

CEMENTAND CONCRETE RESEARCH

Cement and Concrete Research 31 (2001) 731-738

# Analytical models for estimating yield stress of high-performance pseudoplastic grout

A. Yahia<sup>a</sup>, K.H. Khayat<sup>b,\*</sup>

<sup>a</sup>Taiheiyo Cement Corporation, 2-4-2 Osaku, Sakura, Japan <sup>b</sup>Department of Civil Engineering, Université de Sherbrooke, Sherbrooke, PQ, Canada J1K 2R1

Received 15 July 1999; accepted 20 February 2001

#### Abstract

The yield stress of cement grout is commonly determined by extrapolating the shear stress—shear rate flow curve to a zero shear rate using an analytical model. High-performance structural cement grouts, made with relatively low water-to-cementitious materials ratio (W/CM) incorporating various supplementary CMs and rheology-modifying admixtures (RMAs), can exhibit rheological behavior different than that of conventional grout. Such mixtures can exhibit high pseudoplastic shear thinning characteristics. Therefore, the degree of error in estimating the yield value of high-performance grouts can be greater than for conventional ones. In this paper, yield stresses of cement grouts made with 0%, 0.03%, 0.05%, and 0.075% of welan gum RMA, by mass of binder, and various high-range water-reducer (HRWR) concentrations were evaluated. Mixtures with different replacement values of silica fume and blast furnace slag were also investigated. All grouts had a constant W/CM of 0.40. Yield stress values obtained using various rheological models are compared. The results showed that, depending on the adopted analytical model, the deducted yield stress can be quite different. A new method to estimate yield stress of high-performance, pseudoplastic grout is proposed and shown to result in lower yield stress estimates than the other models. For mixtures made with 100% cement, the estimated values of yield stress given by the proposed model are found to be close to those estimated using the De Kee model. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Pseudoplastic grout; Rheology-modifying admixture; Shear thinning; Supplementary cementitious materials; Yield stress

## 1. Introduction

The yield stress of cement-based materials is an important property affecting flow behavior. The existence of a yield stress can dramatically influence the flow rate and filling capacity of non-Newtonian cement-based mixtures. This is especially the case for mixtures placed without vibration. As a result, the yield stress can be used as a quality control index of self-consolidating and self-leveling systems. The estimation of yield stress involves the extrapolation of shear stress—shear rate data corresponding to a zero shear rate using a given analytical rheological model.

Various analytical models have been applied to cement paste with different degrees of success [1-3]. In general, the Bingham model is used to estimate the yield stress. However, for highly pseudoplastic grout mixtures, such as those

E-mail address: kamal-khayat@courrier.usherb.ca (K.H. Khayat).

prepared with low water-to-cementitious materials ratio (W/CM) or those incorporating a rheology-modifying admixture (RMA) and supplementary CMs, the authors found that the Bingham model offered the least favorable fitting of experimental shear stress data compared to other analytical models [3]. Since the estimated yield stress values depend on the efficiency of the model in use to accurately fit the experimental data, the Bingham model may result in quite different estimates from those obtained using the Herschel–Bulkley, De Kee, and Casson models [3]. This can be due to the weakness of this linear model to fit the nonlinear portion of the flow curve observed at low shear rates, especially for highly pseudoplastic suspensions.

The objective of this study is to evaluate the effectiveness of various rheological models to estimate yield stress of high-performance cement grouts containing supplementary CMs and RMAs. The RMA is typically incorporated in high-performance grout to enhance the resistance to washout, bleeding, and sedimentation [4]. The rheological models considered in this investigation included the Bingham,

<sup>\*</sup> Corresponding author. Tel.: +1-819-821-8062; fax: +1-819-821-6949

Table 1 Investigated rheological models

Bingham	$ au =  au_0 + \mu_{\scriptscriptstyle D} \dot{\gamma}$	τ <sub>0</sub> : yield stress (Pa),
Modified Bingham	$ au =  au_0 + \mu_{ m p}\dot{\gamma} + c\dot{\gamma}^2$	$\mu_p$ : plastic viscosity (Pa s),
Herschel-Bulkley	$ au =  au_0 + \dot{K}\dot{\gamma}^2$	$\gamma$ : shear rate (s <sup>-1</sup> ),
Casson	$\tau = \tau_0 + \mu_\infty \dot{\gamma} + 2(\sqrt{\tau_0 \mu_\infty}) \sqrt{\dot{\gamma}}$	c: insignificant constant,
De Kee	$ au =  au_0 + \mu_{ m p} \dot{\gamma} { m e}^{-lpha \dot{\gamma}}$	K: consistency,
Authors' (Ref. [3])	$ au =  au_0 + 2\sqrt{ au_0\mu_p}\sqrt{\dot{\gamma}e^{-\alpha\dot{\gamma}}}$	n: power index representing the deviation from the Newtonian behavior,
	• • •	$\alpha$ : time-dependent parameter,
		$\mu_{\infty}$ : apparent viscosity at very high shear rate.

