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Rheology and Setting Time of Cement Paste

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Abstract Experimental results about effect of superplasticizer dosage, fly ash content and water to cement ratio on rheology and setting time are presented. 16 samples are evaluated using a Brookfield laboratory rheometer and a standard vicat needle apparatus. Spread of the samples is also measured using mini slump cone (d1=60mm, d2=35, h=60mm) and an empirical relationship between spread and yield stress is put forward.

Keywords: *brookfield rheometer, setting time, mini slump cone*

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1. Introduction

Grouts made from a mixture of fly ash (FA) and cement are useful in closing rock fissures where low permeability and high strength are desirable. Many Fly ashes have been reported to reduce water requirement, improve flowability, and reduce viscosity at an early age [1,2]. These effects have been attributed to the better particle size distribution, a filler effect on the cement paste and a spherical ball bearing lubrication effect [3,4].

To improve on the workability of grouts without causing bleeding by adding extra water and affecting the strength, its common practice now to add superplasticizer. Their effects of on rheology of cement pastes have been documented by [5,6]. In these studies the shear thickening effect of Polycarboxylate ether superplasticizer is stated. When compared with naphthalene based superplasticizer, it is seen to be more effective.

To properly describe the flow of slurry, two parameters are needed. Usually the apparent viscosity and yield stress are used. However in field conditions, the availability and practicability of using a rheometer are limited, in such cases a mini slump cone has been proposed by several authors and several empirical relationships developed.

The dimensions of many mini slump cones are different however the principle is the same. In studies [7,8,9] it is shown that the workability of the paste is directly related to the spread it attains. [10] proposes a relationship between the yield stress and spread diameter for concrete, D from the Abrams cone. (Eq 1)

$$\tau_0 = \frac{7200\rho g v^2}{128\Pi^2 D^5} \qquad (1)$$

Where v is the volume, τ_0 the yield stress, g is the gravity and D the diameter. This expression is investigated with results obtained for the samples with varying w/c. It is important because different rheometer gives different absolute values for yield stress. The mini slump cone used in this study is schematically shown in Figure 1.

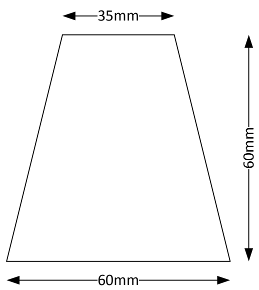

Figure 1. Mini slump cone

2. Materials

Ordinary Portland cement complying with ASTM Type I and Fly ash from a coal plant in China are used for the Cementious materials. A Polycarboxylate ether superplasticizer with a 40% solid content is utilized along with normal tap water. The chemical and physical properties of the materials are given in Table 1 and Table 2.

Table 1. Chemical composition of materials

Compound	FA(fly ash)	C(cement)
Na2O	0.53	0.15
MgO	0.72	2.47
Al2O3	26.87	6.34
SiO2	51.52	23.79
P2O5	0.87	0.14
SO3	1.48	2.29
K2O	0.67	0.59
CaO	6.48	57.29
TiO2	1.20	0.46
Cr2O3	-	0.09
MnO	0.01	0.18
Fe2O3	5.92	3.34
ZnO	0.02	-
SrO	0.14	0.06
ZrO2	0.07	0.02
BaO	0.03	0.06
Cl	0.03	0.05
Loss on ignition	3.43	2.69

Table 2. Physical properties of materials

	Density/kgm^{-3}	Specific surface area cm^2/g
Water	1000	-
Cement	3150	1790
Fly ash	2900	1730

Cement has a volume weighted mean particle size of 17.024μm and the fly ash has a volume weighted mean particle size of 15.952μm.

Samples are prepared and named according to Table 3.

Table 3. Sample preparation

Sample	Cement/ kgm^{-3}	FA/kgm^{-3}	w/b	SP/%
FA0	2380	0	0.3	1
FA10	2142	203	0.3	1
FA20	1904	406	0.3	1
FA30	1666	609	0.3	1
FA40	1428	812	0.3	1
FA50	1190	1015	0.3	1
SP0.4	2380	-	0.3	0.4
SP0.8	2380	-	0.3	0.8
SP1.2	2380	-	0.3	1.2
W0.35	2210	-	0.35	-
W0.40	2040	-	0.40	-
W0.45	1870	-	0.45	-
W0.50	1700	-	0.50	-
T_{30},T_{60},T_{120}	2380	-	0.30	0.8

T_{30}, T60, T_{120} imply curing time in minutes.

