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Estimating rheological properties of cement pastes using various rheological models for different test geometry, gap and surface friction

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Abstract

Shear stress–shear rate flow tests were carried out on various cement pastes incorporating different mineral additions and chemical admixtures using various test geometries. Different gaps and friction capacity of shearing surfaces of the test geometries were employed in the flow tests. Rheological properties of cement pastes were calculated from the resulting flow curves using various rheological models. The Bingham, Modified Bingham, Herchel–Bulkley and Casson models were used to estimate yield stress. Plastic viscosity was estimated by the Bingham, Modified Bingham and Casson models, while the Williamson and Sisko models were used to estimate the theoretical viscosity at zero and infinite shear rates. It was observed that the rheological properties of cement pastes varied with the change of the test geometries and rheological models used for their calculation. The performance of rheological models in estimating the rheological properties of cement pastes, as expressed by a standard error, varied with the test geometries as well as with the composition of cement pastes. The paper highlights the difficulty in reconciling rheological results from different sources and the need for standardizing rheological test methods for rheological interlaboratory results to be critically analyzed and compared.

Keywords: Cement paste; Rheology; Modeling; Admixture; Rheometer

1. Introduction

Rheological models with capabilities to describe phenomena which occur in a very broad intermediate range between elastic solid and viscous fluid states should be able to predict the deformation of cement paste with reasonable accuracy. A review of rheological models for cement paste was done in Ref. [1]. Generally, deformation characteristics of cement paste are explored using flow tests comprised of shear stress vs. shear rate. From the flow curves, it is also possible to produce a relationship between viscosity and shear rate. Existing time-independent rheological models allow fitting shear stress, shear rate and viscosity data to specific trends. However, no such model is free from statistical error. Usually, it is difficult to compute the shear stress at zero shear rate, called yield stress. This problem can be overcome by using different rheological models capable of predicting yield stress statistically. Using such models, it is also possible to establish a generalized viscosity, called plastic viscosity, for a required range of shear rate. As it is very difficult to create mechanical models for the deformation behavior of cement paste using the apparent viscosity at each shear rate point, plastic viscosity can be very useful in formulating such mechanical models.

Variation of the geometric flow path along with variation of the friction characteristic of shearing surfaces and the gap size between them are necessary to simulate the actual flow of cement paste in fresh concrete. It was shown that rheological properties of cement paste obtained from flow tests vary with variation of the test geometry, gap and friction level of the shearing surfaces when the Bingham model was used [2]. There is currently a lack of information regarding the variation of rheological results of cement pastes for different combinations of rheological models and geometric flow paths with various friction characteristics and gap sizes between the shearing surfaces. Most previous investigations on the performance of different rheological models in obtaining rheological properties of cement pastes were based on coaxial cylinder viscometers with no variation of the friction level or gap size. In the

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present study, the Bingham, Modified Bingham, Herchel–Bulkley, Casson, Sisko and Williamson models were used to investigate the rheological properties of various cement pastes using coaxial cylinders (smooth and vaned) and parallel plates (smooth and serrated). Different gaps between the shearing surfaces of both the smooth and serrated parallel plates were used. Cement pastes with various chemical admixtures and mineral additions were tested. The objective is to highlight the interactions between cement paste mixture composition, flow path, friction level and gap size of the test accessory, and the rheological models used, and how such interactions affect the estimated rheological properties.

2. Rheological models

The shear stress vs. shear rate flow curve of a cement paste will not only depend on its mixture composition, but also on the characteristics of the test accessory used in the flow test [2]. It is difficult to capture, with sufficient accuracy, all possible trends of flow behavior using only a single rheological model [3]. This becomes even more complicated when the flow geometry, gap between shearing surfaces, and their friction capacity also vary. Therefore, various rheological models were used in the present study as listed below:

Bingham model $$\tau = \tau_0 + \mu_p \dot{\gamma} \qquad (1)$$

Herchel – Bulkley model $$\tau = \tau_0 + K\dot{\gamma}^2 \qquad (2)$$

Casson model $$\sqrt{\tau} = \sqrt{\tau_0} + \sqrt{\mu_p}\sqrt{\dot{\gamma}} \qquad (3)$$

Modified Bingham model $$\tau = \tau_0 + \mu_p \dot{\gamma} + c\dot{\gamma}^2 \qquad (4)$$

Sisko model $$\mu = \mu_\infty + K\dot{\gamma}^{n-1} \qquad (5)$$

Williamson model $$\mu = \frac{\mu_0}{1 + (K\dot{\gamma})^n} \qquad (6)$$

In these models, τ, $\dot{\gamma}$, μ, μ_p, μ_∞, μ_0, K, n and c are considered as the shear stress, shear rate, apparent viscosity, plastic viscosity, viscosity at infinite shear rate, viscosity at zero shear rate, consistency, rate index, and a regression constant, respectively. In the remainder of the text, plastic viscosity is called viscosity, and μ_∞ and μ_0 are called Sisko and Williamson viscosity, respectively.

Model (1) was commonly used for rheological investigations on cement paste [4–11], and models (2), (3) and (4) were used in comparative studies of rheological models [3,12,13]. Models (1), (2), (3) and (4) are able to estimate yield stress (τ_0). Plastic viscosity (μ_p) can be estimated by models (1), (3) and (4). Models (5) and (6) are able to calculate viscosity at a theoretically infinite shear rate (μ_∞) and viscosity at a theoretically zero shear rate (μ_0), respectively [13,14].

The various models were fitted to measured flow curves using a rheological data analysis software [15], which also estimated the standard error for the various rheological models using Eq. (7). The standard error is used as a scale for measuring the relative level of accuracy of the different rheological models. The calculation of standard error is based on the standard deviation normalized by the difference between the maximum and minimum measured values multiplied by 1000 as follows:

$$\text{S.E.} = \frac{1000 \times \left[\sum (X_m - X_c)^2 / (n-2)\right]^{1/2}}{\text{Range}}. \qquad (7)$$

Here, X_m = measured value, X_c = calculated value, n = number of data points and Range = maximum value of X_m – minimum value of X_m.

3. Apparatus

To measure rheological properties of cement pastes, flow tests were performed using a high-accuracy rheometer (Fig. 1). The rheometer is capable of continuous shear rate sweep, stress sweep and strain sweep. The capabilities of the device regarding shear rate, shear stress, torque and angular velocity sweep are 0–435 s^{-1}, 0–3618 Pa, 0–2×10^5 μN m and 0–300 rad s^{-1}, respectively. The device keeps the temperature of the specimen constant during the entire time span of the rheological test through a water circulation system around the sample container. To calibrate the apparatus, a certified standard Newtonian oil (PTB 1000A) with nominal viscosity = 1 Pa s and yield stress = 0 Pa at 20 °C was used. A standard cone geometry (diameter = 60 mm, angle = 2°) was employed for the test as per the manufacturer's recommendation. The results of the calibration test were as follows: viscosity = 1.009 Pa s and yield stress = 0 Pa. Thus, calibration was considered acceptable (the manufacturer recommends 4% tolerance). A solvent trap was used to prevent evaporation from the tested cement paste sample by covering the parallel plates and top of the hollow cylinder. This solvent trap had an adequate mechanism to allow rotation of the shaft without any interference.

3.1. Flow geometries

In this study, parallel plates (smooth and serrated) and coaxial cylinders (smooth and vaned) were used to characterize the rheology of cement pastes. For the parallel plates, the cement paste sample was put in between the upper rotable plate and the lower fixed base plate (diam-

Fig. 1. Illustration of the rheometer used (shown with coaxial cylinders).

eter = 40 mm). The distance between the two plates (sample thickness) was adjustable. Three gap thicknesses, namely, 0.5, 0.7 and 1 mm, were used in this investigation. The radius of the inner solid smooth cylinder and vane rotor (at the vaned location) is 14 mm. These smooth inner solid cylinder and vane rotate inside a fixed hollow 15-mm-diameter cylinder. The gap between the inner and outer cylinder, and outer cylinder and vane is 1 mm. The movable test accessories were attached to the driving motor spindle of the rheometer.

