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Rheological behavior of a slag cement paste prepared by adjusting the particle size distribution

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In this study, the particle size distributions of a cement powder system were adjusted using granulated blast furnace slag powder, Blaine 2250 cm^2/g and 8300 cm^2/g, with which it was easy to adjust the particle size distribution to examine how the particle size distribution of the binder effect the rheological properties of the cement paste. In addition, the relationship between the n-value of the Rosin-Rammler function and plastic viscosity is discussed. All of the measured flow curves represent thixotropic behavior. Also the hysteresis area was smaller with more added coarse particles such as OBB-4 and OBB-2. When the combination was based on a combination of 20-25 vol% fine particles, 30-40 vol% OPC and 40-45 vol% coarse particles in the total volume, a high fluidity and low yield strength was achieved. The plastic viscosity rises, as the n-value decreases and most of the samples indicated that the n-value was correlated with the plastic viscosity. However, in case of a sample which over 30 vol% of fine particles, the plastic viscosity decreased although the n-value decreased.

Key words: Fluidity, Rheology, Cement, Blast furnace slag, Particle size distribution.

Introduction

Recently, high workability for cement or concrete has been attracting much attention. There are growing social needs for the development of a new type of admixture and studies from a materials science perspective. In partially, the fluidity of cementitious materials is closely related with improvements in performance of concrete and is considered to be one of the most important parameters for high performance concrete.

In order to increase the strength and improve the fluidity and durability of concrete, a dispersing organic agent additives has been added at the stage of concrete manufacturing so as to disperse particles or mineral admixtures were added to adjust the particle size distribution of a cement to enhance the packing density of powder. [1-4] Fly ash and blast furnace slag are typical mineral additives and an increase in use of these additive materials for achieving these properties is noted. [5-7]

Furthermore, economics and environmental considerations have also had a role in the growth of mineral additives usage. The lower cement requirement also leads to a reduction in the amount of carbon dioxide(CO_2) generated by the production of cement, while the use of a mineral additives utilizes a product that would ordinarily be bound for land fill. Thus, there is a double environmental benefit from using mineral additives. [8]

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Experiments

Materials

We used ordinary Portland cement (OPC) as intermediate particles with granulated blast furnace slag (BFS) as the coarse and fine particles. The coarse particles were ground with a ball mill from Blaine specific surface area 2250 cm^2/g and the fine particles produced by a classifier. The specific surface area of the fine particles was Blaine 8300 cm^2/g.

The chemical composition of the OPC and blast furnace slag, and the physical properties of the materials used are given Table 1 and 2, respectively.

Mix design

The raw materials were mixed according to the volume

Table 1. Chemical compositions of raw materials

(Unit: mass%)

	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Ig.loss
OPC	62.51	21.10	5.13	3.30	2.72	2.73	1.39
Blast Furnace Slag	44.3	33.3	13.3	0.3	5.8	0.2	0.6

Table 2. Physical properties of raw materials

	Density (g/cm^3)	Blaine (Specific surface area) (cm^2/g)	Mean Particle Size(μm)
OPC (Intermediate particles)	3.15	3450	12.2
Blast furnace slag (Fine particles)	2.90	8300	5.1
Blast furnace slag (Coarse particles)	2.89	2250	26.0

combination by blending, OPC up to 30-70 vol% and the coarse and fine particles BFS at 5-65 vol%, respectively. The mix designs in the study are shown in Fig. 1. For preparing pastes the ratio of water/binder was 1.4 : 1 volume basis.

Test apparatus

Rheological measurements were carried out using a coaxial cylinder viscometer Rheostress-1 Haake, measuring device Z38 with serrated surfaces. The temperature was kept strictly constant at 23 ± 1 °C by an automatic controller. The tests were accomplished under both continuous and physically defined conditions in the CR(Controlled Rate) mode. Details of the serrated spindle and measuring cup are given in Fig. 2.

Mixing procedure and measuring procedure

The slag cement pastes were prepared by the following procedures; the samples were weighed according to blending percentage and water with 1.4 vol% was poured into the sample cup and then the mixture was hand mixed for 5 minutes. After mixing the paste was transferred into the measuring cup for measuring.