Casson, Herschel-Bulkley, and De Kee models, as well as a model proposed by the authors [3]. In the case of the Bingham model, linear and second-degree responses were employed to derive the rheological parameters for mixtures made with 100% cement. The second order model consists of assuming a second order polynomial response and then suppressing the second order term that is insignificantly low [4]. The linear Bingham model was used for mixtures containing silica fume and blast furnace slag. All of the investigated models are described in Ref. [3] and summarized in Table 1. Given the highly pseudoplastic nature of the investigated mixtures, especially those incorporating RMA and low dosages of high-range water-reducer (HRWR), the accuracy of a given model to fit the experimental shear stress values depends on its ability to properly fit the nonlinear region at low shear rate [3]. Therefore, yield stress values estimated using various analytical models can offer different values. The effectiveness of a new proposed method to estimate the yield stress for highly fluid and pseudoplastic grout was evaluated by comparing estimated yield stress values to those obtained from various rheological models. The deducted values are also compared among each other.

### 2. Proposed log-method approach to estimate yield stress

Cement grouts made with low W/CM, or those incorporating an RMA, exhibit a pseudoplastic behavior where a nonlinear portion of the shear stress-shear rate flow curve is observed at low shear rate. The apparent viscosity is therefore dependent on the shear rate. Three different regions in the flow curve can be observed [5,6]. The first Newtonian region corresponds to very low shear rate and is characterized by a nonlinear shape that is difficult to fit with conventional rheological models. The efficiency of an empirical model to estimate the yield stress depends therefore on the degree of nonlinearity of this portion of the flow curve. The degree of such nonlinearity is related to the mixture proportioning. In general, a nonlinear shape curve is obtained for highly pseudoplastic mixtures made with low W/CM, or those incorporating an RMA and a low concentration of HRWR [3,4].

The decrease in apparent viscosity with the increase of shear rate of the pseudoplastic grout is mainly due to progressive rupture of the interparticle friction bond of the undisturbed structure [7]. This progressive rupture depends on the duration and rate of the shearing process. The breakdown of the structure can be achieved in a short time when high shear rates are applied. However, after a given time of shearing, an equilibrium state is reached or a total breakdown of the structure is achieved, and no further significant drop in apparent viscosity can take place [3,4,7].

The new proposed approach described herein allows to transform the shear stress-shear rate flow curve into a linear shape that eliminates the nonlinear portion. This enables the minimization of error when extrapolating the data to a zero shear rate to determine yield stress. Considering the shearthinning behavior of cement grout, the apparent viscosity can be expected to decrease from an initial value at low shear rate to an equilibrium state at the highest shear rate of interest. Since the apparent viscosity is calculated as the ratio of shear stress to a given shear rate, the equilibrium value of viscosity should correspond to a maximum of shear stress  $(\tau_{max})$  obtained at the highest shear rate. In the case of the viscometer used in this research, the maximum shear rate  $(\dot{\gamma}_{\rm max})$  was set to 1022 s  $^{-1}$ , corresponding to a rotational velocity of 600 rpm. Therefore, for each investigated grout, the maximum shear stress ( $\tau_{max} = 1022 \times \mu_{600}$ ) is determined, as shown in Fig. 1.

The spread between  $\tau_{max}$  and experimental shear stress can be defined by  $\phi(\dot{\gamma})$  which is a function of shear rate. As can be seen in Fig. 1, this function decreases with the

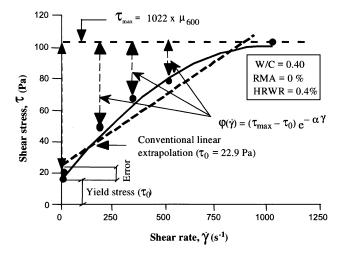


Fig. 1. Flow curve for cement grout made with 0.40 W/CM and 0.4% HRWR.

increase in shear rate. On the other hand, this function must satisfy the boundary conditions given by Eqs. (1) and (2):

$$\varphi(\dot{\gamma}) = \tau_{\text{max}} - \tau_0 \qquad \text{when } \dot{\gamma} = 0 \tag{1}$$

$$\varphi(\dot{\gamma}) = 0 \quad \text{when } \dot{\gamma} \to \infty$$
 (2)

where  $\tau_{max}$  is the maximum shear stress (asymptotical value),  $\tau_0$  is the yield value (Pa), and  $\dot{\gamma}$  is the shear rate (s<sup>-1</sup>). Therefore, the function  $\varphi(\dot{\gamma})$  can be written as (Eq. (3)):

$$\varphi(\dot{\gamma}) = b e^{-\alpha \dot{\gamma}} \tag{3}$$

where b is a constant evaluated using the first boundary condition (Eq. (1)), and is given by (Eq. (4)):

$$\varphi(0) = (\tau_{\text{max}} - \tau_0)e^{-0\alpha} = \tau_{\text{max}} - \tau_0 = b.$$
 (4)

The function  $\varphi(\dot{\gamma})$  can then be expressed as follows (Eq. (5)):

$$\varphi(\dot{\gamma}) = (\tau_{\text{max}} - \tau_0) e^{-\alpha \gamma} \tag{5}$$

 $\alpha$  is a time parameter introduced to eliminate the unit of  $\dot{\gamma}$ . Therefore, the term  $e^{-\alpha\dot{\gamma}}$  takes a constant value. As shown in Fig. 1, for any shear rate value the shear stress can be expressed as the spread between the maximum shear stress and the function  $\varphi(\dot{\gamma})$ .