3.2. Models for estimating shear stress and shear rate

The shear rate–shear stress data obtained using the various test geometries is based on models built in the software of the computer controlling the rheometer as described elsewhere [2]. A more detailed discussion of models used to describe flow measurements using various test geometries is available in the literature [16,17].

4. Experimental program

4.1. Materials

Mineral additions [slag, silica fume (SF), fly ash (FA)] and ASTM Type-I portland cement (OPC) were used and their mean particle sizes were measured using laser diffraction and are 12.60, 0.16, 16.56 and 13.53 μm, respectively. Welan gum was used as a rheology-modifying admixture (RMA). The superplasticizer (SP) used is a naphthalene sulphonate based with a solid mass fraction of 42%. Cement paste identification can be explained by the following example: 25% FA (w/b = 0.4) means that the paste includes 25% FA, 75% OPC and a water/binder ratio (w/b) of 0.4.

4.2. Mixing and preparing cement pastes

A variable-speed high-shear mixer with a vertical shaft and blades at two vertical levels was used. Cement paste was made with distilled water having a constant temperature of (19 ± 0.5 °C). Mineral admixtures were first dry mixed by hand with OPC. The mixing water was poured into the mixer. Subsequently, cement (or the dry mix of cement and mineral admixtures) was gently added into the mixer over 1 min of mixing at low speed. The mixing was continued for another minute at high speed. Then, the mixer was stopped for 1.5 min. During this time, the sidewall of the mixing container was scrapped with a rubber spatula to recover the material sticking to the container's wall. Mixing resumed for an additional minute at high speed. The cement paste was then kept at rest for 30 s. The sample of cement paste was poured thereafter into the rheometer.

4.3. Preshearing and flow test

The cement paste sample was presheared for 2 min by applying a shear rate sweep from 0 to 70 s^{-1}. Then, the sample was sheared from 0 to 50 s^{-1} within 1 min and 30 s to produce the up-curve of the flow test. After providing an equilibrium time of 15 s, the cement paste sample was sheared from 50 to 0 s^{-1} within 1 min and 30 s to produce the down-curve of the flow test. The preshearing action was intended to cause structural breakdown of the cement paste sample and create uniform conditions before testing. During the preshearing and flow test, the cement paste temperature was maintained at 21 °C. The down-curve was used to calculate the various rheological properties.

5. Analysis of results

5.1. Effect of w/b and mineral admixtures

5.1.1. Yield stress

Fig. 2a and b illustrate the variation of yield stress for cement pastes with various w/b and mineral additions as estimated by different rheological models for the coaxial cylinders and vane rotor flow tests, respectively. Generally, the Modified Bingham model estimated the lowest yield-stress values among all rheological models, probably because this model reflected more the influence of very low shear stresses induced in the instable low shear rate region of the shear stress–shear rate curve. The Herchel–Bulkley model measured the highest yield-stress values among all rheological models for most cement pastes, followed by the Bingham and Casson models, respectively.

Generally, for all rheological models used with coaxial cylinders and vane rotor flow tests, yield stress increased with a reduction of the w/b. The difference between the Bingham and Modified Bingham yield-stress values generally decreased with a reduction of the w/b. The influence of the w/b on the difference between the Bingham and Herchel–Bulkley, and Bingham and Casson yield-stress values did not follow any specific trend. For the 8% SF cement paste, all

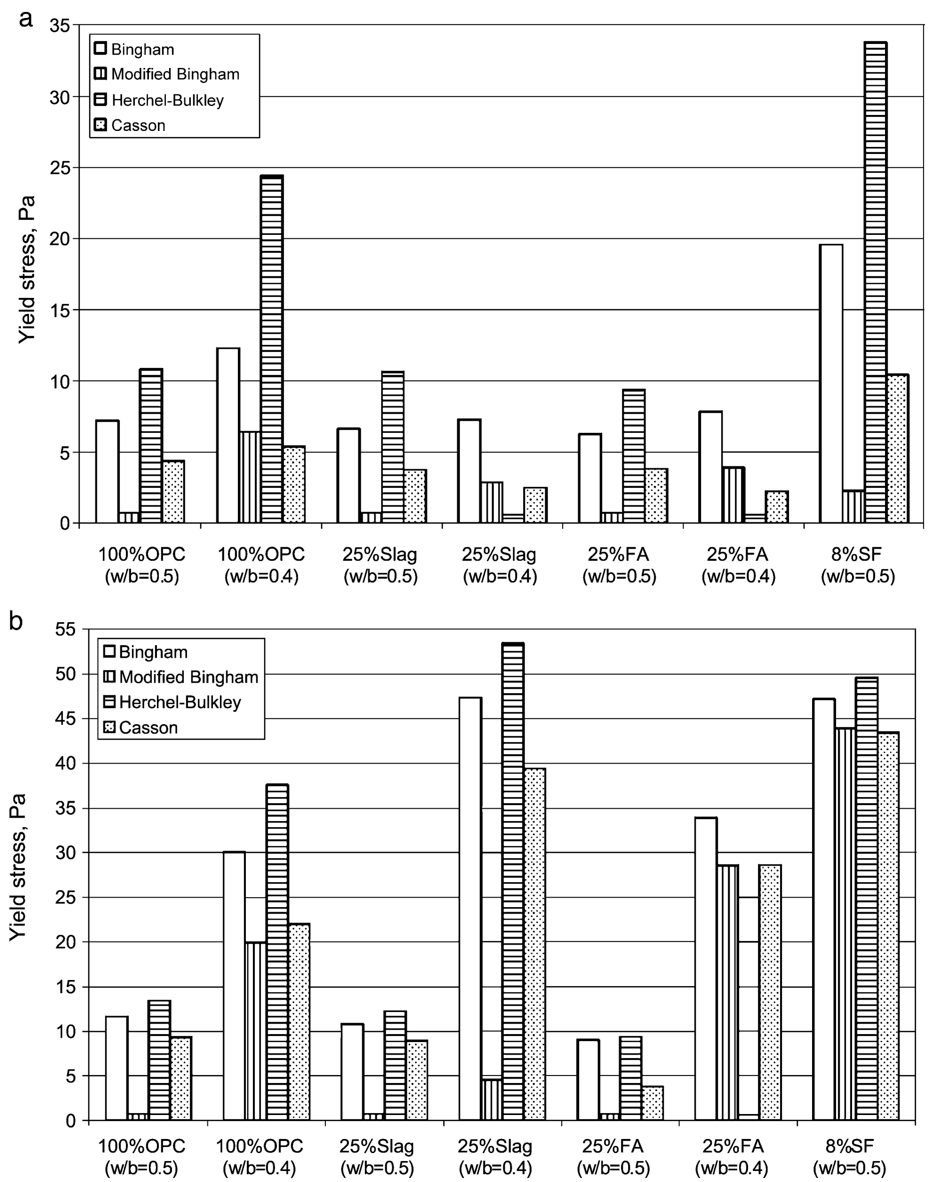

Fig. 2. (a) Yield stress estimated by various rheological models for cement pastes with different w/b and mineral admixtures (tested by coaxial cylinders). (b) Yield stress estimated by various rheological models for cement pastes with different w/b and mineral admixtures (tested by vane rotor).

rheological models estimated comparable values of yield stress when the vane rotor was used. However, there was a significant difference between these values when the coaxial cylinders were used, which suggests that slippage may be an influencing parameter in the case of coaxial cylinders.

Fig. 3a and b illustrate the variation of yield-stress values as estimated by various rheological models for the smooth and serrated parallel plates tests. The Bingham, Herchel–Bulkley and Casson models estimated higher yield-stress values for the serrated plate compared to those of the smooth plate for all cement pastes, while the modified Bingham model did not show such a clear trend and generally estimated comparable yield-stress values for both smooth and serrated plates. Similarly to coaxial cylinders and vane rotor tests, the Herchel–Bulkley model generally estimated highest yield-stress values, followed by the Bingham and Casson models, respectively. The Modified Bingham model estimated the lowest yield-stress values among all rheological models at a w/b of 0.5 for all cement pastes for both smooth and serrated plates. However, at a w/b of 0.4, the Casson model generally estimated the lowest yield-stress values among all rheological models.