The following rheological procedures were applied: in order to get an equilibrium state, the paste sample was not sheared for 0 to 10 minutes by applying a shear rate 0/s. Then the sample was sheared from 0 to 200/s in 2 minutes and 30 s to produce the up-curve of the flow test. Then, the paste sample was sheared from 200 to 0/s in 2 minutes and 30 s to produce the down-curve of the shear rate. [12]

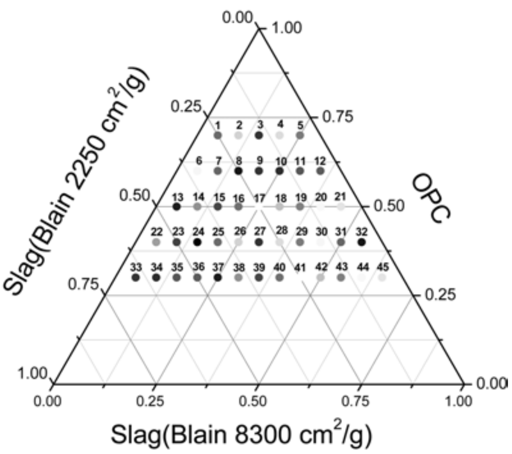

Fig. 1. The mix design of the study (vol%).

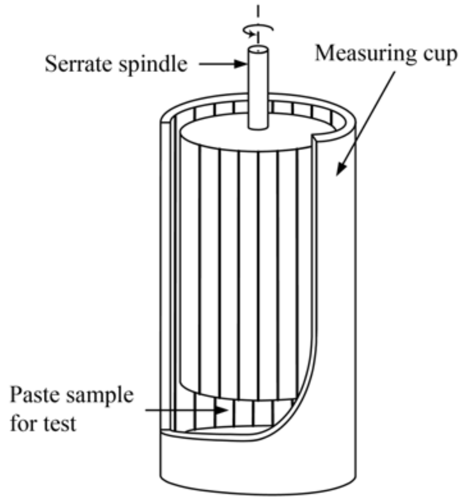

Fig. 2. Details of the serrated spindle and the measuring cup of the Z38.

Analysis of rheological properties

A typical measured flow curve is given in Fig. 3. The flow behavior of fresh paste is a complex rheological phenomenon that is approximately described by the Bingham model. The flow behavior of a test sample can be quantified by the measurable parameters of shear stress and shear rate, as demonstrated by the Bingham model. [9-11] The Bingham model can be represented with the following equation:

$$\tau = \tau_0 + \eta\dot{\gamma} \quad (1)$$

The term τ is the shear stress(Pa), τ_0 is the yield stress (Pa), η is the plastic viscosity(Pa·s) and γ is the shear rate (1/s).

Particle size analysis

The particle size distributions of all samples, 45, which were mixed according to blending contents were measured using isopropyl alcohol as the suspending medium and using a laser particle size analyzer, Analysette 22 (Fritsch, German).

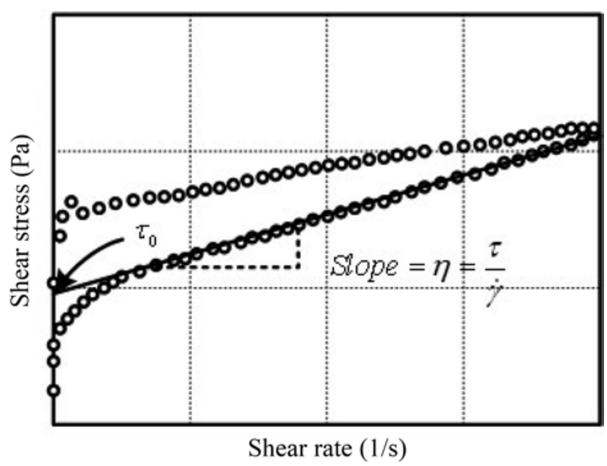

Fig. 3. Typical measured flow curve.

Results and Discussions

The materials used

Raw materials were mixed according to volume combination by blending, OPC up to 30-70 vol% and the coarse and fine particles BSF at 5-65 vol%, respectively. The particle size distribution of all samples were measured by a laser particle size analyzer and classified into 5 types. The representative samples of respective types used in this study and the blending contents of them are given Table 3.