The evaluation of  $\ln \phi(\dot{\gamma})$  leads to the following expression:

$$\ln\varphi(\dot{\gamma}) = \ln(\tau_{\text{max}} - \tau_0) - \alpha \dot{\gamma} = k - \alpha \dot{\gamma}. \tag{6}$$

The  $\ln \phi(\dot{\gamma}) - \dot{\gamma}$  curve becomes therefore linear in shape, and the parameters  $\alpha$  and k can be evaluated by simple regression fit from that curve (Fig. 2). The  $\alpha$  parameter presents the slope of the curve, and the constant k, represented by  $\ln(\tau_{\rm max} - \tau_0)$ , is the intercept to a zero shear rate. The yield stress is therefore estimated as follows:

$$\tau_0 = \tau_{\text{max}} - e^k \tag{7}$$

As can be seen in Fig. 1, using the conventional linear extrapolation results in a higher yield stress value than that obtained when using a nonlinear response. Such spread can be greater when dealing with highly pseudoplastic

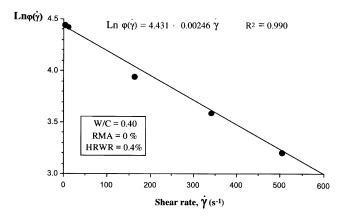


Fig. 2. Representation of the  $\ln \phi(\dot{\gamma})$  versus shear rate.

Table 2 Shear stress data for mixture prepared with 0.40 W/CM and 0.40% HRWR

Shear rate, $\dot{\gamma}$ (s <sup>-1</sup> )	5.1	10.2	170	340	510	1022
Shear stress, τ (Pa)	17.88	21.46	54.67	68.00	80.22	104.8
$\tau_{max} - \tau^a$	86.87	83.29	50.08	36.75	24.53	0
$\ln(\tau_{max} - \tau) = \ln \phi(\dot{\gamma})$	4.464	4.422	3.914	3.604	3.200	_

 $<sup>\</sup>tau_{\text{max}} = 104.8 \text{ Pa.}$ 

grouts, such as mixtures incorporating RMA and low content of HRWR.

For the grout mixture presented in Fig. 2, the rheological measurements are summarized in Table 2 with the maximum shear stress measured at  $1022 \text{ s}^{-1}$  being 104.8 Pa. The relationship between  $\ln \varphi(\gamma)$  versus shear rate is presented in Fig. 2. A simple regression fit is carried out to establish the following relationship (Eq. (8)):

$$\ln\varphi(\dot{\gamma}) = 4.431 - 0.00246\dot{\gamma} \qquad (R^2 = 0.990) \tag{8}$$

Using Eq. (6) the term  $ln(\tau_{max}-\tau_0)$  becomes equal to 4.431, and the yield value can be estimated using Eq. (7) as:  $\tau_0 = \tau_{max} - e^{4.431} = 104.8 - 84.0 = 20.8$  Pa.

#### 3. Experimental program

The experimental program presented in this paper consists of three phases. Phase 1 deals with the rheological properties of cement grout made with 100% of portland cement and various contents of RMA and HRWR. A Type 10 portland cement complying with Canadian Standards CSA-CAN A5 was used. The mixtures were proportioned with four concentrations of RMA of 0%, 0.03%, 0.05%, and 0.075%, by mass of cement. Welan gum was used for the RMA. Such RMA is highly effective in increasing viscosity at low concentration and results in highly pseudoplastic suspensions [4]. For each RMA content, the grout was prepared with various dosages of a naphthalene-based HRWR. The solid content of the HRWR is 42%, and its specific gravity is 1.21. Water present in the HRWR was accounted for to maintain a constant W/CM.

The estimates of yield stress of grout made with silica fume were investigated in Phase 2. The mixtures were prepared with replacement values of silica fume corresponding to 1.5%, 3%, and 5%, by mass of CMs. For each concentration, four grouts made with different HRWR-RMA combinations were prepared. In Phase 3, mixtures were proportioned with blast furnace slag replacements of 20%, 30%, and 40%, by mass of CM. Three combinations of HRWR-RMA were employed for each slag dosage to secure different fluidity levels. All grouts were prepared with a constant W/CM of 0.40 that is typical of high-performance structural grout. A total of 23, 12, and 9 mixtures were evaluated in Phases 1, 2, and 3, respectively. The physical and chemical characteristics of the CMs are summarized in Table 3.

