Viscoelastic Properties of Fresh Cement Paste to Study the Flow Behavior

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Abstract: During concrete pumping, the migration and redistribution of particles occur in a pipe and the lubrication layer that forms between the bulk concrete and the pipe wall is the governing factor determining the flow behavior. In order to identify flow behavior of pumping, in this study, the viscoelastic properties related to the microstructural behavior of a flocculated suspension were examined by using dynamic oscillatory measurements. Cement paste is assumed to be a constituent material of the lubrication layer and ten cases of mixing design are employed by changing the proportions of mineral admixtures. The relationship between the yield stress obtained from the steady shear test and the dynamic modulus resulted from the oscillatory shear measurement was derived and the implications of the correlation are discussed. Moreover, based on the investigation of the viscoelastic properties with oscillatory measurements, the initial behavior of pumped concrete was analyzed systematically.

Keywords: concrete pumping, lubrication layer, dynamic oscillatory measurement, viscoelastic.

1. Introduction

Concrete pumping involves the flow of a complex fluid under high pressure in a pipe, and thus predicting flow behaviors of concrete pumping is challenging research area. For the characterization and prediction of the flow of concrete pumping, the fundamental understanding of various factors is needed, which include rheological properties, dynamic segregation, stability of constituent materials, geometry of a pumping circuit, slip layer formed between bulk concrete and a pipe wall, and relationship between the pressure and flow rate. The flow of concrete in a pipe differs from the one of typical viscous fluids like water or oil. The primary difference is that concrete is a yield stress fluid. Therefore, there is at the center of the pipe (i.e. around the symmetry axis where the shear stress is equal to zero) a zone where the concrete is not sheared (Jacobsen et al. 2009). Most researches on concrete pumping in the literature account for the yield stress and the existence of the unsheared zone (Vassiliev 1953; Kaplan et al. 2005; Feys and Schutter 2005) by assuming that concrete behaves as either the Bingham fluid or the Herschel Buckley fluid. The second reason for the difference is that under the action of shear, the redistribution of particles occurs within a pipe, which is a common feature of particle suspensions. Initially well mixed particles in a concentrated suspension flows undergo migration from high shear rate regions to low shear rate regions (Phillips et al. 1992; Ingber et al. 2009; Lu et al. 2008). During concrete pumping, shear concentrates in the fluid layer of material depleted of the coarsest particles of concrete. Therefore, the pumping of concrete can be generally considered to be the shearing of an annular layer of unknown thickness and made of a material with certain rheological properties.

While particle collisions in highly sheared and/or highly concentrated zones enforce particles to migrate from these zones, it is counterbalanced by the local increase in concrete viscosity resulting from this migration (i.e. in the bulk). The shear induced particle migration (Choi et al. 2013a) is due to the competition between gradients in particle collision frequency and gradients in the viscosity of concrete within a pipe. When cement particles migrate, they encounter a high viscosity of bulk concrete, in which sand and gravel particles have already migrated and should be prevented from migrating inside the bulk. Therefore, the migration of small particles like cement particles can be neglected, compared to the migration of large particles, such as sand and gravel particles. Because of such migration characteristics, the slip layer is generated along the high shear and/or concentrated zone, which can be considered, as an approximation, as being similar to the constitutive cement paste in concrete pumping. This layer is often called the lubrication layer or the slippage layer.

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The existence of the lubrication layer was first suggested by Aleekseev (1952) and Weber (1968). Morinaga (1973) noted that based on theoretical flow behaviors of concrete, the pumping of concrete would not be possible without the formation of the lubrication layer. Sakuta et al. (1979) reported that the flow properties of the bulk material were irrelevant; the only property that matters is the ability of the material to form this layer. Jacobsen et al. (2009) conducted experimental research with colored fresh concrete after flowing ordinary concrete to observe the flow conditions in various pipes. Rossig and Frischbeton (1974) pumped colored concretes in a pipe for the direct observation of the flow profiles. Their results revealed the existence of a high velocity and paste rich zone at the vicinity of the pipe wall. The thickness of the lubrication layer was estimated in the range of 1 mm and 5 mm (Choi et al. 2013a, b; Browne and Bamforth 1977). Feys and Schutter (2005) reported that the thickness and rheological properties of the layer appear to depend on the mix proportions of the pumped concrete. The macroscopic consequences of this layer on the pumping pressure were considered by introducing interface properties measured macroscopically using a tribometer to the pumping process prediction (Kaplan et al. 2005). In summary, the formation and characteristics of the lubrication layer are essential on concrete pumping and thus a detailed analysis of the layer is necessary to understand flow behaviors.