3. Experimental

3.1. Mixing

Mixing was done using a planetary mixer at 45rpm for two minutes, followed by a break of 15 seconds and then at 90rpm for a further two minutes. For the sample FA50 containing 50% fly ash, mixing at a high speed was extended for a further two minutes to have uniform slurry.

3.2. Mini Slump

Paste flow ability was measured using the mini slump test according to standard UNE-EN 1015-3. The spread average diameter, D (mm) of the paste is measured using a ruler across two perpendicular diameters after 15 seconds and the spread recorded.

3.3. Rheology

The range of applied shear rate was fixed from 0 to 200/s for all the test runs. All samples are pre sheared at 30S^{-1}, then the shear rate raised from 0-200/s in 150 seconds for the upward curve, it was then reduced from 200/s -0/s in 150 seconds for the down ward curve. The samples are tested 5 minutes from the point of contact between the water and the cement paste. The samples that are left to stand are covered with a glass plate to prevent evaporation.

3.4. Setting Time

Setting time was obtained using a standard Vicat apparatus based on ASTM C191-04b.

4. Results

4.1. Rheology

Figure 2-Figure 5 clearly indicate the variation of both the shear stress and viscosity with shear rate. The power law (Eq 2) is used to calculate τ for samples SP, T and FA.

$$\tau = K\dot{\gamma}^n \qquad (2)$$

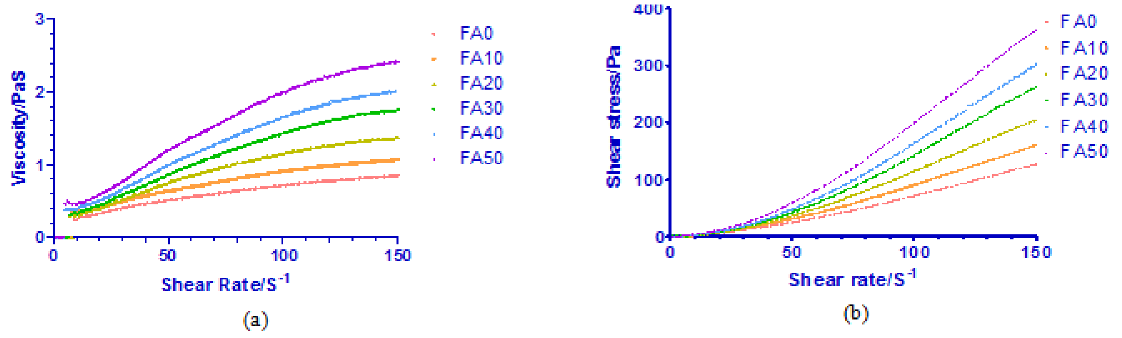

Figure 2. (a) Viscosity (b) rheogram showing effect of fly ash

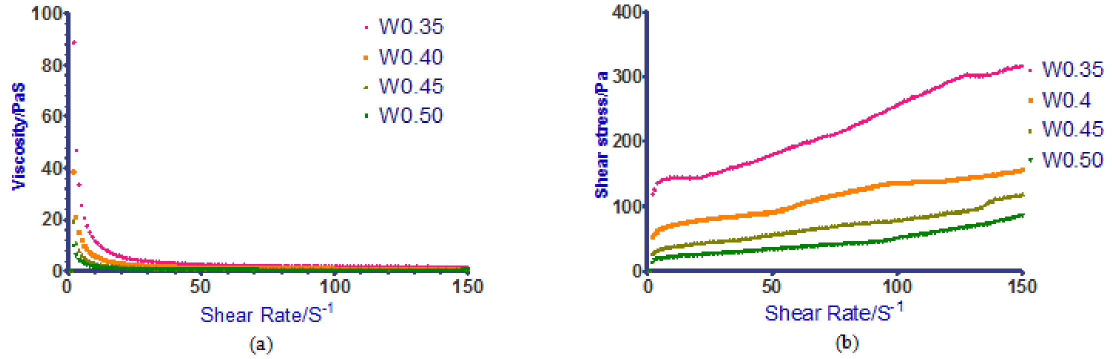

Figure 3. (a) Viscosity (b) rheogram showing effect of water to cement ratio

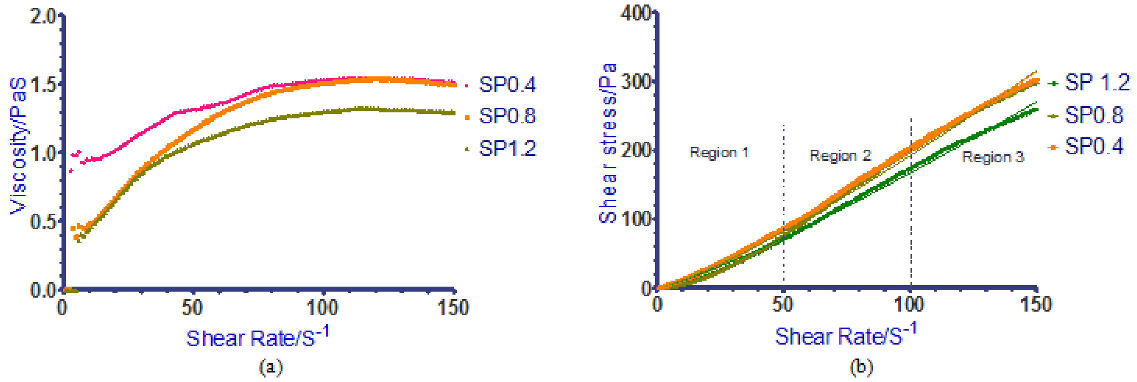

Figure 4. (a) Viscosity (b) rheogram showing effect of dosage of superplasticizer

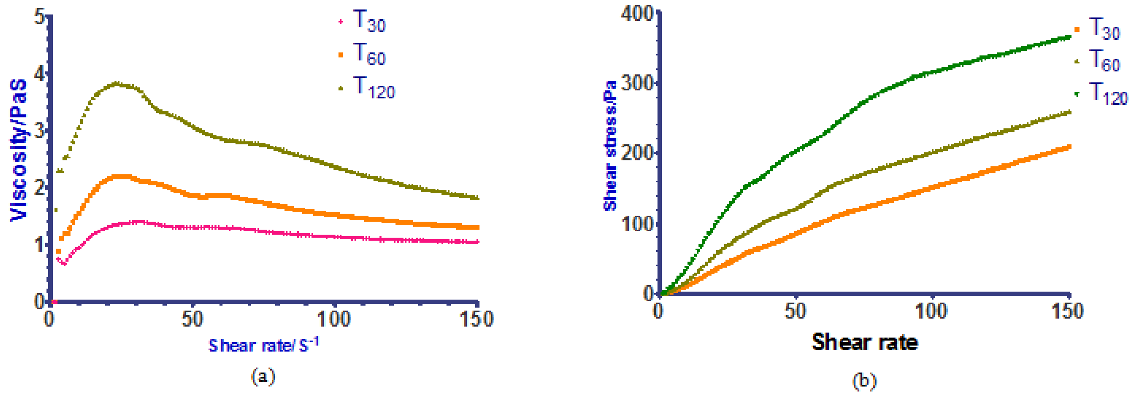

Figure 5. (a) Viscosity (b) rheogram showing effect of time

Consistency K, and power law index n indicate the energy required for flow and the behavior of particles at different shear rates. The samples W and T are evaluated using a Bingham model (Eq 3)

$$\tau = \tau_0 + \eta\dot{\gamma} \tag{3}$$

Table 4. Rheology of samples

Sample	τ_0/Pa	η/PaS	K/PaS^n	n
FA0	0	-	0.075	1.470
FA10	0	-	0.80	1.474
FA20	0	-	0.09	1.540
FA30	0	-	0.095	1.633
FA40	0	-	0.10	1.640
FA50	0	-	0.11	1.624
W0.35	113	0.42	-	-
W0.40	62	0.53	-	-
W0.45	29	0.67	-	-
W0.50	13	1.41	-	-
SP0.4		0.85	1.021	1.143
SP1.2		0.30	0.688	1.192
T_{30}	-	-	3.2	0.83
T_{60}	-	-	7.5	0.71
T_{120}	-	-	17.6	0.62

4.2. Setting Time

The setting time of the samples is shown graphically in Figure 6.