Table 3 Chemical and physical properties of CMs

Chemical analysis	Silica fume	Blast furnace slag	Type 10 cement		
SiO <sub>2</sub>	93.6	36.1	20.8	$C_3S = 59.9$	
$Al_2O_3$	0.3	10.0	4.1	$C_2S = 14.6$	
$Fe_2O_3$	0.5	0.5	3.1	$C_3A = 5.6$	
CaO	0.3	33.9	63.4	$C_3AF = 9.5$	
MgO	0.5	15.4	2.5	Compressive strength (MPa)	
Na <sub>2</sub> O eq.	1.4	0.42	0.77	3d	22.9
C	1.9	_	_	7d	30.0
LOI	2.8	_	1.6	28d	35.9
Specific gravity	2.22	2.88	3.16	Initial setting time	e (min): 145
BET (m <sup>2</sup> /kg)	20250	440	340	Final setting time	(min): 261
Percentage passing 45 μm	100	-	87.4		

All grouts were prepared in batches of 4 1 and mixed using a high-shear mixer with a paddle rotating at 2500 rpm. The mixing procedure consisted of adding the water and HRWR to the mixer along with a welan gum RMA that was prehydrated in a 1% solution. The CM was introduced gradually over 60 s. The grout was then mixed for 60 s, and after a rest period of 30 s, the mixing was resumed for an additional 60 s. The initial temperature of the fresh grouts was maintained at  $23 \pm 3^{\circ}\text{C}$ .

Two coaxial-cylinder viscometers (FANN 35 and CHAN A35) having a gap size of 1.17 mm were employed. The shear stress was determined at 6 and 12 rotation speeds for the FANN and CHAN viscometers, respectively. The shear rates varied from 5.1 to  $1022 \, \mathrm{s}^{-1}$  for the FANN viscometer, and 1.7 to  $1022 \, \mathrm{s}^{-1}$  for the CHAN viscometer. The test procedures and measured rheological properties on the 44 investigated grout mixtures are discussed in Ref. [3]. The discussion in this paper compares the yield stress values estimated given various rheological models. Such values are also compared to those estimated using the proposed logmethod presented above.

#### 4. Test results and discussion

Table 4 summarizes the yield stress values obtained in Phase 1 for mixtures made with 100% cement and different HRWR-RMA combinations. Yield stress values obtained for mixtures incorporating silica fume and blast furnace slag are presented in Tables 5 and 6, respectively. The  $\tau_{max}$ values in each table correspond to the maximum shear stresses measured at 1022 s<sup>-1</sup>, and are used to calculate the yield stress given the proposed log-method. For the application of the Bingham model, the yield stress was estimated assuming a linear model and a second order polynomial response. The latter model is expressed as  $\tau = \tau_0 + \mu_n \dot{\gamma}$  after eliminating the second order value that is insignificant. This expression represents, then, the original Bingham model and provides a better correlation than the linear Bingham model [4]. The Casson, Herschel-Bulkley, De Kee, and authors' models discussed in Ref. [3] were also used to estimate the yield stress given the shear stress-shear rate data of the 44 tested mixtures. Finally, the new proposed log-method was applied

Table 4
Estimated yield stress values obtained for mixtures made with 100% cement

	HRWR (%)	μ <sub>3</sub> (Pa s)	$\tau_{max}$ (Pa)	Estimated yield stress, $\tau_0$ (Pa)						
RMA (%)				Bingham	Binghama	Casson	Herschel-Bulkley	De Kee	Authors	log-method
0	0.4	3.506	104.8	22.9	20.6	16.6	4.9	18.0	10.3	20.7
	0.6	1.602	75.6	9.3	8.6	5.8	7.4	8.3	5.2	7.5
	0.8	0.200	44.0	1.1	1.2	0.3	1.3	1.2	0.2	0.4
	1.0	0.100	38.3	0.7	0.7	0.2	0.3	0.5	0.1	0.1
0.03	0.6	2.104	_	21.0	17.3	15.0	20.9	10.7	10.1	_
	0.8	1.902	101.7	10.0	9.7	5.7	10.3	10.3	7.3	7.4
	1.0	1.402	95.5	7.2	6.8	3.6	6.7	6.9	3.9	5.0
	1.5	0.700	82.8	4.2	3.7	1.8	2.8	3.3	0.3	2.5
0.05	0.8	3.206	136.8	17.0	16.6	11.0	16.5	16.6	_	14.5
	1.0	2.510	136.4	14.3	13.6	8.8	11.7	12.8	7.9	12.1
	1.5	1.706	128.6	10.6	9.5	5.9	4.7	8.4	2.9	8.5
	2.0	1.098	108.8	6.3	5.6	3.0	4.1	5.0	0.8	4.5
0.075	1.0	2.902	142.9	17.5	14.6	10.3	9.4	13.4	4.8	13.8
	1.5	2.304	131.7	12.9	11.3	7.0	10.2	11.3	5.4	8.7
	2.0	1.608	128.6	9.2	8.6	4.6	8.1	8.4	4.1	7.2
	2.5	1.314	118.5	7.6	6.9	3.6	6.4	6.8	2.7	4.7

<sup>&</sup>lt;sup>a</sup> Second order model.