Several studies have examined the rheology of cement slurries which is the constituent material of the lubrication layer for other applications, which include the descriptions of the thixotropic behavior (Papo 1988), and the hysteresis effects in the shear rate/shear stress relationship (Xuequan and Roy 1984; Atzeni et al. 1985; Grzeszczyk and Kucharska 1988). Modified consistometers have been applied to provide gel build up information, as used by Keating and Hannant (1990). Banfill and Kitching (1990) used controlled stress rheometers to examine the yield stresses of cement slurry.

As one of the important information of the rheology, the viscoelastic properties are associated with the slurry microstructure. Several researchers have investigated viscoelastic properties of cement slurry mixtures. Tattersall and Banfill (1983) demonstrated that the paste undergoes the breakdown of structure while the amplitude of shear increases. Cooke et al. (1988) examined viscoelastic parameters for both flowing and curing cement slurries. Chow et al. (1988) discussed the use of the viscoelastic parameters as an alternative to consistometer studies. Saaka et al. (2001) measured viscoelastic properties of cement paste, and illustrated the effect of wall slip on the shear yield stress. Figura and Prud'homme (2010) considered the viscoelastic response of cements with curing-rate control additives. However, little attention has been paid to the viscoelastic properties of flowing materials for examining the flow behaviors, particularly in the pumping industry.

Therefore, the present study examined the viscoelastic properties using dynamic oscillatory measurements to quantify the flow behavior of concrete pumping. Dynamic modulus measurements on the cement paste, which could be considered a constituent material of the lubrication layer, were taken. Steady shear measurements were simply conducted to examine the basic rheological properties like the yield stress and the plastic viscosity while the dynamic oscillatory tests were performed to evaluate the dynamic modulus. The relationship between the measured yield stresses and the dynamic modulus was derived and the implications of the correlation are discussed.

2. Theoretical Background for Viscoelastic Measurements

A dynamic oscillatory shear test is a rheological technique that provides information on viscoelastic materials. This is a dynamic method, in which strain or stress oscillates according to a sinusoidal function. The measured output is the level of stress or strain and the extent to which the stress and strain is in phase with the applied strain or stress. By limiting the strain to small amplitude, the particles remain in close contact with one another and the microstructure is not disturbed.

When a material of a yield stress fluid is sheared under an applied sinusoidal oscillation with a sufficiently small amplitude, the measured dynamic behavior may be linear and can be analyzed in terms of the theory of linear viscoelasticity. Details of the theory and the methods for measuring the viscoelastic properties are well documented in the literature (Ferry 1970; Marin 1988; Nguyen and Boger 1992; Schultz and Struble 1993).

In general, the oscillatory strain at time t is defined as

$$\gamma = \gamma_0 \sin(\omega t) \tag{1}$$

where γ is the strain at time t, γ_0 is the strain amplitude, and ω is the frequency. The resulting shear stress is sinusoidal and proportional to the shear strain in the out-of-phase mode

$$\tau = \tau_0 \sin(\omega t + \delta) \tag{2}$$

where τ_0 is the maximum shear stress and δ is the phase angle between the applied strain wave and the stress response. The shear stress (Eq. (2)) can be expressed using two viscoelastic terms, in-phase term and out-of-phase term, as expressed below;

$$\tau = \tau' + \tau'' = \gamma_0(G' \sin(\omega t) + G'' \cos(\omega t)) \tag{3}$$

where G' is the storage modulus and G'' is the loss modulus. The storage modulus G', representing the in-phase component of stress, is a measure of the elastic energy stored per deformation cycle. The loss modulus, G'', representing the out-of-phase component, is associated with the dissipated viscous energy per cycle. The ratio of G'' to G' provides the tangent of the phase angle. A perfectly elastic fluid leads to $G'' = 0$ and $\delta = 0$, whereas an inelastic viscous fluid results in $G' = 0$ and $\delta = 90°$. In viscoelastic liquids, both moduli are nonzero and the phase angle, which lies between 0° and 90°, depends on the relative contribution between the elastic

effect and the viscous effect. Through the dynamic oscillatory measurements, the oscillatory parameters, G' and G'', can be analyzed to determine viscoelastic properties.