OPC was similar to a log-normal distribution and is defined as type 1.

OBB-1 was an extended log-normal distribution of OPC and is defined as type 2.

OBB-2 represented a binomial distribution at the region of coarse and intermediate particles and is defined as type 3.

OBB-3 represented a multi-distribution with 3 preferential size particles and is defined as type 4.

OBB-4 represented a binomial distribution at the region of intermediate and fine particles and is defined as type 5.

The frequency distribution and cumulative mass distribution of samples are given in Fig. 4 and 5, respectively.

Thixotropy behavior

Commonly, the definition of thixotropy is a decrease in plastic viscosity under the action of shearing at a rate, followed by a gradual recovery when the shear rate is removed, thus producing a hysteresis loop. The area enclosed by the up and down curve of the hyteresis loops was used to evaluate the structure remaining in the cement pastes as a function of particle size distributions for each samples, and the hysteresis loop area of each sample is shown in Fig. 6. The hysteresis area was smaller for the case with more added coarse particles such as OBB-4 and OBB-2. In contrast, the hysteresis area was larger for the case with more added fine particles, Blaine 8300 cm^2/g, indicating that the structure was comparatively broken down with more added fine particles. Because, the fine particles had a large specific surface area rapid progress was made in agglomeration and hydration between particles.

Table 3. Blending combination of raw materials

(vol %)

	OPC	BFS (Fine particles)	BFS (Coarse particles)	Type of particle size distribution
OPC	100	-	-	Type-1
OBB-1	70	25	5	Type-2
OBB-2	40	20	40	Type-3
OBB-3	40	45	15	Type-4
OBB-4	30	25	45	Type-5

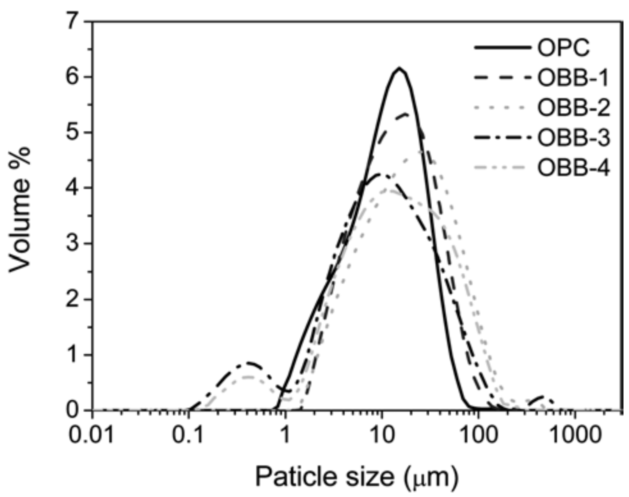

Fig. 4. Frequency distribution of samples.

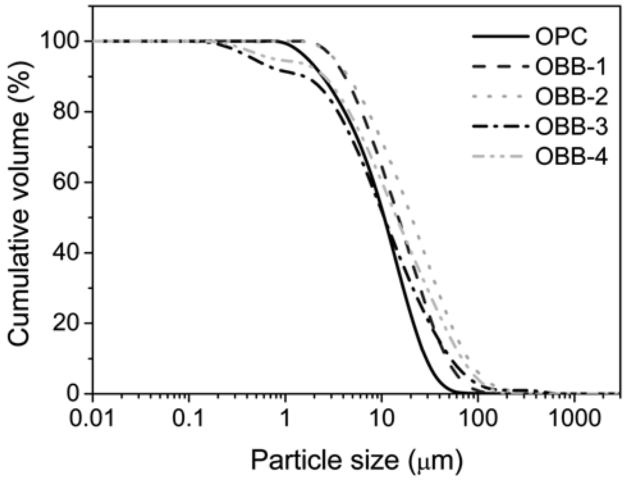

Fig. 5. Cumulative mass lager distribution of samples.