Table 5
Estimated yield values obtained for mixtures containing silica fume

	HRWR-RMA (%)	μ <sub>3</sub> (Pa s)	τ <sub>max</sub> (Pa)	Estimated yield stress, $\tau_0$ (Pa)						
SF (%)				Bingham	Casson	Herschel-Bulkley	De Kee	Authors	log-method	
1.5	0.8-0.05	3.00	91.8	23.8	16.7	10.0	14.1	_	14.6	
	1 - 0.04	2.00	81.6	13.3	9.6	6.9	11.1	6.3	12.3	
	0.8 - 0.03	1.60	61.2	12.3	9.2	6.7	8.1	3.3	10.9	
	1.5 - 0.04	1.00	49.0	8.9	5.9	2.9	6.4	2.1	8.1	
3	0.8 - 0.05	2.90	81.6	19.0	14.9	12.3	16.2	9.6	17.8	
	1 - 0.04	3.80	91.8	32.6	25.3	20.1	20.4	5.7	18.3	
	0.8 - 0.03	3.00	_	20.9	17.6	16.3	17.5	12.0	_	
	1.5 - 0.04	1.20	_	9.4	6.4	3.8	7.4	3.5	_	
5	0.8 - 0.05	3.70	_	39.3	27.0	11.6	15.6	_	_	
	1 - 0.04	3.00	76.5	19.2	16.5	15.4	17.6	_	18.9	
	0.8 - 0.03	3.50	_	30.8	19.9	5.6	17.3	_	_	
	1.5 - 0.04	2.10	_	11.8	10.1	9.3	11.5	_	_	

to estimate the yield value according to the approach presented above.

It is important to note that the Bingham model obtained with the second order polynomial fit did allow suitable estimate of yield stresses for mixtures containing supplementary CMs, especially those made with silica fume [3]. Therefore, the original model given by the linear relationship was used, rather, to fit the experimental shear stress—shear rate data and estimate the yield stress values reported in Tables 4 and 5 for the Bingham model.

The effectiveness of the six rheological models in determining the yield stress is evaluated by considering the relationships between the estimated values with the apparent viscosity measured at low shear rate of 5.1 s<sup>-1</sup> ( $\mu_3$ ). The yield stress defines the limit between the solid and liquid states. Given the way in which this value is estimated — the shear stress corresponding to zero shear rate — this parameter was found to correlate well to the apparent viscosity at low shear rate. This finding was established using a wide variety of grout prepared with W/CM ranging between 0.30 and 0.50 and various silica fume replacements, up to 8% [8]. Based on 210 data points, a relationship was proposed between the yield stress and  $\mu_3$  ( $\tau_0 = -0.19 + 5.5 \mu_3$ , R = .98). Fig. 3 shows the relationships between estimates of yield stress and apparent viscosity obtained at  $5.1 \text{ s}^{-1}$ , as well as the corresponding correlation coefficient (R) for mixtures made with 100% cement. On the other hand, Figs.

4 and 5 report the relationships obtained for mixtures containing silica fume and blast-furnace slag, respectively. Fig. 6 summarizes the relationships obtained for all of the 44 investigated mixtures.

The correlation coefficient is established by considering a power relationship between yield stress and apparent viscosity measured at  $5.1 \text{ s}^{-1}$ . The power relationship offers a useful index of the effectiveness of the analytical model to estimate the yield stress of high-performance cement grout. Such relationship was selected among various possible relationships since it provided the highest R value. The apparent viscosity values from Ref. [3] used to establish the correlation curves are presented in Tables 4-6.

#### 4.1. Yield stress estimates of grouts made with 100% cement

The estimated yield values obtained by extrapolating the shear stress—shear rate data using the six analytical models evaluated in this paper were quite different. In general, it can be said that the linear and modified Bingham models resulted in the highest yield stress values compared with other remaining models for the 44 investigated grouts. It can be observed, on the other hand, that the model proposed by the authors in Ref. [3], the Casson and Herschel—Bulkley models, resulted in the lowest prediction of yield values in all cases. For example, the estimated yield stresses of a mixture prepared with 1% HRWR and 0.05% RMA were

Table 6
Estimated yield values obtained for mixtures containing blast-furnace slag