3. Experimental Program

3.1 Materials

The redistribution of particles under the action of shear occurs within a pipe and the lubrication layer can be considered as being similar to the constitutive cement paste of pumped concrete, as discussed in Sect. 1. Based on this assumption, the material for the lubrication layer was obtained from wet-screened cement paste from fresh concrete. The compressive strength of 50 MPa was selected, as shown in Table 1. Portland cement CEM I 52.5N was used with the specific gravity of 3150 kg/m^3. Three types of mineral admixtures were selected, i.e. blast furnace slag (BFS) with the specific surface area of 3950 cm^2/g, fly ash (FA) with 3060 cm^2/g, and silica fume (SF) with 200,000 cm^2/g. Table 2 lists the chemical compositions of the cement and mineral admixtures. The concrete mixes were designed as unitary, and binary blends by replacing cement with mineral admixtures in the study, as shown in Table 3. The sand was natural river sand with the density of 2590 kg/m^3 and fineness modulus of 2.81. The sand particles ranged in size from 0.08 to 5 mm with the water absorption capacity of 2.43 %. The coarse aggregate was limestone with the water absorption capacity of 0.8 %, the density of 2610 kg/m^3, and the fineness modulus of 6.72. The maximum coarse aggregate size was 20 mm. The amount of mixing water was corrected to consider the water absorbed by the fine and coarse aggregates. A polycarboxylate-based high-range water-reducing admixture (HRWRA), which is marked as % HRWRA, meaning the percentage of the admixture relative to the binder content (in weight) was used to obtain the target slump flow.

3.2 Test Procedure

A rheometer was used to measure the rheological properties. In addition to the steady shear measurements, the rheometer is also capable of continuous strain sweep (i.e., at a constant frequency of oscillation the amplitude of the displacement is continuously varied) and frequency sweep (i.e., at a constant stress or strain of oscillatory measurements the frequency of the oscillation is continuously varied). In this study, a parallel plate geometry was adopted to minimize the effects of the particle settling of particles during the measurements. The radius of the bob plate was 25 mm and the distance between the two parallel plates was 1.5 mm. The surface of the bob plate was serrated, as provided by the manufacturer to avoid slippage during the measurement (Fig. 1). The temperature of the rheometer was 25 °C.

Two different measurement procedures were employed, i.e. steady shear and dynamic oscillatory shear. For the steady shear measurement, the sample was subjected to a stepped ramp with 20 different shear rates starting from 0.1 to 550 s^{-1} after a 275 s^{-1} continuous shear for 30 s. Subsequently, the data were acquired at a descending shear rate from 550 to 0.1 s^{-1} with 20 steps, allowing 10 s at each step. Figure 2 presents a schematic representation of the steady shear program. Three replicate tests are performed for each case of mixing design.

For the dynamic oscillatory measurement, it is important to use a strain within the linear viscoelastic region (LVR). To ensure that the measurements were in the LVR, the strain and frequency sweeps were initially performed and checked to identify the LVR. These experiments confirmed that the measurements with the 0.1 % strain and the 1 rad/s frequency resulted in the LVR over the course of the experiment. Thus, strain sweeps were performed at a constant frequency of 1 rad/s within the LVR, ranging from 0.001 to 10 % strain. Frequency sweeps were performed at a constant strain amplitude of 0.1 % within the LVR ranging from 0.01 to 100 rad/s.

4. Results and Discussions

4.1 Steady Shear Properties

Figure 3 shows the relationship between the shear rate and the shear stress from the steady shear measurement for the mixtures. The Bingham model was used to determine the yield stress, τ_y, and the plastic viscosity, μ. Most of the cement slurry data followed this Bingham model quite well;

Table 1 Mixture proportions for C50 (Unitary).

Contents	Values
Design strength	C50
Cement (kg/m^3)	500
W/B ratio	0.33
Sand (kg/m^3)	736
Coarse aggregate (kg/m^3)	871
% Polycarboxylate-based HRWRA*	0.9
Slump flow (mm)	600 ± 20

HRWRA* high-range water-reducing admixture.

Table 2 Chemical compositions of raw materials used in the study (wt%).

Chemical compositions	OPC	BFS	FA	SF
Al_2O_3	4.19	14.55	23.66	1.30
SiO_2	17.76	29.98	49.83	92.00
Fe_2O_3	3.24	0.50	9.03	2.40
CaO	67.16	45.92	8.77	–
MgO	2.26	4.90	1.83	0.40
TiO_2	0.23	0.73	1.62	–
K_2O	1.21	0.60	1.97	1.20
Na_2O	0.09	0,21	0.72	0.10
SO_3	2.99	–	0.96	–
Loss on ignition	0.84	–	2.88	–

OPC ordinary portland cement, *BFS* blast furnace slag, *FA* fly ash, *SF* silica fume.

Table 3 Mix designs.