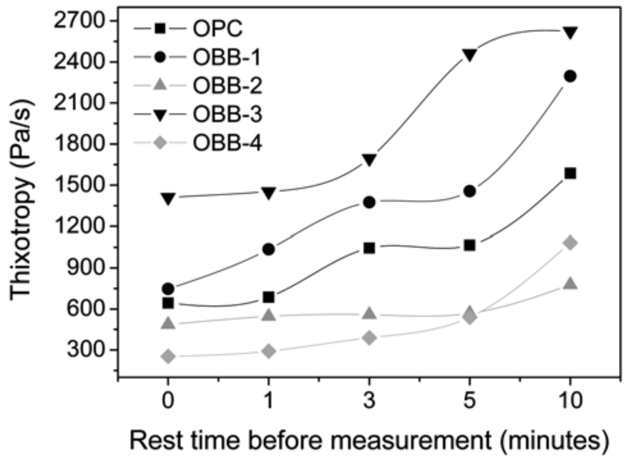

Fig. 6. Comparison of the thixtropy areas.

Plastic viscosity and yield stress

The result of a comparison with the plastic viscosity and the yield stress of all samples, including OPC, are given Fig. 7 and 8, respectively. Both plastic viscosity and yield strength increase by the passage of time for all samples. OBB-4 that had a particle size distribution of type 5 has the best fluidity and lowest yield strength. The yield strength decreases with a decrease in the amount of fine particles. In order words, more coarse

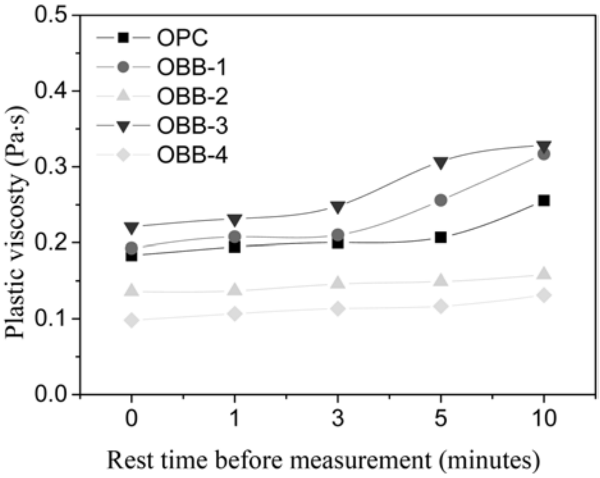

Fig. 7. Effect of the passage of steady on the plastic viscosity.

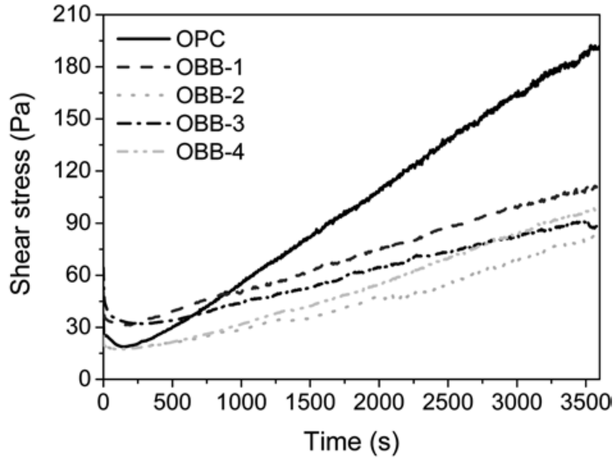

Fig. 9. The transition of the shear stress for I hour at constant shear rate 10/s.

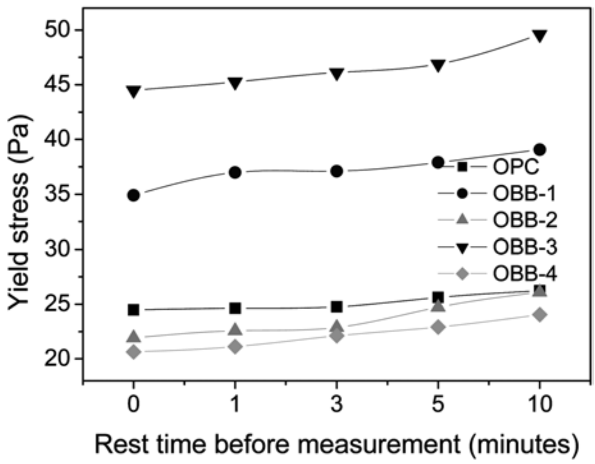

Fig. 8. Effect of the passage of time on the yield stress.