				Estimated yield stress, $\tau_0$ (Pa)						
Slag (%)	HRWR-RMA (%)	$\mu_3$ (Pa s)	$\tau_{max} \; (Pa)$	Bingham	Casson	Herschel-Bulkley	De Kee	Authors	log-method	
20	0.8-0.03	2.20	71.4	12.3	9.6	_	11.4	7.7	11.8	
	1.5 - 0.05	1.30	40.8	6.8	4.7	5.6	6.2	4.4	6.1	
	0.6 - 0.00	0.60	39.8	4.5	3.0	0.7	3.1	1.2	4.0	
30	1.5 - 0.05	1.50	61.2	9.9	7.2	3.1	7.5	4.5	9.2	
	0.8 - 0.03	0.70	71.4	3.6	1.3	3.8	1.3	1.5	2.7	
	0.6 - 0.00	1.50	61.2	9.4	6.5	4.3	7.6	4.5	8.5	
40	1.5 - 0.05	1.40	_	7.9	5.3	4.1	6.2	3.3	7.0	
	0.8 - 0.03	0.45	71.4	3.2	1.1	2.9	3.0	0.6	2.2	
	0.6 - 0.00	0.52	44.9	2.4	1.2	0.9	1.8	0.1	1.9	

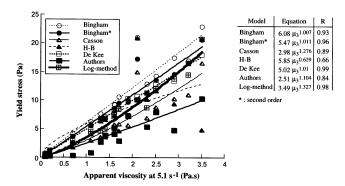


Fig. 3. Relationship between estimates of yield stress and apparent viscosity of mixtures with 100% cement.

14.3 and 13.6 Pa when using the Bingham model to consider the linear and second order responses, respectively. Such values were 7.9, 8.8, 11.7, 12.8, and 12.1 Pa when the models proposed by the authors, Casson, Herschel-Bulkley, De Kee, and log-method were considered, respectively. In general, the log-method resulted in comparable values to those predicted using the De Kee model. For the mixture made with 1.5% HRWR and 0.05% RMA, estimated yield stress values of 8.4 and 8.5 Pa were obtained when the De Kee and log-method models were employed, respectively. However, when dealing with mixtures that possess low viscosity, the yield estimates from the log-method are found to be lower than those estimated by the De Kee model. For the mixture made without any RMA and with 0.6% HRWR having an apparent viscosity at 3 rpm of 1.602 Pa s, the estimated yield values were 8.3 and 7.5 Pa when the De Kee model and log-method were used, respectively. Increasing the HRWR from 0.6% to 0.8% and 1% resulted in lower apparent viscosities of 0.200 and 0.100 Pa s and lower yield stress estimates of 1.2 and 0.5 Pa obtained with the De Kee model. Such values were 0.4 and 0.1 Pa when the logmethod was applied.

The comparison of the yield stress values estimated from the original linear Bingham model and those obtained from the Casson, De Kee, and log-method models showed that for a given HRWR dosage the spread between the estimated values is greater in the case of the more viscous mixtures,

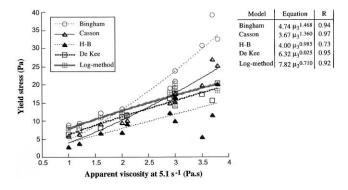


Fig. 4. Relationship between estimates of yield stress and apparent viscosity of mixtures with silica fume.

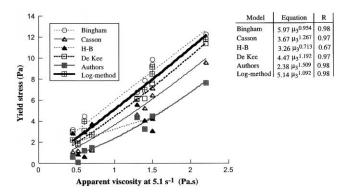


Fig. 5. Relationship between estimates of yield stress and apparent viscosity of mixtures with blast-furnace slag.

such as those containing high RMA dosages. For the grout made with 1% HRWR and no RMA (apparent viscosity of 0.100 Pa s), yield stress values of 0.7 and 0.1 Pa were estimated using the Bingham model and log-method, respectively. However, when dealing with RMA mixtures a great spread was observed. For example, for the grout made with 0.03% RMA and 0.8% HRWR having an apparent viscosity at 5.1 s<sup>-1</sup> of 1.902 Pa s, the yield stress estimated with the Bingham model was 10.0 Pa. This value dropped to 7.4 Pa (2.6 Pa difference) when estimated by the log-method. Increasing the HRWR content to 1.0% and 1.5% resulted in a further drop in yield stress from 7.2 to 5.0 Pa (2.2 Pa spread) and 4.2 to 2.5 Pa (1.7 Pa difference), respectively. By increasing the RMA to 0.075% and 1% HRWR (apparent viscosity of 2.902 Pa s), yield stress values of 17.5 and 13.8 Pa (3.7 Pa difference) were estimated using the Bingham and log-method, respectively. This can be due to the pseudoplastic nature of the RMA grout combined with low HRWR dosage. When dealing with such mixtures, a nonlinear portion of the flow curve is generally observed at low shear rate [3]. Therefore, the effectiveness of a rheological model to estimate the yield stress is mainly related to its accuracy in fitting the experimental shear stress-shear rate data, especially in the nonlinear portion. The method that consists of extrapolating the shear stress values to zero shear rate results, therefore, in estimated values that are highly affected by the degree of pseudoplasticity of the mixture.

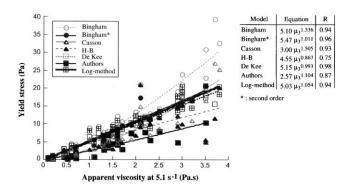


Fig. 6. Relationship between estimates of yield stress and low shear rate viscosity of 44 investigated mixtures.