Blended types	Notation	W/B	OPC (%)	Mineral admixtures (%)			HRWRA (%)
				BFS	FA	SF	
Unitary	UO	0.33	100	–	–	–	0.9
Binary	BFS40		60	40	–	–	
	BFS50		50	50	–	–	
	BFS60		40	60	–	–	
	FA10		90	–	10	–	
	FA20		80	–	20	–	
	FA30		70	–	30	–	
	SF5		95	–	–	5	
	SF10		90	–	–	10	
	SF20		80	–	–	20	

Fig. 1 Serrated bob plate used in the measurements to minimize the effects of slip and settling.
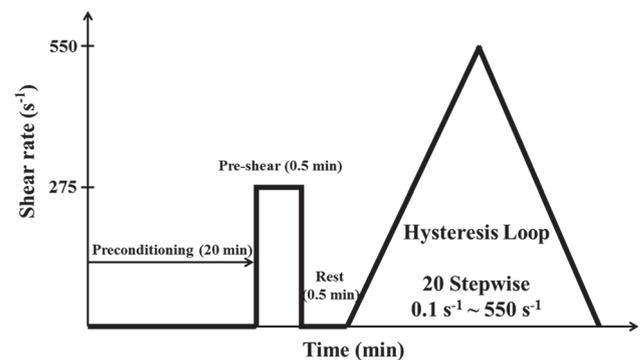

Fig. 2 Schematic of steady shear measurement. In the hysteresis loop 20 points separated by 10 s each were measured for the ascending shear history and the descending shear history.

the viscosity was relatively linear over the range of shear rates. Table 4 lists the calculated Bingham parameters obtained from fitting the data in Fig. 3. The measurements indicated the following:

1. The plastic viscosity and the yield stress of the constitutive cement paste are varied according to the types and replacement ratios of the mineral admixtures.
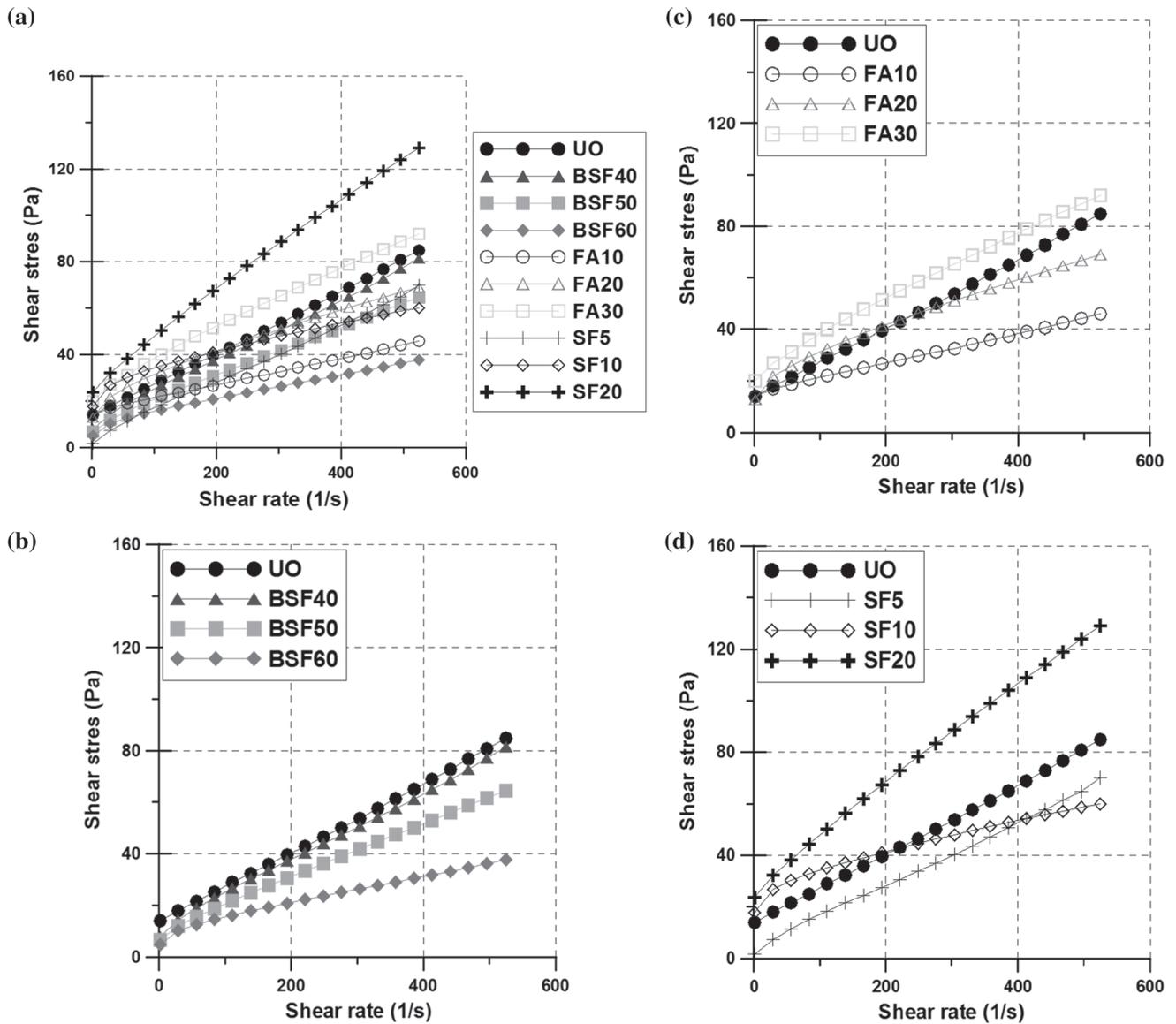
Fig. 3 Results of steady shear measurement. **a** All cases, **b** UO and BSF cases, **c** UO and FA cases, **d** UO and SF cases.

