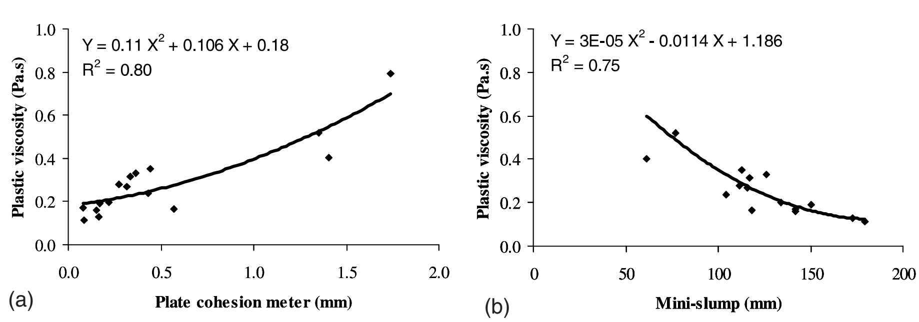

Fig. 12. Correlations between plastic viscosity vs. plate cohesion meter or vs. mini-slump.

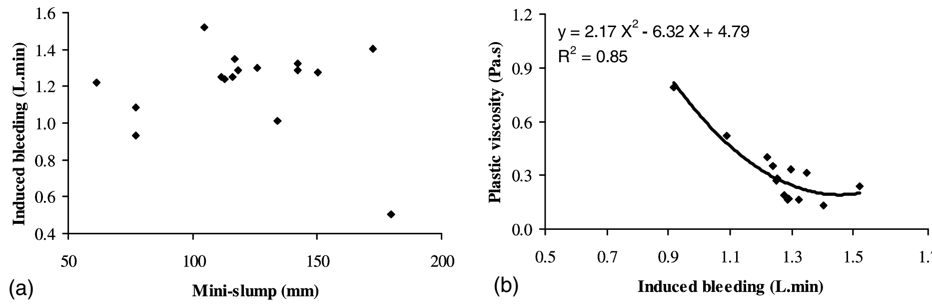

Fig. 13. Correlations between induced bleeding vs. mini-slump or vs. plastic viscosity.

proportional to the plastic viscosity. This finding confirms what has been reported by Nedhi et al. [12].

5. Conclusions

The influence of different W/B, dosage of SP, the proportion of limestone powder and the dosage of viscosity agent combinations on rheology behaviour of cement grout were investigated. Based on the results presented in this paper, the following conclusions can be drawn:

(1) The W/B ratio is shown to exhibit a great effect on mini-slump, plastic viscosity and compressive strength. The increase in W/B ratio has an influence on increasing mini-slump and induced bleeding, and decreasing plastic viscosity, plate cohesion meter and compressive strength.
(2) The mini-slump, plate cohesion meter, and yield value of grouts are dominated primarily by the dosage of SP. The increase in the SP dosage led to an increase in mini-slump and a reduction in plate cohesion meter, yield value, and plastic viscosity. However, the induced bleeding seemed to increase when the SP dosage increased for low W/B, and reduced with higher W/B.
(3) The viscosity agent significantly affected the measured properties of this study, except compressive strength. The increase in VA dosage is shown to exhibit a reduction in mini-slump and an increase in plate cohesion meter and plastic viscosity. For low W/B, the increase in VA dosage reduced the induced bleeding, and increased it when W/B is higher.
(4) For a given W/B, and dosages of SP and VA, the mini-slump and induced bleeding increased when the proportion of LSP increased, while the plate cohesion meter, yield value, plastic viscosity and compressive strength reduced. The LSP replacement of cement had a greater effect on compressive strength than the change of W/B ratio.
(5) The proposed method can be used with other sets of materials such as fly ash or ground granulated blast slag as replacement of cement, to predict the rheological properties and compressive strength of grout but the differences between the predicted and measured values will then indicate the effect of the new materials on the accuracy of the proposed models.

References

[1] Khayat KH, Ballivy G. High-performance cement grout for underwater crack injection. In: Malhotra VM, editor. Proceedings of Third CANMET/ACI International Conference on Performance of Concrete in Marine Environment. 1996. p. 138–162.
[2] Houlsby AC. Construction and design of cement grouting. In: A guide to grouting in rock foundations. New York: John Wiley and Sons; 1990. p. 442.
[3] Khayat KH, Yahia A, Duffy P. High-performance cement grout for post-tensioning applications. ACI Mater J 1999;96(4):471–7.
[4] Khayat KH, Yahia A. Effect of welan gum-high-range water reducer combinations on rheology of cement grout. ACI Mater J 1997;94(5):365–72.
[5] Ghio VA, Monteiro PJM, Demsetz LA. The rheology of fresh cement paste containing polysaccharide gums. Cem Concr Res 1994;24(2):243–9.
[6] Bury JR, Farzam H. Laboratory evaluation of a unique anti-washout admixtures in grouts. In: Malhotra VM, editor. Proceedings of Fifth CANMET/ACI International Conference on Superplasticizers and other Chemical Admixtures in Concrete, ACI SP-173, Rome, 1997. p. 445–473.
[7] Sonebi M. Factorial design modelling of mix proportion parameters of underwater composite cement grouts. Cem Concr Res 2001;31(11):1553–60.
[8] Sonebi M. Experimental design to optimize high-volume of fly ash grout in the presence of welan gum and superplasticizer. Mater Struct 2002;35(250):373–80.
[9] Yahia A, Khayat KH. Experiment design to evaluate interaction of high-range water-reducer and antiwashout admixture in high-performance cement grout. Cem Concr Res 2001;31(5):749–57.
[10] Khayat KH, Yahia A. Simple field tests to characterize fluidity and washout resistance of structural cement grout. Cem Concr Aggregates 1998;20(1):145–56.
[11] Nehdi M, Mindess S, Aïtcin P-C. Optimization of high strength limestone filler cement mortars. Cem Concr Res 1996;26(6):883–93.
[12] Nehdi M, Mindess S, Aïtcin P-C. Statistical modelling of the microfiller effect on the rheology of composite cement pastes. Adv Cem Res 1997;9(33):37–46.
[13] Montgomery DC. Design and analysis of experiments. 4th ed. USA: John Wiley & Sons; 1996. p. 704.

[14] Design-Expert 5.0 software for design of experiments. Stat-Ease Inc., Statistics Made Easy, USA, 1996.
[15] Lombardi G. The role of cohesion in the cement grouting of rock. In: Proceedings of Fifteenth Congress on Large Dams. International Commission on Large Dams, Lausanne, vol. 3. 1985. p. 235–261.
[16] Shupack M. Admixture for controlling bleed in cement grout used in post-tensioning. PCI J 1974:28–39.
[17] Fann Instrument Company, Model 35 Viscometer—Instruction Manual, 1996.
[18] Khayat KH, Saric-Coric M, Liotta F. Influence of thioxotropy on stability characteristics of cement grout and concrete. ACI Mater J 2002;99(3):234–41.
[19] Khayat KH, Saric-Coric M. Effect of welan gum-superplasticizer on properties of cement grouts, Sixth CANMET/ACI International Conference on Superplasticizers and Other Chemical Admixtures in Concrete, SP-195, Nice, 2000. p. 249–268.
[20] Yahia A, Khayat KH. Analytical models for estimating yield stress of high-performance pseudoplastic grout. Cem Concr Res 2001;31(5):731–8.