The use of the original Bingham model resulted in higher estimated values than those obtained with the log-method or the De Kee model. This can be argued by an accurate fitting of the experimental shear stress—shear rate data by eliminating the nonlinear portion of the flow curve, or by using a more complex model.

The regression coefficient values (R) obtained with the new log-method were found to be similar to those secured with the De Kee model, and higher than those estimated using the method presented by the polynomial method in Ref. [3] (modified Bingham model). It can be seen from Fig. 3 that the yield stress values estimated using the De Kee model and log-method correlate well with the apparent viscosity measured at low shear rate for mixtures made with 100% cement (R of .99 and .98, respectively). However, lower R values of .93, .96, .89, .66, and .84 were obtained when using the linear Bingham, modified Bingham, Casson, Herschel-Bulkley, and authors' models, respectively. The worst correlation (R = .66) was obtained when the Herschel-Bulkley model indicates higher dispersion in estimated yield stress. The high correlation coefficients secured with the De Kee model and log-method demonstrate the efficiency of these methods to estimate yield stress for mixtures made with 100% cement and RMA.

The proposed log-method is reliable and can be employed to estimate yield stress. In contrast to a complex analytical model, such as the De Kee model that can result in complicated data treatment, the log-method can be simply adopted to accurately estimate yield stress values of highly pseudoplastic mixtures, such as those incorporating RMA or those with low W/CM.

#### 4.2. Yield stress estimates of mixtures containing silica fume

Yield stress estimates predicted from the various analytical models for the grouts containing 1.5%, 3%, and 5% silica fume replacements were different. In general, the linear Bingham model resulted in higher estimates, while the Herschel-Bulkley model had the lowest values compared to the other models. For example, the yield stress of the grout made with 0.05% RMA, 0.8% HRWR, and 1.5% silica fume was 23.8 Pa when the linear Bingham model was used. This value was 16.7, 10, 14.1, and 14.6 Pa when the Casson, Herschel-Bulkley, De Kee, and log-method models were used, respectively. It can be observed, on the other hand, that the log-method resulted in values comparable to those estimated using the De Kee model. However, it must be noted that the log-method did not allow accurate prediction of yield stress for the more viscous mixtures, such as those made with low HRWR and high RMA contents, as the values of maximum shear stress  $(\tau_{max})$  are not available (missing in Table 5). This can be due to the test procedure adopted during this study. Indeed, for such mixtures, characterized by high consistency, it is suggested that the shearing time of 20 s allowed for each shear rate level during testing was not sufficient to ensure full breakdown of the structure and obtain an equilibrium state. This resulted in higher than expected shear stress values. Given the viscometer in use, the maximum stress,  $\tau_{max}$ , necessary for yield stress prediction with the log-method cannot be accurately determined.

By comparing yield stress values of various mixtures, it can be observed that the spread between the estimated results was higher for the more viscous mixtures. In the case of a grout made with 0.03 RMA, 0.8% HRWR, and 1.5% silica fume having a  $\mu_3$  value of 1.60 Pa s, yield stresses of 12.3 and 9.2 Pa (3.1 Pa difference) were estimated using the Bingham and Casson models, respectively. Increasing the silica fume to 3% ( $\mu_3$  of 3.00 Pa s) and 5% ( $\mu_3$  of 3.50 Pa s) resulted in spread values of 3.3 and 10.9 Pa, respectively. On the other hand, regardless of the silica fume content, the spread between estimated yield stresses obtained from various models increased with the content of RMA. For the mixture made with 1.5% silica fume, 0.8% HRWR, and 0.03% RMA, the estimated yield stress was 12.3 and 9.2 Pa (difference of 3.1 Pa) when the Bingham and Casson models were used, respectively. Increasing the RMA dosage to 0.05% resulted in a higher spread of 7.1 Pa. In the case of 5% silica fume, such spread increased from 10.9 to 12.3 Pa when the RMA dosage increased from 0.03 to 0.05%.

In general, viscous silica fume mixtures, such as those incorporating 3% and 5% silica fume, exhibit high-pseudoplasticity. Therefore, linear models, such as the Bingham model, may not provide accurate fitting of the nonlinear portion of the flow curve at low shear rate. This results in higher error in predicting the yield stress compared to the other nonlinear models. Except for the Herschel–Bulkley model (R = .73), all investigated models resulted in good relationships between the estimated yield stress and  $\mu$  at 5.1 s<sup>-1</sup>, with R values ranging between .92 and .97. The relationships established using the Casson and De Kee models had the highest R values of .97 and .95, respectively. The proposed log-method did not allow accurate prediction of yield values for the relatively viscous silica fume mixtures (R = .92).