The Bingham model estimated higher yield stress due to a reduction of the w/b irrespective of the type of parallel plate or mineral addition used. Generally, for all rheological models, the increase of the yield stress due to a reduction of the w/b was more significant for the serrated plate tests compared to that of the smooth plate tests, indicating that slippage may have offset the effect of the w/b in the case of the smooth plate. For the serrated plate, the influence of the

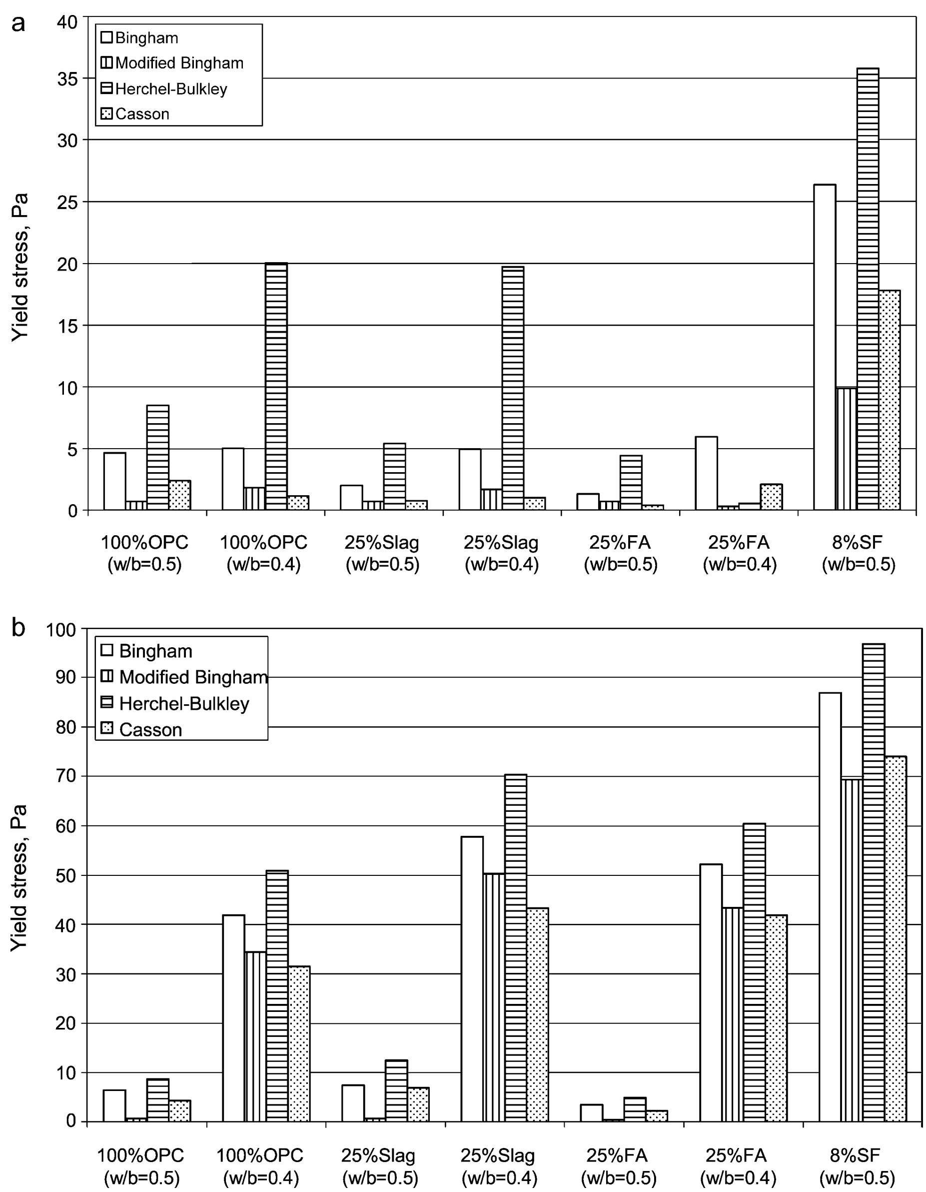

Fig. 3. (a) Yield stress estimated by various rheological models for cement pastes with different w/b and mineral admixtures (tested by smooth plate). (b) Yield stress estimated by various rheological models for cement pastes with different w/b and mineral admixtures (tested by serrated plate).

reduction of the w/b on increasing the yield stress was most significant for the Modified Bingham model regardless of the mineral addition used, while it was least significant for the Herchel–Bulkley model. Such a behavior was not observed for the smooth plate.

5.1.2. Viscosity

Table 1a summarizes plastic viscosity data estimated by the Bingham, Modified Bingham and Casson models, and the viscosity at zero and infinite shear rate estimated by the Williamson and Sisko models, respectively, for various cement pastes tested using the coaxial cylinders and vane rotor. For both of these test geometries, the Modified Bingham model estimated the highest plastic viscosity values followed by the Bingham and Casson models, respectively, for all cement pastes. Generally, for all three models, a reduction of the w/b resulted in higher plastic viscosity, and this effect was highest for the 100% OPC paste, while it was lowest for the 25% FA cement.

The difference between the Bingham and Modified Bingham, and between the Bingham and Casson plastic viscosity values decreased with a reduction of the w/b for all cement pastes tested by the coaxial cylinders and vane rotor, while the 25% slag cement paste (tested by the vane rotor) showed an opposite trend. Reducing the w/b increased viscosity at zero shear rate as estimated by the Williamson model more significantly for the vane rotor tests than for coaxial cylinders tests. For the coaxial cylinders, the viscosity at an infinite shear rate as estimated by the Sisko model consistently increased with a reduction of the w/b, while no clear trend was observed for the vane rotor.

Table 1b lists the plastic viscosity values estimated by the Bingham, Modified Bingham and Casson models, and the viscosity at zero and infinite shear rate estimated by the Williamson and Sisko models, respectively for various cement pastes tested using the smooth and serrated parallel plates. For all cement pastes, the Modified Bingham model estimated the highest plastic viscosity values followed by the Bingham and Casson models, which is similar to the case of the coaxial cylinders and vane rotor discussed earlier. Again, the difference between the Bingham and Modified Bingham plastic viscosity values decreased with a reduction of the w/b for both the smooth and serrated parallel plates. However, the influence of reducing the w/b played an opposite role on the difference of plastic viscosity values estimated by the Bingham and Casson models for the serrated plate tests. For the smooth plate, this difference generally decreased with a reduction of the w/b. The results

Table 1
Viscosity estimated by various rheological models for cement pastes with different w/b and mineral admixtures