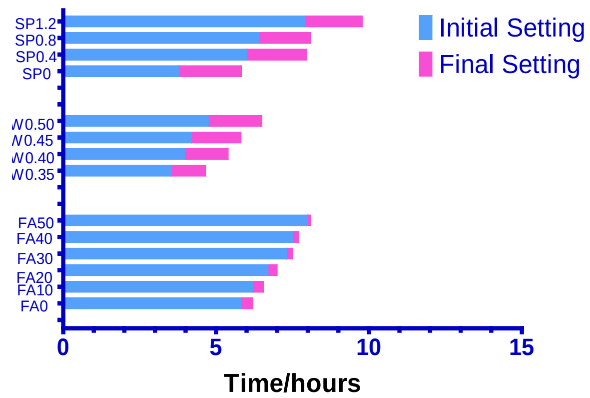

Figure 6. Setting time of the samples

4.3. Spread

The flow spread of all the paste samples was measured after 15 strokes using a ruler. The diameters are measured to the nearest mm and results are presented in Figure 7.

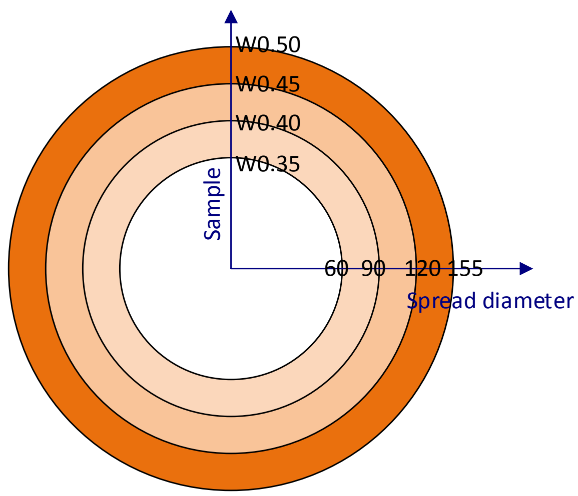

Figure 7. Spread of paste with different w/c

5. Discussions

5.1. Rheology and Spread

From Table 3, increasing the fly ash content from 0% (FA0) to 50% (FA50) corresponds to a gradual increase in index n from 1.47 to 1.624. All samples are shear thickening with values of n>1. Similarly increasing dosage of superplasticizer from 0.4% (SP0.4) to 1.2% (SP1.2) increases n from 1.143 to 1.192. From Figure 4 (a) and 4(b), the viscosity and stress of SP0.8 is very close to that of SP1.2 at low shear rate (<50). At a higher shear rate (>100S^{-1}) the viscosity and stress of SP0.8 is close to that of SP0.4. A longer curing time of 120 minutes increases the initial viscosity from 3.2PaS at 30 minutes to 17.6Pas. At setting times of 30, 60 and 120 minutes, cement paste containing superplasticizer transforms from a shear thickening to a shear thinning fluid. This can be attributed to start of the hydration process. From Figure 3, increasing the w/c ratio reduces the viscosity and the yield stress. Water lubricates the particles better thus reducing the force required for flow to occur. Legrand pointed out possible causes to explain the shear thinning; especially the fact that all the bonds are not broken and those flocs can remain and be dragged by the flow. The number, size, and shape of such flocs will change as a function of the strain rate. Many theories exist to explain the shear thickening behavior; a less-ordered structure dissipates more energy while flowing due to particles ``jamming,'' and hence the viscosity increases at high shear rate. Shear thickening was also attributed by simulation to formation of hydrodynamic clusters that could jam flow. [5] From the spread of pure paste samples with varying w/c (Figure 7) and the yield stresses listed in Table 3, an empirical relationship (Eq 4) is built up between the spread diameter, d and stress, $\tau_{0.}$

$$\tau_0 = 30 + 0.2\frac{\rho g v^2}{\pi^2 d^2} \qquad (4)$$

5.2. Setting Time

Fly ash and superplasticizer increase the initial and final setting time as shown in Figure 6. Fly ash has a retarding effect due to lower pozzolanic activity that OPC. Increasing the water cement ratio also delays the setting.

6. Conclusions

From the study about the effect of a mineral admixture (fly ash), chemical admixture (superplasticizer), time and water/cement ratio on rheology and setting time of cement pastes, the following conclusions can be drawn.

a) Initial and final setting time increase with increase in water to cement ratio, dosage of superplasticizer and fly ash content.

b) All samples containing fly ash or superplasticizer are shear thickening with power index, n increasing with dosage of superplasticizer and content of fly ash.

c) Reducing the w/c increases the viscosity and shear stress at all shear rates but reduces the flow spread of the paste. An empirical relationship is developed between τ_0 and spread, d.

$$\tau_0 = 30 + 0.2\frac{\rho g v^2}{\pi^2 d^2}.$$

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