Table 4 Rheological parameters calculated from the steady shear measurements and the dynamic oscillatory measurements.

Blended types	Notation	Steady shear measure		Dynamic oscillatory measure
		τ_y (Pa)	μ (Pa s)	$G^{'}$ (Pa)
Unitary	UO	13.6	0.134	1.49E+02
Binary	BFS40	10.6	0.132	6.43E+01
	BFS50	9.3	0.106	6.19E+01
	BFS60	9.0	0.055	3.51E+01
	FA10	15.1	0.058	1.62E+02
	FA20	20.3	0.097	1.65E+02
	FA30	24.3	0.132	6.78E+02
	SF5	3.5	0.123	8.90E+00
	SF10	25.7	0.069	6.90E+02
	SF20	27.6	0.197	1.86E+03

2. When the replacement ratio of BFS increases, the yield stress and the plastic viscosity of the constitutive cement paste gradually decrease in this study. The measured rheological properties of the mixes with BFS are lower than the mix without BFS, which indicates that BFS improves flowability in this system. This is because the BFS particles fill into spaces among larger particles and decrease frictional forces of matrix.

3. The yield stress and the plastic viscosity slightly increase when the replacement ratio of FA increases. The FA, from a theoretical point of view, should improve flowability as the spherical shape of FA reduces the frictional force among the angular particles, as called ball bearing effect. However, there is a critical factor making worse flowability. The unburned carbon in FA is known to adsorb superplasticizer (SP), which lead to worse workability of matrix. Therefore, in case of the FA mixes tested, the effects of unburned carbon are a more governing factor than the ball bearing effect.

4. The yield stress and plastic viscosity of SF mixtures decrease at 5 % replacement but increase steeply as the SF reaches more than 10 % replacement. Based on these rheological test results for the SF mixes, the ball bearing effect due to the spherical shape of SF could be governing the flowability of matrix at 5 % replacement. However, when the replacement of SF is greater than 5 %, e.g. 10 and 20 % replacement, because of the high specific surface area of the very fine particles of SF (mean size $\approx$ 0.1 μm), the SF particles become highly reactive and easily adsorb SP molecules, which form multi-layers. As the replacements with SF exceed 5 %, the quantity of SP in the system decreases substantially because the significant adsorption of SP into the SF occurred, which leads to a steep increase in the yield stress and plastic viscosity at 10 and 20 % replacements.

4.2 Viscoelastic Properties

Figure 4 presents one of test results of a dynamic oscillatory shear using strain sweep and frequency sweep. Based on the measured test results, the storage modulus, G' (representing the elastic portion in viscoelastic materials), and the loss modulus, G'' (representing the viscous portion). The strain sweep showed variations in the storage modulus with strain amplitude, which was expected for a flocculated suspension. The storage modulus, G', was independent of the strain up to 0.3 % strain amplitude and then at higher strain amplitude, the storage modulus decreased with increasing strain amplitude. This reflects the breakdown of the flocculated network structure as the strain is increased. The product of the modulus and the strain at which breakdown is first observed, i.e. the "critical strain" γ_c, is the threshold for breakdown and has been associated with the yield stress of the system under static conditions. This static yield stress is not necessarily related to the yield stress under flowing conditions, which is reflected in the Bingham or Herschel-Bulkley yield stresses. The loss modulus, G'', was smaller than the storage modulus at LVR, indicating a solid-like response, where most of the stress in the system is supported by the solid colloidal network structure. As the strain amplitude goes beyond the critical value, the loss modulus becomes higher than the storage modulus, which means the behavior changes to a more fluid-like response. Note that the level of G' is still significant, and this structure results in a yield stress in steady flow.