Test geometry	Cement paste	Bingham	Modified Bingham	Casson	Sisko	Williamson
(a) Tested by coaxial cylinders and vane rotor						
Coaxial cylinders	100% OPC (w/b = 0.5)	0.34	0.76	0.15	2.62e − 08	2.00
	100% OPC (w/b = 0.4)	1.28	2.05	0.79	1.86e − 07	4.50
	25% Slag (w/b = 0.5)	0.38	0.76	0.18	7.85e − 08	2.11
	25% Slag (w/b = 0.4)	1.21	1.78	0.86	6.01e − 07	5.09
	25% FA (w/b = 0.5)	0.29	0.76	0.13	8.96e − 07	1.39
	25% FA (w/b = 0.4)	0.91	1.43	0.59	2.22e − 06	2.63
	8% SF (w/b = 0.5)	1.37	3.51	0.72	4.76e − 06	1697.00
Vane rotor	100% OPC (w/b = 0.5)	0.16	0.76	0.03	1.49e − 05	15.87
	100% OPC (w/b = 0.4)	0.69	2.09	0.21	5.95e − 07	245.00
	25% Slag (w/b = 0.5)	0.13	0.76	0.03	1.92e − 08	37.90
	25% Slag (w/b = 0.4)	0.57	4.51	0.11	2.41e − 06	2000.00
	25% FA (w/b = 0.5)	0.14	0.76	0.13	8.95e − 07	1.39
	25% FA (w/b = 0.4)	0.35	1.04	0.06	1.48e − 08	478.60
	8% SF (w/b = 0.5)	0.22	0.64	0.02	3.65e − 06	1072.00
(b) Tested by smooth and serrated plates						
Smooth plate	100% OPC (w/b = 0.5)	0.37	0.76	0.19	6.50e − 07	0.80
	100% OPC (w/b = 0.4)	1.62	2.13	1.32	1.68	3465.00
	25% Slag (w/b = 0.5)	0.34	0.76	0.23	5.10e − 06	0.48
	25% Slag (w/b = 0.4)	1.61	2.05	1.33	1.58	3834.00
	25% FA (w/b = 0.5)	0.32	0.76	0.24	4.56e − 06	1119.00
	25% FA (w/b = 0.4)	1.05	1.85	0.74	3.10e − 01	0.82
	8% SF (w/b = 0.5)	0.80	2.91	0.29	1.20e − 05	40.58
Serrated plate	100% OPC (w/b = 0.5)	0.22	0.76	0.08	8.70e − 07	18.09
	100% OPC (w/b = 0.4)	0.90	1.86	0.25	0.25	3.16e + 05
	25% Slag (w/b = 0.5)	0.23	0.76	0.09	3.82e − 06	23.72
	25% Slag (w/b = 0.4)	1.29	2.27	0.37	0.62	1.18e + 06
	25% FA (w/b = 0.5)	0.14	0.46	0.06	2.27e − 08	6.68
	25% FA (w/b = 0.4)	0.80	1.93	0.18	0.08	6.45e + 05
	8% SF (w/b = 0.5)	0.84	3.11	0.14	3.91e − 06	1.28e + 06

above reflect the dependence of the calculated plastic viscosity data not only on the mixture composition of the cement paste, but also on the test accessory used in the rheological test and the model used in calculating viscosity from the resulting flow curve.

5.1.3. Standard error

Table 2a shows the standard error values calculated by various rheological models for cement pastes tested using the coaxial cylinders and vane rotor. Standard error values of the Bingham, Modified Bingham, Casson and Sisko models decreased with a reduction of the w/b for all cement pastes tested by both the coaxial cylinders and vane rotor. In addition, flow curves of cement pastes with higher yield stress seemed to fit better with the various rheological models compared to those with lower yield stress. Conversely, the standard error for the Herchel–Bulkley model either increased or decreased with a reduction of the w/b depending on the type of mineral addition used. Table 2b lists the standard error values of the various models for smooth and serrated parallel plates flow tests. For the smooth plate, the Modified Bingham model showed lowest standard error compared to that of the other yield-stress-producing rheological models for all cement pastes. For the serrated plate, the Casson model generally proved to be the rheological model with lowest standard error in estimating yield stress for all cement pastes.

5.1.4. Consistency and rate index

The parameter denoted by K in the Williamson, Sisko and Herchel–Bulkley models, multiplied by the variable shear rate $\dot{\gamma}$, is termed as *consistency*, while the power of the same variable is termed as rate index for the Williamson and Sisko models. Using consistency and rate index values, it is possible to predict the trend of viscosity vs. shear rate data for the Sisko and Williamson models, and shear stress vs. shear rate data for the Herchel–Bulkley model. Consistency and rate index values estimated by various rheological models for cement pastes with different w/b and mineral admixtures for the various flow geometries are shown in Table 3. Consistency values increased with a reduction of the w/b for all cement pastes irrespective of the test geometry used and rheological model considered. Rate index values decreased with a reduction of the w/b for the Williamson model when cement pastes were tested using the coaxial cylinders, vane rotor and smooth plate. A similar trend was observed for the Sisko model with both the smooth and serrated parallel plates.

Table 2
Standard error estimated by various rheological models for cement pastes with different w/b and mineral admixtures

Test geometry	Cement paste	Bingham	Modified Bingham	Herchel–Bulkley	Casson	Sisko	Williamson
(a) Tested by coaxial cylinders and vane rotor							
Coaxial cylinders	100% OPC (wb = 0.5)	122.90	131.00	178.20	93.43	115.40	24.03
	100% OPC (wb = 0.4)	45.60	7.96	116.50	24.31	16.23	34.18
	25% Slag (wb = 0.5)	111.90	114.80	173.40	84.09	90.43	30.01
	25% Slag (wb = 0.4)	40.83	17.70	233.50	24.40	45.44	35.37
	25% FA (wb = 0.5)	131.90	102.90	187.60	96.92	156.50	22.84
	25% FA (wb = 0.4)	49.65	27.10	257.50	29.12	140.10	61.24
	8% SF (w/b = 0.5)	109.90	21.33	179.00	84.79	44.62	44.60
Vane rotor	100% OPC (wb = 0.5)	115.10	259.30	140.00	92.05	30.38	7.03
	100% OPC (wb = 0.4)	102.60	40.67	144.61	74.16	16.36	15.90
	25% Slag (wb = 0.5)	78.21	237.40	101.80	57.45	12.28	3.04
	25% Slag (wb = 0.4)	65.09	192.70	92.38	42.23	4.56	4.56
	25% FA (wb = 0.5)	131.90	96.92	187.60	102.90	156.50	22.84
	25% FA (wb = 0.4)	70.41	39.99	561.00	49.92	7.11	6.48
	8% SF (w/b = 0.5)	43.38	31.68	54.24	32.31	4.42	3.85
(b) Tested by smooth and serrated parallel plates							
Smooth plate	100% OPC (wb = 0.5)	128.60	68.42	197.50	103.40	255.30	110.50
	100% OPC (wb = 0.4)	37.77	16.22	118.20	29.83	49.95	80.70
	25% Slag (wb = 0.5)	103.10	97.75	181.40	88.11	280.20	154.10
	25% Slag (wb = 0.4)	28.14	13.42	106.00	21.95	28.72	71.49
	25% FA (wb = 0.5)	76.05	0.76	153.60	66.29	96.72	102.00
	25% FA (wb = 0.4)	65.09	10.67	260.30	49.98	131.30	76.60
	8% SF (w/b = 0.5)	170.90	71.73	218.80	141.30	65.36	64.12
Serrated plate	100% OPC (wb = 0.5)	84.66	98.32	132.80	55.55	14.34	7.83
	100% OPC (wb = 0.4)	55.05	25.54	94.78	28.49	3.37	6.72
	25% Slag (wb = 0.5)	96.60	187.80	140.60	67.67	16.97	10.12
	25% Slag (wb = 0.4)	40.96	22.88	79.87	14.99	1.65	10.90
	25% FA (wb = 0.5)	93.41	83.54	143.10	66.80	25.89	21.70
	25% FA (wb = 0.4)	59.35	23.59	92.62	15.18	4.92	5.23
	8% SF (w/b = 0.5)	77.95	17.09	102.40	60.67	14.86	14.86

Table 3
Consistency and rate index estimated by various rheological models for cement pastes with different w/b and mineral admixtures tested by the smooth and serrated parallel plates, coaxial cylinders and vane rotor