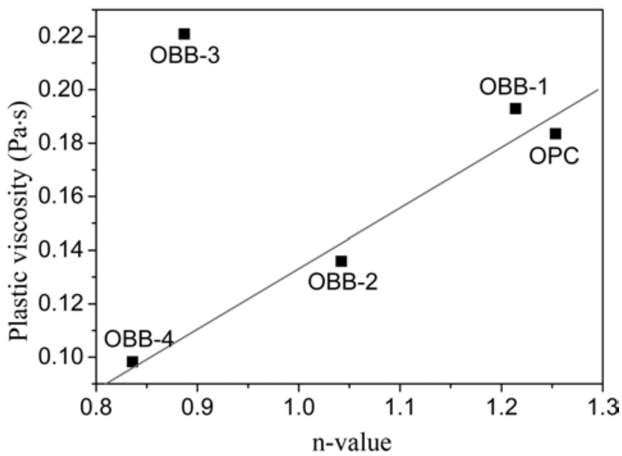

Fig. 10. Relationship between n-value and plastic viscosity.

particles led to a higher yield strength in the case of a batch with smaller amount of fine particles. This because more fine particles produce more contact points among the particles, and as result a higher stress is needed for deformation.

Transition of shear stress

The transition of the shear stress over 1 hour was studied, setting the shear rate at 10/s. The shear stress decreased steadily up to 5 minutes due to a break down of agglomerated particles by the external torque. Also the plot of shear stress against shearing time had a point of inflection which means the shear stress increased from 5 minutes to 1 hour due to a rise in the cohesion among the particles by the hydrate reaction with the binder. OBB-4 starts with the lowest cohesion but has more cohesion than the other samples after 5 minutes. By cohesion, do we mean plastic viscosity.

The relationship between fluidity and RR function

In order to study the relationship between particle size distribution and fluidity, the n-value of the Rosin-Rammler function and plastic viscosity of each sample were compared. Many have reported that particle size distribution of those ground materials such as cement generate a Rosin-Rammler function relatively better. In this experiment, the particle size distribution of each sample was measured employing the laser particle size analyzer in addition the n-value of the Rosin-Rammler(RR) function via nonlinear square fitting was determine. RR function is represented by the following:

$$R(Dp) = 100 \cdot \exp[-(Dp/De)^n] \quad (2)$$

where R(Dp) is the cumulative mass percentage, Dp is the particle diameter, De is a characteristic size parameter for which the cumulative mass percentage is 36.8% and n is the index indicating the size dispersion. Thus, as the n-value decreases, the particle size distribution becomes wider. [13]

The plastic viscosity rises, as the n-value decreases, and most of the samples showed an n-value had a correlation with plastic viscosity. However, in the case of the sample which more than 30 vol% of fine particles(OBB-3), the plastic viscosity decreased although the n-value decreased.

Conclusions

There experiments were carried out to study the relationship between particle size distrivution and fluidity in cement pastes containing blast furnace slag.

- All of the measured flow curves showed thixotropic behavior. Also the hysteresis areas were smaller for the samples with more coarse particles added such as OBB-4 and OBB-2.
- When the mixture was based on 20-25 vol% fine particles, 30-40 vol% OPC and 40-45 vol% coarse particles of the total volume, a high fluidity and a low yield stress were achieved. Furthermore the cohesion and hydration rate of particles were lowest in the early measurements, but they dramatic ally increased after measuring for more than 5 minutes, except for the OPC sample.
- The plastic viscosity rises, as the n-value decreases and most of the samples showed that the n-value had a correlation with the plastic viscosity. However, in the case of the sample which more than 30 vol% of fine particles (OBB-3), the plastic viscosity decreased although the n-value decreased.

In conclusion, we have achieved a high fluidity and low yield stress though controlling the particle size distribution of cementitious materials.

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