Based on the results of the strain sweep, the strain amplitude of 0.1 %, which exists completely within LVR, was selected for the frequency sweep measurement ranging from 0.01 to 100 rad/s. In Fig. 4b, the frequency sweep showed a plateau in G' in the applied frequency region, over which the modulus varied slightly with frequency. The frequency applied for the strain sweep test, 1 rad/s, also lays on this plateau. As in the strain sweep, the storage modulus in the frequency sweep was higher than the loss modulus in the
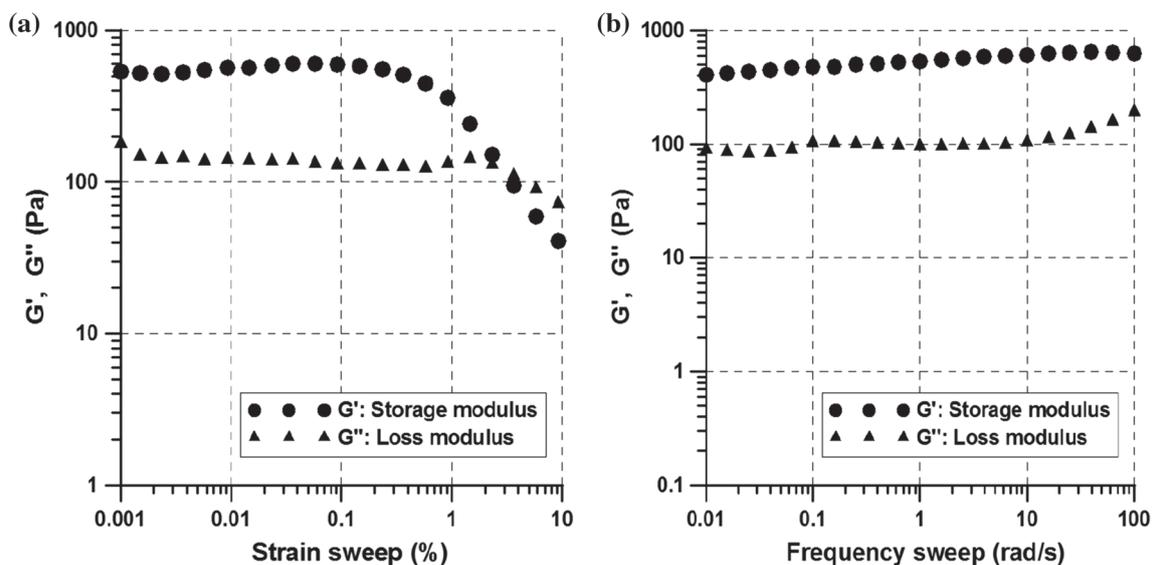

Fig. 4 Representative results of dynamic oscillatory shear measurement. a Strain sweep, b Frequency sweep.

region tested. Both parameters for the dynamic oscillatory tests were within the LVR and the frequency sweep tests were then performed. The results are shown in Fig. 5. The observations for the storage modulus, G', complement the observations from the steady shear flow results. Several conclusions can be drawn from these measurements:

(1) The dispersions were all structured and displayed G' values that were essentially unchanged over the experimental frequency range.

(2) The storage modulus in the frequency sweep decreased gradually in the region tested as the replacement ratios of BFS were increasing but the difference between BSF40 and BSF50 was not distinguished clearly. In the case of FA mixtures, the storage modulus increased slightly with increasing replacement ratios of FA but this was not observed clearly. On the other hand, the SF mixtures showed a clear difference. The storage modulus decreased at 5 % replacement but increased steeply when the SF was over 10 % replacement. The overall variation of the oscillatory shear measurement has a close relationship with the results of the steady shear measurement.

(3) A comparison with the results of the steady shear measurement showed that the tendency of the calculated yield stress follows the trend of the storage modulus variation. Therefore, in this study, a detailed investigation was conducted to determine the relationship between the variation of the storage modulus and yield stress.