Rheological models	Cement paste	Coaxial cylinders		Vane rotor		Smooth plate		Serrated plate	
		Consistency	Rate index	Consistency	Rate index	Consistency	Rate index	Consistency	Rate index
Sisko	100% OPC (w/b = 0.5)	2.29	0.62	7.52	0.24	0.88	0.88	3.89	0.37
	100% OPC (w/b = 0.4)	7.28	0.36	19.57	0.29	1.92	0.25	32.81	0.19
	25% Slag (w/b = 0.5)	2.25	0.64	8.20	0.18	0.49	0.96	6.00	0.33
	25% Slag (w/b = 0.4)	3.31	0.77	38.69	0.16	2.69	0.27	47.77	0.15
	25% FA (w/b = 0.5)	1.84	0.64	1.84	0.64	0.33	0.86	1.91	0.43
	25% FA (w/b = 0.4)	2.82	0.76	27.27	0.15	2.11	0.53	41.60	0.18
	8% SF (w/b = 0.5)	8.99	0.57	42.59	0.07	11.47	0.46	71.80	0.14
Williamson	100% OPC (w/b = 0.5)	0.07	1.08	1.19	0.92	0.02	2.54	6.46	0.70
	100% OPC (w/b = 0.4)	0.51	0.45	28.68	0.73	6.37e + 15	0.19	1.52e + 05	0.77
	25% Slag (w/b = 0.5)	0.08	0.90	4.34	0.88	0.02	4.45	4.35	0.75
	25% Slag (w/b = 0.4)	0.29	0.41	38.69	0.84	9.57e + 12	0.23	4.61e + 05	0.78
	25% FA (w/b = 0.5)	0.05	1.34	0.05	1.34	0.02	1.30	4.25	0.65
	25% FA (w/b = 0.4)	0.04	0.89	25.63	0.87	1.25e + 14	0.16	6.45e + 05	0.81
	8% SF (w/b = 0.5)	4.19e + 07	0.43	29.61	0.94	4.48	0.63	85140	0.86
Herchel–Bulkley	100% OPC (w/b = 0.5)	5.79e − 03		2.54e − 03		6.34e − 03		3.83e − 03	
	100% OPC (w/b = 0.4)	0.02		0.01		0.03		0.02	
	25% Slag (w/b = 0.5)	6.61e − 03		2.29e − 03		6.04e − 03		4.44e − 03	
	25% Slag (w/b = 0.4)	0.04		9.70e − 03		0.03		0.02	
	25% FA (w/b = 0.5)	4.84e − 03		8.48e − 03		5.89e − 03		2.47e − 03	
	25% FA (w/b = 0.4)	0.03		0.03		0.03		0.01	
	8% SF (w/b = 0.5)	0.02		3.76e − 03		0.01		0.01	

5.2. Effect of RMA

5.2.1. Yield stress

Fig. 4 shows the yield-stress values estimated by different rheological models for cement pastes incorporating various dosages of an RMA and tested using coaxial cylinders. Generally, the yield stress estimated by each model increased with an increase of the RMA dosage. The Modified Bingham model estimated the lowest yield-stress values for the cement pastes at an RMA dosage in the 0–0.03% range. However, the Casson model estimated the lowest yield-stress values when the dosage of the RMA was 0.05%. The Herchel–Bulkley model calculated the highest yield-stress values for all cement pastes irrespective of the RMA dosage. The increase of the RMA dosage in the range 0–0.05% was most effective in increasing the yield-stress values obtained by the Modified Bingham model compared to that of the other models.

Fig. 5a and b illustrate yield-stress data for cement pastes incorporating various dosages of a RMA as estimated by

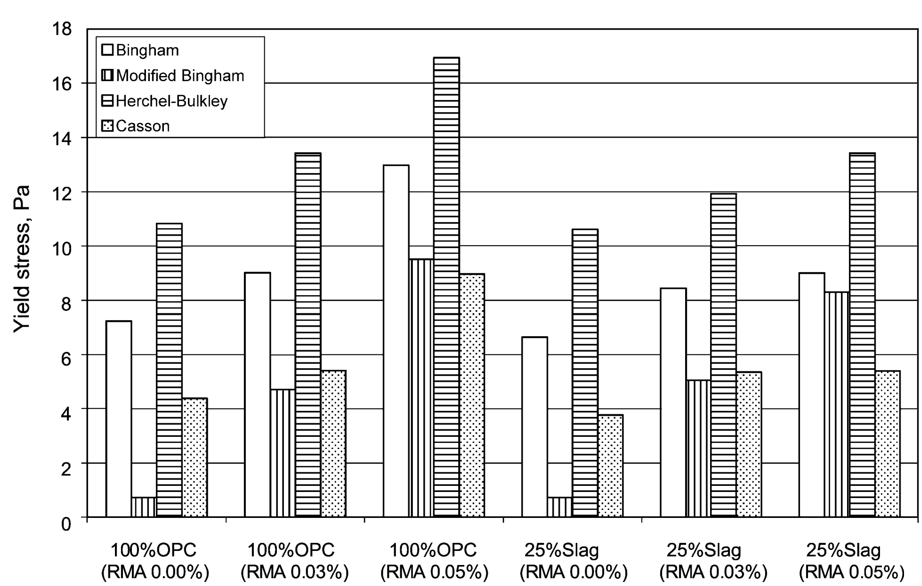

Fig. 4. Yield stress estimated by various rheological models for cement pastes with different w/b and RMA dosages (tested by coaxial cylinders).

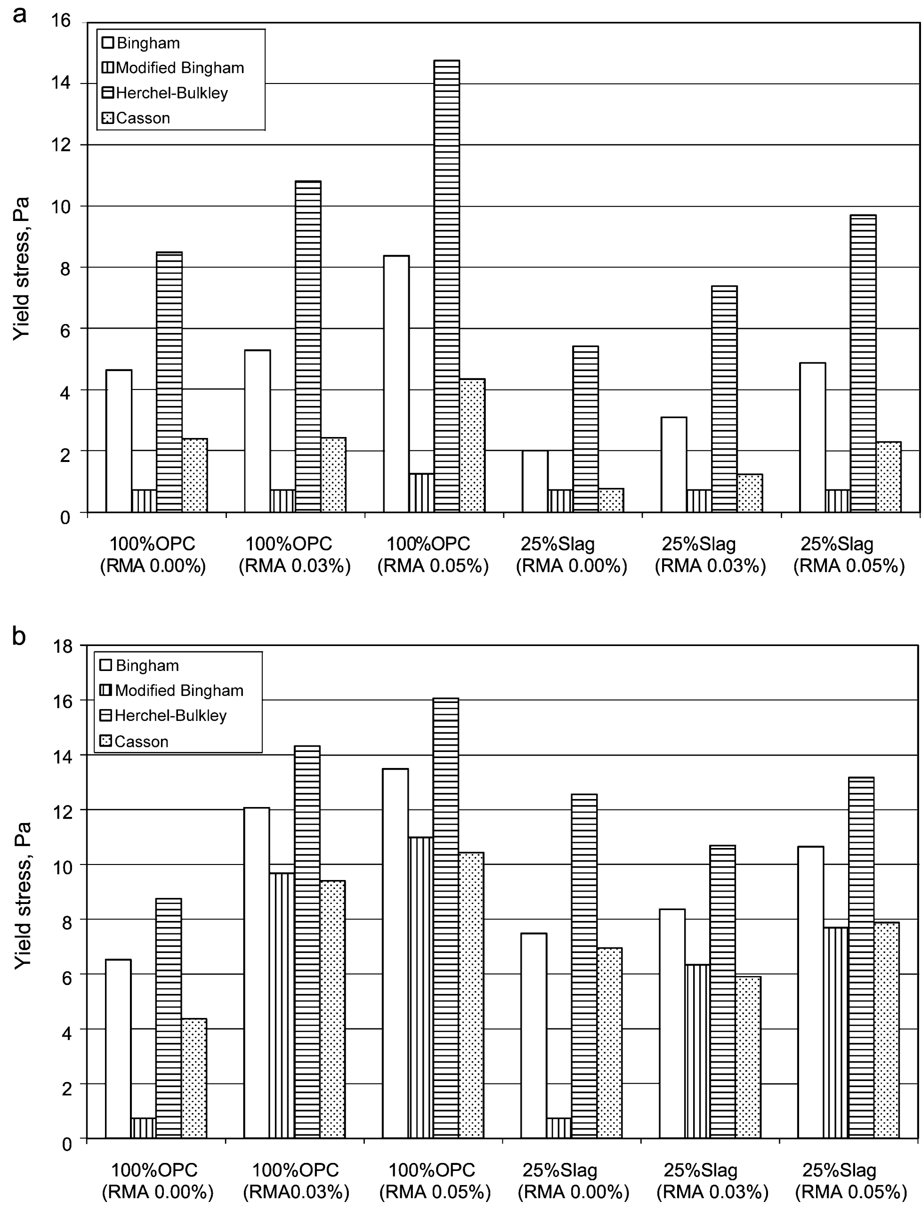

Fig. 5. (a) Yield stress estimated by various rheological models for cement pastes tested by the smooth plate with different RMA dosages at w/b = 0.5 (tested by smooth parallel plates). (b) Yield stress estimated by various rheological models for cement pastes with different RMA dosages w/b = 0.5 (tested by serrated parallel plates).