# 4.3. Yield stress estimates of grouts containing blast-furnace slag

As can be observed in Table 6 and Fig. 5, mixtures made with blast-furnace slag resulted in lower viscosity than those made with 100% cement and silica fume, and equivalent HRWR dosages. Except yield stress estimates obtained using the Herschel–Bulkley and the authors' models, the remaining models resulted in comparable values. For the grout containing 20% slag, 1.5% HRWR, and 0.05% RMA, estimated yield stress values of 6.8, 6.2, and 6.1 Pa were obtained when using the Bingham, De Kee, and Logmethod models, respectively.

The Herschel-Bulkley and authors' models resulted in lower yield value estimates. As can be seen from Fig. 5, the

spread between the estimates of yield stress given the various rheological models is lower than that observed for the 100% cement and silica fume mixtures. On the other hand, the spread between the yield stress estimates of the blast-furnace slag mixtures is not dependent on the viscosity of the mixture. For example, the grout containing 20% slag, 0.8% HRWR, and 0.03% RMA with  $\mu_3$  of 2.20 Pa s had yield estimates of 12.3 and 11.8 Pa (spread of 0.5 Pa) when using the Bingham model and log-method, respectively. Increasing the HRWR and RMA dosages to 1.5% and 0.05%, respectively, resulted in a mixture with lower viscosity of 1.3 Pa s. For such grout, the spread between the estimated yield stress from the Bingham and log-method was 0.7 Pa. Therefore, for the more fluid slag grouts that behave closer to Newtonian material, the investigated models allowed comparable estimates of yield stress values. On the other hand, such values correlate well to the low shear rate apparent viscosity at 5.1 s<sup>-1</sup> where R values ranging between .97 and .98 were obtained.

By considering all of the investigated mixtures (44 mixtures), it can be observed in Fig. 6 that the De Kee and modified Bingham models resulted in higher *R* values, .98 and .96, respectively, compared to the other models. However, the log-method resulted in a relatively lower *R* value of .94. This can be due to the weakness of such an approach in predicting yield stress of mixtures containing silica fume, especially those with a relatively high consistency.

#### 5. Conclusions

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

- (1) Different estimates of yield value were obtained when using the Bingham, Casson, Herschel-Bulkley, authors', and De Kee rheological models. The limitation of the investigated models to predict yield stress is found to be greater in the case of pseudoplastic mixtures.
- (2) The use of the Bingham model resulted in higher yield stress estimates than the other models, and the Herschel-Bulkley model resulted in the lowest values for all tested mixtures. On the other hand, the log-method and De Kee model resulted, in general, in comparable yield estimates. However, for the more fluid mixtures, the logmethod resulted in lower estimates than those obtained from the De Kee model. The new proposed method did not result

in accurate yield values for the viscous mixtures containing silica fume.

- (3) The model proposed by the authors in Ref. [3] resulted in lower yield stress estimates than those obtained with the De Kee and Casson models.
- (4) The log-method used to predict yield stress of pseudoplastic grout allows the transformation of the flow curve into a linear shape that eliminates the nonlinear portion observed at low shear rates. This approach is shown to result in lower estimated yield values than those obtained with the De Kee and Bingham models. For mixtures containing 100% cement, the proposed approach results in comparable values to those estimated with the De Kee model. The proposed log-method is shown to be more efficient in predicting the yield value, especially for fluid grouts.
- (5) In general, the Casson and De Kee models allowed comparable estimates of yield stress of the silica fume mixtures. In the case of mixtures with blast furnace slag, all six models resulted in comparable estimates with the logmethod and linear Bingham model providing closer estimates.
- (6) The De Kee model resulted in accurate estimates of yield stress for all 44 grouts investigated.
- (7) A power relationship between yield stress and apparent viscosity at low shear rate (5.1 s<sup>-1</sup>) can be used to estimate yield stress.

# References

- C. Atzeni, C. Massida, U. Sanna, Comparison between rheological models for portland cement pastes, Cem. Concr. Res. 15 (1985) 511-519.
- [2] A. Papo, Rheological models for cement pastes, Matér. Const. (Mater. Struct.). 21 (1988) 41–46.
- [3] A. Yahia, K.H. Khayat, Applicability of rheological models to highperformance cement grout containing various supplementary materials and rheology modifying admixtures, Concr. Sci. Eng., in press.
- [4] K.H. Khayat, A. Yahia, Effect of welan gum high-range water reducer combinations on rheology of cement grout, ACI Mater. J. 94 (5) (1997) 365–372.
- [5] G. Couarraze, J.L. Grossiord, Initiation to Rheology, (Available in French) Laboratoire de Physique Pharmaceutique, Centre de Chatenay-Malaby, Université de Paris, second ed., (1991) 272 pp.
- [6] R. Darby, Viscoelastic fluids: an introduction to their properties and behavior, Chem. Process. Eng. 9, (1976) 637 pp.
- [7] G.H. Tattershall, P.F.G. Banfill, The Rheology of Fresh Concrete, Pitman Advanced Publishing Program, New York, 1983.
- [8] K.H. Khayat, A. Yahia, Simple field tests to characterize fluidity and washout resistance of structural cement grout, Cem., Concr., Aggregates, CCAGDP 20 (1) (1998) 145-156.