4.3 Relation Between the Yield Stress and the Dynamic Moduli

The yield stress provides information concerning the microstructure of the sheared suspension. In this experiment, it is the structure that exists as the shear rate is decreased. In dynamic oscillatory shear, the storage modulus and critical strain are related to the fundamental microstructural aspects of the flocculated suspension at rest (Schultz and Struble 1993). Several studies have shown that systems with G' values independent of frequency also show yield stresses in steady shear flow (Lobe and White 1979; Davis 1971a, b; Ahuja 1980). Onogi et al. (1973) was the first to propose a correlation between the plateau modulus and the yield stress. They analyzed data on a crosslinked polystyrene latex
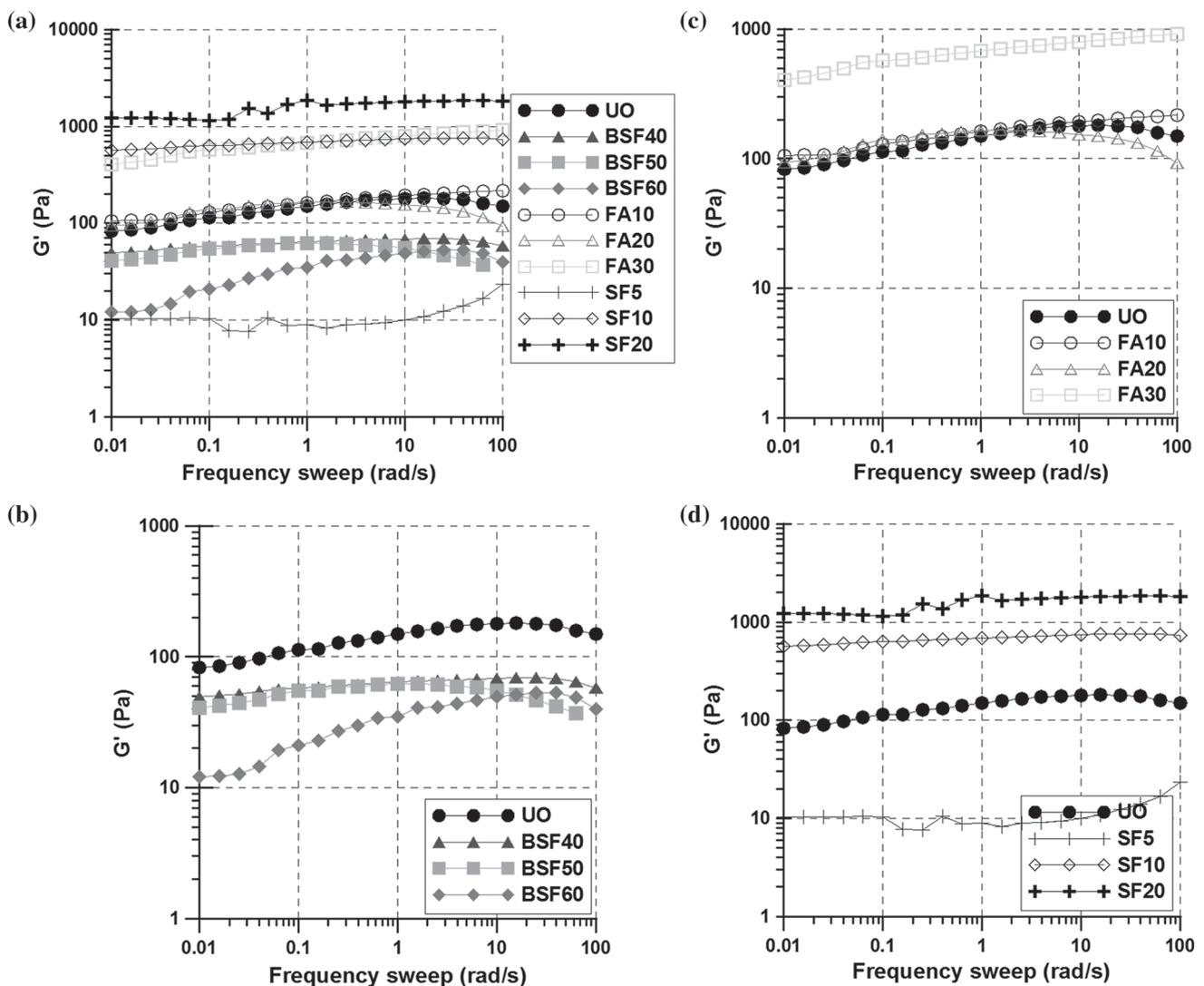

Fig. 5 Frequency sweep test results. a All cases, b UO and BSF cases, c UO and FA cases, d UO and SF cases.