different models for rheological tests carried out using the smooth and serrated parallel plates, respectively. For the smooth plate, the Modified Bingham model estimated lowest yield-stress values for all cement pastes and did not exhibit sensitivity to change in the RMA dosage. Generally, yield stress increased with an increase of the RMA dosage for the other rheological models. For the serrated plate with RMA addition, the Modified Bingham and Casson models gave comparable yield-stress values. In addition, similar to the coaxial cylinders, the Herchel–Bulkley model estimated the highest yield-stress values for both the smooth and serrated plates, followed by the Bingham and Casson models, respectively.

5.2.2. Viscosity

Table 4a and b show viscosity values calculated by various rheological models for cement pastes incorporating various dosages of an RMA and tested using coaxial cylinders and parallel plates (smooth and serrated). For coaxial cylinders, plastic viscosity values generally increased with an increase of the RMA dosage for the Bingham and Casson models. A similar trend was observed for the theoretical viscosity values at infinite and zero shear rates estimated by the Sisko and Williamson models, respectively. For the Modified Bingham model, an intermediate dosage of RMA (0.03%) gave peak viscosity values. The effect of the RMA on the viscosity estimated by various

Table 4
Viscosity estimated by various rheological models for 100% OPC and 25% slag cement pastes with w/b = 0.5 and various dosages of RMA

Test geometry	Cement paste	Bingham	Modified Bingham	Casson	Sisko	Williamson
(a) Tested by coaxial cylinders						
Coaxial cylinders	100% OPC (RMA 0.00%)	0.33	0.76	0.15	2.62e − 08	2.00
	100% OPC (RMA 0.03%)	0.43	0.99	0.19	0.15	2.25e + 04
	100% OPC (RMA 0.05%)	0.45	0.84	0.24	0.22	1.54e + 05
	25% Slag (RMA 0.00%)	0.38	0.76	0.18	7.85e − 08	2.11
	25% Slag (RMA 0.03%)	0.43	0.78	0.14	1.59e − 03	1.65e + 04
	25% Slag (RMA 0.05%)	0.44	0.76	0.20	0.27	1.29e + 05
(b) Tested by smooth and serrated parallel plates						
Smooth plate	100% OPC (RMA 0.00%)	0.37	0.76	0.19	6.50e − 07	0.80
	100% OPC (RMA 0.03%)	0.54	0.76	0.33	1.56e − 07	1.71
	100% OPC (RMA 0.05%)	0.62	1.51	0.33	2.41e − 06	12.81
	25% Slag (RMA 0.00%)	0.34	0.76	0.23	5.10e − 06	0.48
	25% Slag (RMA 0.03%)	0.44	0.76	0.29	7.75e − 08	1.51
	25% Slag (RMA 0.05%)	0.48	0.76	0.28	1.99e − 07	1.22
Serrated plate	100% OPC (RMA 0.00%)	0.21	0.76	0.08	8.70e − 07	18.09
	100% OPC (RMA 0.03%)	0.22	0.53	0.05	0.01	1.00e + 05
	100% OPC (RMA 0.05%)	0.25	0.57	0.06	0.04	1.65e + 05
	25% Slag (RMA 0.00%)	0.23	0.76	0.08	3.82e − 06	23.72
	25% Slag (RMA 0.03%)	0.23	0.49	0.08	9.53e − 07	589.40
	25% Slag (RMA 0.05%)	0.24	0.62	0.07	5.76e − 07	49.57

rheological models for parallel plates tests did not have a clear trend. For all RMA dosages, the Modified Bingham model gave highest plastic viscosity values, followed by the Bingham and Casson models, respectively. Generally, the Sisko model estimated very low values of viscosity at infinite shear rate as expected, and an RMA dosage of 0.03% appeared to give a peak values for the viscosity at zero shear rate estimated by the Williamson model.

5.2.3. Standard error

Table 5 summarizes the standard error values calculated by various rheological models for fitting flow curves obtained using coaxial cylinders and parallel plates (smooth and serrated) for cement pastes incorporating various dosages of a RMA. For coaxial cylinders, there was a general tendency for the standard error to decrease with increasing RMA dosage for all rheological models, except for the Modified Bingham and Williamson models. For the smooth parallel plates, the trend was that lowest standard error values were observed at the intermediate RMA dosage of 0.03% with a number of exceptions. For the serrated plate, the standard error decreased with higher RMA dosage for all rheological models for the 100% OPC paste, while for the 25% slag

Table 5
Standard error estimated by various rheological models for 100% OPC and 25% slag cement pastes with a w/b = 0.5 and various dosages of RMA

Test geometry	Cement paste	Bingham	Modified Bingham	Herchel–Bulkley	Casson	Sisko	Williamson
Coaxial cylinders	100% OPC (RMA 0.00%)	122.90	131.00	178.20	93.43	115.40	24.03
	100% OPC (RMA 0.03%)	87.37	20.99	147.00	60.95	20.04	26.87
	100% OPC (RMA 0.05%)	66.49	25.03	114.70	37.26	8.87	11.39
	25% Slag (RMA 0.00%)	111.90	114.80	173.40	84.09	90.43	30.01
	25% Slag (RMA 0.03%)	84.18	30.25	138.40	55.36	22.20	18.25
	25% Slag (RMA 0.05%)	76.41	159.70	136.70	50.28	18.20	29.41
Smooth plate	100% OPC (RMA 0.00%)	128.60	68.42	197.50	103.40	255.30	110.50
	100% OPC (RMA 0.03%)	97.78	108.00	172.30	76.27	87.60	68.72
	100% OPC (RMA 0.05%)	107.70	13.72	178.20	82.64	47.69	46.64
	25% Slag (RMA 0.00%)	103.10	97.75	181.40	88.11	280.20	154.10
	25% Slag (RMA 0.03%)	77.24	16.61	154.90	58.89	84.57	79.56
	25% Slag (RMA 0.05%)	99.75	83.79	173.20	76.96	122.00	49.36
Serrated plate	100% OPC (RMA 0.00%)	84.66	98.32	132.80	55.55	14.34	7.83
	100% OPC (RMA 0.03%)	65.67	25.45	102.40	39.58	5.20	5.32
	100% OPC (RMA 0.05%)	60.17	25.82	97.22	33.78	3.78	4.92
	25% Slag (RMA 0.00%)	96.60	187.80	140.60	67.67	16.97	10.12
	25% Slag (RMA 0.03%)	66.90	32.97	111.00	38.71	1.92	1.87
	25% Slag (RMA 0.05%)	59.31	45.94	130.20	60.82	10.27	3.63

paste, the intermediate dosage of RMA gave lowest standard error.

5.3. Effect of gap between shearing surfaces

5.3.1. Yield stress

Fig. 6a and b illustrate yield-stress values for the 25% slag (w/b=0.5) and 8% SF (w/b=0.3, SP=0.75%) cement pastes estimated by various rheological models for different gaps between the shearing surfaces of the smooth and serrated parallel plates. For smooth parallel plate tests, yield-stress values tended to decrease with increasing gap between the plates for all rheological models except for the modified Bingham model, which was not sensitive to change in the gap. For the serrated plate tests, yield-stress values tended to reach a maximum value at the intermediate gap of 0.7 mm for the 25% slag paste, and this was most dramatic for the Modified Bingham model. However, for the 8% SF paste, the intermediate gap of 0.7 mm provided lower yield stress.

5.3.2. Viscosity

Table 6a shows viscosity values calculated by different rheological models with variation of the gap between the

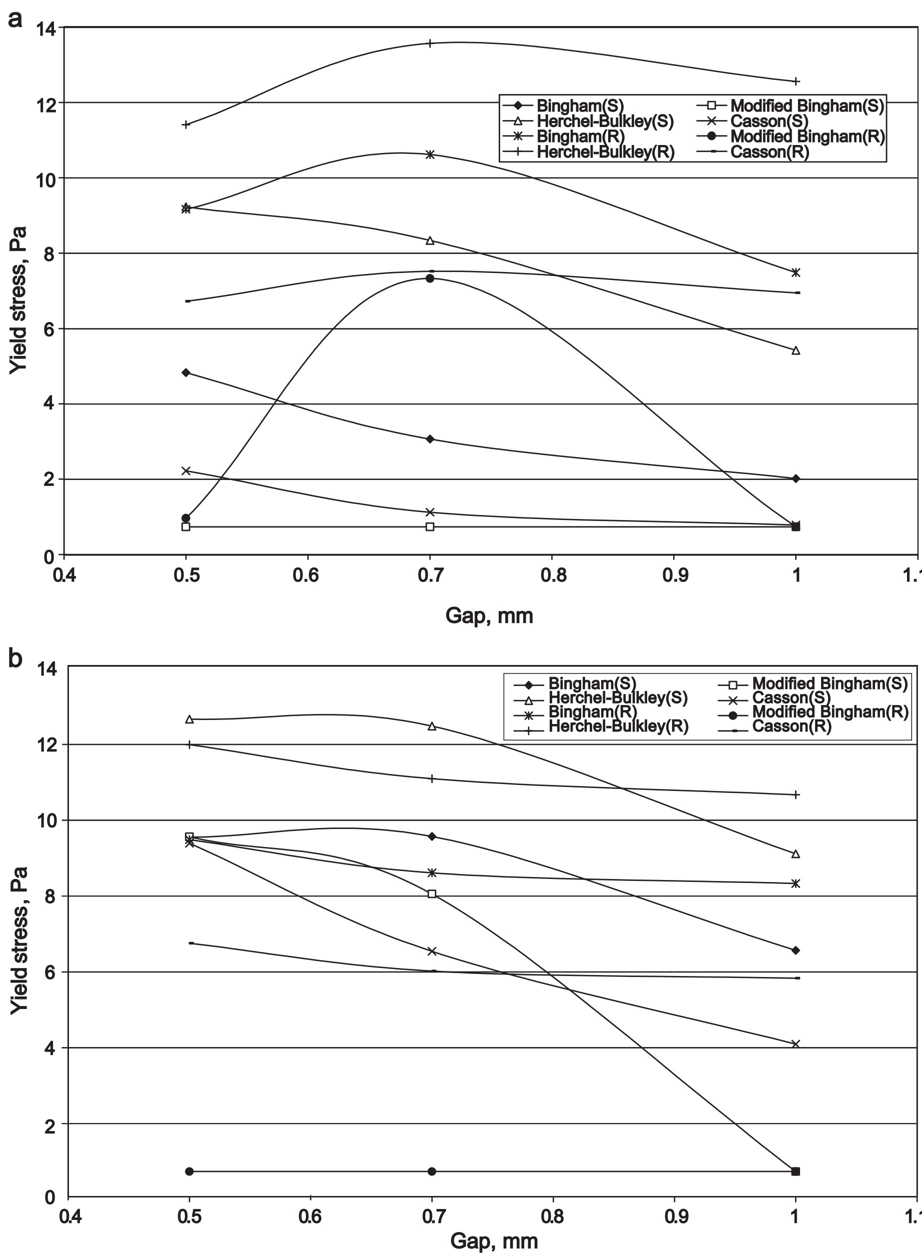

Fig. 6. (a) Effect of gap between smooth and serrated parallel plates on yield stress of 25% slag (w/b = 0.5) cement paste as estimated by various rheological models (S = smooth, R = serrated). (b). Effect of gap between smooth and serrated parallel plates on yield stress of 8%-SF (w/b = 0.3, SP = 0.75%) cement paste as estimated by various rheological models (S = smooth, R = serrated).

shearing surfaces of the smooth and serrated parallel plates. For both smooth and serrated parallel plate tests, the viscosity estimated by the Bingham and Casson models tended to decrease with increasing gap between the parallel plates for the 8% SF cement paste, while an intermediate gap of 0.7 mm provided highest viscosity for the 25% slag cement paste. Generally, the viscosity estimated by the Modified Bingham model was not sensitive to change in the gap between parallel plates, while that of the Sisko and Williamson models tended to increase with higher gap, with the exception of the 25% slag paste tested by the smooth plate.

5.3.3. Standard error

Table 6b shows the standard error values resulting from fitting various rheological models to flow curves of cement pastes tested using smooth and serrated parallel plates at different gaps between their shearing surfaces. Generally, for a given gap between the shearing surfaces of parallel plates, the Casson model showed lower standard error values in estimating yield stress compared to that of the other models (Bingham, Modified Bingham and Hershel–Buckley). The standard error for the Bingham model, both for smooth and serrated parallel plates tests, decreased with an increase of the gap for the SF cement paste, while an opposite trend was observed for the slag cement paste. There was a general tendency for the Modified Bingham model to give a lower standard error at an intermediate gap of 0.7 mm with both smooth and serrated parallel plates tests. The standard error for the Casson model decreased with increasing gap for the SF cement paste, while no clear trend was observed for the slag cement paste. There was a general trend for the standard error to increase with increasing gap in estimating viscosity at zero shear rate by the Williamson model, while the Sisko model did not exhibit a consistent trend in estimating viscosity at infinite shear rate; both models had relatively low standard errors. It is interesting to note that while the standard error of most models was higher for smooth plate tests compared to that for serrated plate tests (with a few exceptions generally involving the 25% slag cement paste at low gap), the Modified Bingham model had higher standard error values for serrated plate tests.

6. Discussion

Differences in measured rheological results for cement pastes reported in the present study originate from three

Table 6
Viscosity and standard error of 8% SF (w/b = 0.3, SP = 0.75%) and 25% slag (w/b = 0.5) cement pastes estimated by various rheological models for different gaps between the shearing surfaces of the smooth and serrated parallel plates

(a) Viscosity

Cement paste and geometry	Gap (mm)	Bingham	Modified Bingham	Casson	Sisko	Williamson
8% SF (smooth)	0.50	0.32	0.76	0.12	1.19e − 06	50.99
	0.70	0.31	0.50	0.11	0.05	1.80e + 03
	1.00	0.27	0.76	0.11	0.12	5.38e + 04
8% SF (serrated)	0.50	0.26	0.76	6.75	0.07	7.00e + 04
	0.70	0.26	0.76	6.02	0.10	1.50e + 05
	1.00	0.25	0.76	0.09	0.12	1.32e + 05
25% Slag (smooth)	0.50	0.45	0.76	0.27	3.00e − 06	4.71
	0.70	0.54	0.76	0.38	4.53e − 05	0.84
	1.00	0.34	0.76	0.23	5.10e − 06	0.48
25% Slag (serrated)	0.50	0.20	1.04	0.06	6.68e − 07	10.91
	0.70	0.28	0.70	0.09	5.31e − 07	23.17
	1.00	0.23	0.76	0.08	3.82e − 06	23.72