dispersed in a polystyrene solution. While depletion floc-culation was initially described by Asakura and Oosawa (1958), it was not until Heath and Tadros (1983) that the depletion effect was connected to gelation and the rheolog-ical measurements of the modulus. Onogi obtained the yield stress by fitting the non-linearity in their viscosity data with a Casson model, even though their data showed no actual yield point. They obtained G' and G'' as functions of fre-quency, and the data did not display a frequency-indepen-dent plateau. They then proposed a relationship between the yield value and the complex stress defined by

$$\tau^* = \gamma_c \sqrt{(G')^2 + (G'')^2} \approx \gamma_c G_0^* \tag{4}$$

where G_0^* is the complex modulus. Gandhi and Salovey (1988) also reported for their carbon black polymer melt systems that the ratio $\left|G_0^*\right|/\tau_y$ was independent of the par-ticle concentration and surface area, but was dependent on the molecular properties of the polymer medium; they found $\left|G_0^*\right|/\tau_y \simeq 10$ for a low molecular weight polystyrene sus-pension. Yang et al. (1986) plotted the elastic modulus component $G_0' \gamma_0$ versus γ_0; the maximum of the curve cor-responded to the yield stress measured in steady flow. Franck (1988) reported excellent agreement between the yield stress and the component, $G_0' \gamma_{0c}$, where γ_{0c} is the critical strain amplitude corresponding to the onset of nonlinear vis-coelastic behavior.

Based on the power law relationship between the yield stress and complex modulus, the following equation is proposed, which is a modified version of the equation pro-posed by Onogi et al. (1973):

$$\tau_y = a\left|G_0^*\right|^b = a\left|\sqrt{(G')^2 + (G'')^2}\right|^b \approx a(G')^b \tag{5}$$

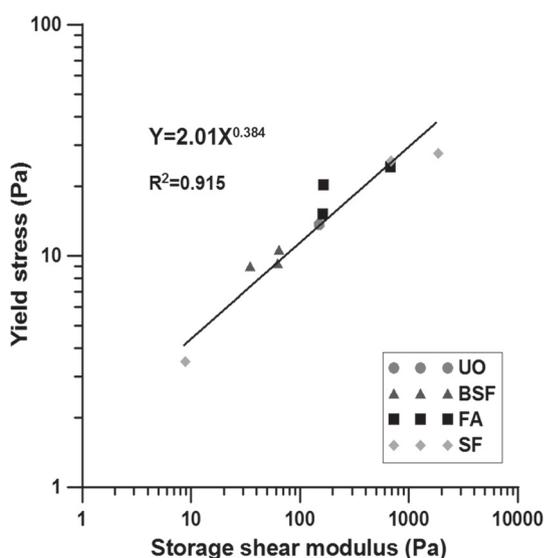For the measured results from all mixtures, the relation-ship between the yield stress and storage shear modulus was

Fig. 6 Correlation between yield stress τ_y and storage shear modulus G'.

examined. As shown in Fig. 6, the mixtures tested in this study showed a good fit to this equation. The variables of the proposed equation were $a = 2.01$, $b = 0.384$, and the coefficient of determination was $R^2 = 0.915$. The final approximation in Eq. (5) provided a similar fit, reflecting the relatively small magnitude of G''. The prefactor, a, in Eq. (5), is an order of magnitude larger than the critical strain. Moreover, the dynamic oscillatory measurements are much faster to perform than the shear history that needs to be imposed to obtain the yield stress. Therefore, the measured parameters from the oscillatory shear measurement were utilized to understand the rheological properties. In addition, the viscoelastic properties, which provide information on the microstructure of the sheared suspension should be exam-ined carefully to predict the flow behaviors in a pipe

5. Conclusions

To understand the flow behavior in a pipe under high pressure, the properties of the lubrication layer between bulk concrete and a pipe wall should be examined. In this study, along with steady shear measurements, oscillatory shear measurements were performed to examine the viscoelastic properties. The following conclusions were obtained:

(1) Due to the migration and redistribution of particles in a pipe under shear by the pumped pressure, the lubrica-tion layer could be considered as being similar to the constitutive cement paste in concrete pumping.

(2) To extend the knowledge of the properties of the lubrication layer, the dynamic oscillatory shear, which is a rheological technique that provides information on viscoelastic materials that are related to the microstruc-tural behavior of flocculated suspension, was mea-sured, which is a relatively new approach to evaluate the rheological performance of pumped concrete. Each of the three mixture systems incorporating BSF, FA, and SF displayed different interactions depending on the types and replacement ratios of the mineral admixtures. Through integrating the results from the steady shear and the dynamic oscillatory measurements it was possible to draw conclusions about the types of interactions occurring.

(3) The yield stress and storage modulus, which arise from fundamental microstructural aspects of the flocculated suspension, can be correlated for all the mixtures tested. The best correlation between the measured yield stress and the complex modulus followed a power law that is somewhat different in form from the mechanical models that are generally used to relate the yield stress to the modulus.

(4) The yield stress is directly related to the flow behaviors in a pipe, which determines the unshared zone in the bulk and governs the microstructure of the sheared suspension. The initial behavior of pumped concrete can be analyzed systematically by examining the viscoelastic properties via oscillatory measurements.

Acknowledgments

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