(b) Standard error

Cement pastes and geometry	Gap (mm)	Bingham	Modified Bingham	Herchel–Bulkley	Casson	Sisko	Williamson
8% SF (smooth)	0.50	56.11	162.00	99.17	31.95	11.96	10.41
	0.70	51.81	39.34	92.24	28.61	10.10	11.06
	1.00	48.01	95.67	98.01	23.64	8.38	15.65
8% SF (serrated)	0.50	47.83	166.60	87.76	23.76	5.95	9.54
	0.70	47.69	146.40	88.51	23.18	5.88	11.37
	1.00	41.26	141.60	82.14	20.45	7.07	15.26
25% Slag (smooth)	0.50	67.37	57.51	136.10	47.35	36.09	27.81
	0.70	80.91	51.62	160.10	66.08	152.70	92.98
	1.00	103.10	97.75	181.40	88.11	280.20	154.10
25% Slag (serrated)	0.50	121.50	118.50	159.80	93.98	32.92	12.59
	0.70	86.96	38.26	131.40	58.55	15.11	6.13
	1.00	96.60	187.80	140.60	67.67	16.97	10.12

major sources: (i) mixture composition of cement paste, (ii) geometry, gap and surface friction of the test accessory and (iii) model used in estimating the rheological properties from experimental flow curves. The effects of mixture composition (w/b, mineral and chemical admixtures, etc.) on rheology of cement paste are well studied and documented. They have been included as parameters in this study only to investigate their interaction with test accessories and rheological models. Factors, such as mixing, testing technique, and test geometries, can affect the measured rheological properties of cement paste. This issue has been reviewed by Shaughnessy and Clark [18]. In this study, we maintained all experimental variables constant except the geometry of the flow test, and friction and gap of its shearing surfaces.

Variation of rheological results due to difference in surface friction of the test accessory has often been attributed in this study to effects of slippage. At the wall of shearing test accessories, a reduction of the suspension density adjacent to the solid wall occurs due to the geometric difficulty in arranging particles and to particle migration from regions of high shear rate, a phenomenon called the *wall effect*. Moreover, if the material of the wall does not have affinity to the dispersed particles, the dispersing medium (water in the case of cement paste) tends to form a layer that acts as a lubricant so that there is slippage of the solid suspension along the wall especially under low shearing rates [19,20]. In such a case, roughening the wall surface is desirable, but the extent of which depends to a great extent on the nature and size of the dispersed particles. However, no one has proven yet that slippage does not occur with roughened surfaces [21]. In addition, while often no direct evidence of slip is reported and it is not clear whether it does happen or not, it has proved to be a convenient notion in explaining and interpreting anomalous results reported in the literature [22]. In this study, significant differences between rheological properties measured by smooth and serrated parallel plates, and smooth and vaned coaxial cylinders, respectively, indicate that the effect of the surface roughness of the shearing wall is an important factor.

Another variable that could have affected rheological results in this study is particle sedimentation. Settling of particles during a rheological test is a function of liquid viscosity and density, particle diameter, shape and density, and fractional volume concentration of the dispersed particles [22]. Such parameters have been maintained constant in this study. However, it is conceivable that for the same cement paste, particle sedimentation would be more significant in the case of parallel plates compared to that in coaxial cylinders. Sedimentation or "creaming" in viscometers can result in an increase in the indicated viscosity [14]. However, in most rheological setups, particle settling leads to a decreasing torque with time and leads to misleading results [19]. In cement pastes, it is necessary to keep the water/cement ratio below 0.4 for coaxial cylinder tests to avoid creating a vertical concentration gradient in the tested sample that can affect the validity of results [21]. In this study, no direct evidence of particle settlement is provided, but it is possible that it affected some of the results, especially those of parallel plates at high w/b.

Another reason for difference in rheological properties of cement pastes reported in this study is that for the same testing conditions, such results differ according to the constitutive model used in their calculation. Cement paste exhibits a yield stress, which is indicative of static friction that must be overcome before any flow can take place. While this property is not defined by the model used in its calculation, most authors attribute discrepancy in values of yield stress to mechanisms, such as slip and time dependence [22]. In most cases, shear stress–shear strain data are extrapolated to zero shear rate, the intercept on the coordinate is taken as the yield stress. This process is however, sensitive to the range of data being extrapolated and the rheological model used [22]. This is clear in the significant difference between values of yield stress estimated by the various rheological models for the same cement pastes in this study. Differences in yield-stress values of high-performance pseudoplastic grouts estimated using various rheological properties have been observed elsewhere [3].

For a linear viscous fluid (the flow curve is a straight line), viscosity defines the "constant" rate of increase of shear stress with the increase of shear rate. However, for nonlinear flow curves, the rate of increase of shear stress with the increase of shear rate is not constant, but depends on the particular value of shear rate. To simplify rheological analysis, a constant value known as plastic viscosity [models (1), (3) and (4)], is introduced. It should be understood that such different constitutive equations are trying to predict the same properties [23] and that before accepting a particular model, it is important to see whether the model predictions are physically reasonable and correspond to the type of behavior it is trying to predict [24]. It is observed in this study that estimating plastic viscosity from cement paste flow curves using various rheological models provided significantly different results and the accuracy of curve fitting for the various models, as expressed by a standard error, varied significantly.

These complex interactions between mixture composition, characteristics of the test accessory and constitutive model used in estimating the rheological properties of cement pastes pose a challenge in comparing rheological results from different sources. Added to the fact that the behavior of cement paste is time, temperature and shear-history dependent, there is need to develop detailed and comprehensive standards for testing the rheology of cement pastes for a critical understanding of published rheological data on cement pastes to be gained.

7. Conclusions

In this study, cement pastes of different w/b and incorporating various mineral additions and chemical admixtures were made. Their rheological behavior was tested using coaxial cylinders (smooth and vaned) and parallel plates (smooth and serrated). In the latter case, the gap between the shearing surfaces of the parallel plates was varied. The rheological properties of the various cement pastes were calculated from the resulting flow curves using a variety of rheological models, and the values obtained were analyzed and discussed. From this work, the following conclusions can be drawn:

- Generally, for all test geometries, the Modified Bingham model estimated lower yield-stress values compared to those of the other rheological models, indicating that this model reflected more the influence of very low shear stresses induced in the low shear rate region of flow curves. The Herchel–Bulkley model measured highest yield-stress values, followed by the Casson and Bingham models, respectively. The Bingham and Modified Bingham models consistently estimated an increase of yield stress with a reduction of the w/b for all cement pastes. This general trend was not observed in the case of the other models. Moreover, the yield-stress values estimated by rheological models generally increased with an increase of the RMA dosage.
- For all test geometries, the Modified Bingham model estimated higher plastic viscosity values, followed by the Bingham and Casson models, respectively, for all cement pastes. Generally, a reduction of the w/b resulted in higher plastic viscosity for all three models.
- Generally, for the coaxial cylinders, plastic viscosity and viscosity at zero and infinite shear rates increased with an increase of the RMA dosage for the Bingham, Casson, Williamson and Sisko models, whereas for the Modified Bingham model, peak plastic viscosity values were observed at an intermediate RMA dosage. For the parallel plates tests, no clear trend was observed.
- For the smooth plate, the Bingham, Herchel–Bulkley and Casson models estimated lower yield stress with an increase of the gap between the shearing surfaces of parallel plates, indicating that the effect of slippage may be higher at higher gap. For the serrated plate, a similar trend was observed for the 8% SF cement paste, while a peak value of yield stress was estimated at an intermediate gap of 0.7 mm for the 25% slag cement paste.
- The standard error resulting from fitting various rheological models to experimental flow curves varied with the w/b, mineral additions, chemical admixtures, flow geometry, and friction of, and gap between, the shearing surfaces of the test accessory.
- It is shown that the measured rheological properties of a cement paste depend not only on the w/b and chemical admixtures and mineral additions used, but also on the geometry of the test accessory, the gap and friction capacity of its shearing surfaces, along with the rheological model used to calculate such rheological properties. Therefore, a concerted effort to standardize the testing method and approach used in calculating rheological characteristics of cement pastes is needed to be able to compare interlaboratory results.

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