Effect of Particle Size Distribution of Binder on the Rheological Properties of Slag Cement

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1. 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. Especially, the fluidity of cementitious materials is closely related with improvement in performance of concrete and considered one of the most important parameter of high performance concrete.

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

Furthermore, economics and environmental considerations have also had a role in the growth of mineral admixture 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 admixture utilizes a product that would ordinarily be bound for the land fill. Thus, there is a double environmental benefit from using mineral admixtures.[8,9]

In this study, particle size distribution of cement powder system were adjusted using the granulated blast furnace slag powder, Blaine 2250cm^2/g, and 8300cm^2/g, which easy to adjust particle size distribution to examine how particle size distribution of the binder has an effect on rheological properties of the cement paste. In addition, the relationship between n-value of Rosin-Rammler function and plastic viscosity were discussed.

2. Experimental

2.1 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 grounded with a ball mill at blaine 2250cm^2/g and the fine particles produced by classifier. Specific surface area of fine particles was blaine 8300cm^2/g.

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

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 (cm^2/g)	Mean Particle Size( )
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

2.2 Mix design

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

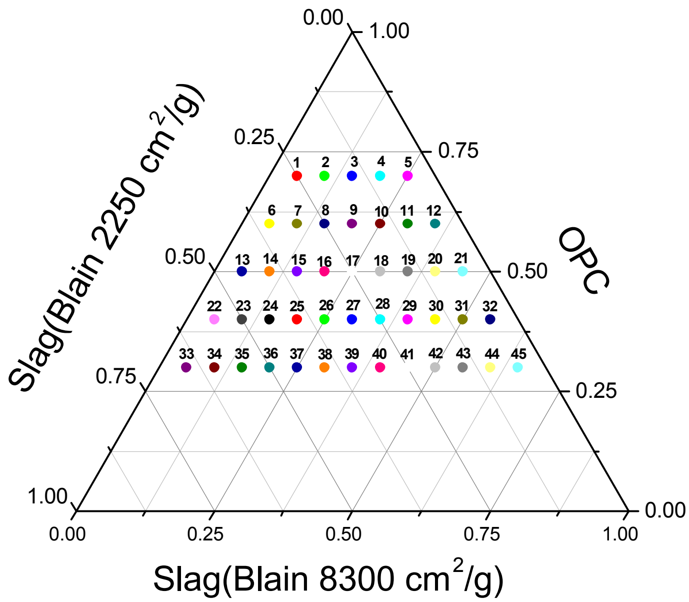

Fig. 1 The mix design of the study (vol%)

2.3 Test apparatus

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

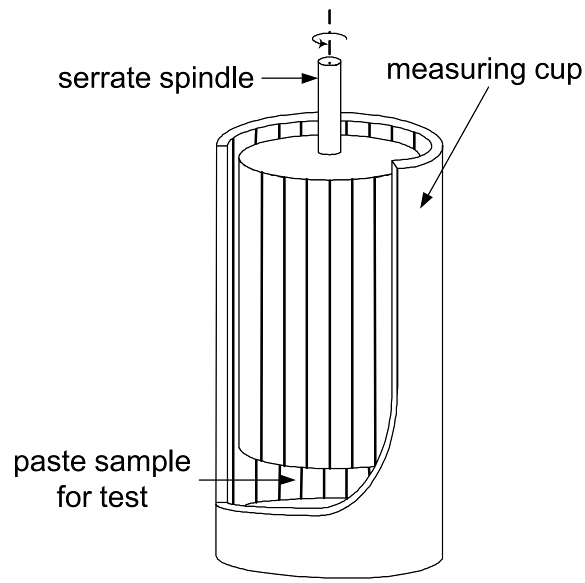

Fig. 2 Detail of the serrated spindle and the measuring cup, Z38

2.4 Mixing procedure and measuring procedure

The slag cement pastes were prepared as following procedures; the samples were weighed according to blending ratio and water with 1.4vol% was poured in sample cup and then the mixture was mixed for 5 minutes by hand mixing. 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 sheared for 0 to 10 min by applying a shear rate 0/s. And the sample was sheared from 0 to 200/s within 2 min and 30 s to procedure the up-curve of the flow test. Then, the paste sample was sheared from 200 to 0/s within 2 min and 30 s to produce the down-curve of the flow test.[14]

2.5 Analysis of rheological properties

Typical measured flow curve is represented in Fig. 3. Flow behavior of fresh paste is a complex rheological phenomenon that is roughly described by 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 Bingham model.[10-13] The Bingham model can be represented with the following equation:

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

The term τ is shear stress(Pa), τ_0 is the yield stress(Pa), η is the plastic viscosity(Pa.s) and $\dot{\gamma}$ is shear rate(1/s).

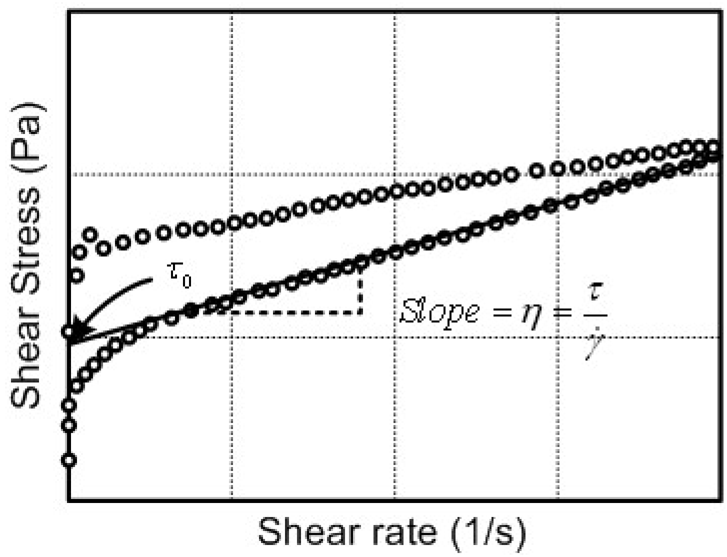

Fig. 3 Typical measured flow curve

2.6 Particle size analysis

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

3. Results and discussion

3.1 The used materials

In earlier study, the raw materials were mixed according to the volume ratio by blending, OPC up to 30~70 vol% and the coarse and fine particles at 5~65 vol%, respectively. The particle size distribution of all samples were measured by laser particle size analyzer and classified into 5 types. The representative samples of respective type were used in this study and the blending ratio of them are given table 3.

OPC was similar to lognormal distribution and defined as type 1.

OBB-1 was extended log-normal distribution of OPC and defined as type 2.
OBB-2 represented binomial distribution at the region of coarse and intermediate particles and defined as type 3.
OBB-3 represented multi-distribution with 3 preferential size particles.
OBB-4 represented binomial distribution at the region of intermediate and fine particles. Respectively they defined as type 4 and 5.
Frequency distribution and cumulative mass lager distribution of samples are given Fig. 1 and 2, respectively.

Table 3. Blending Ratio of Raw Materials

	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

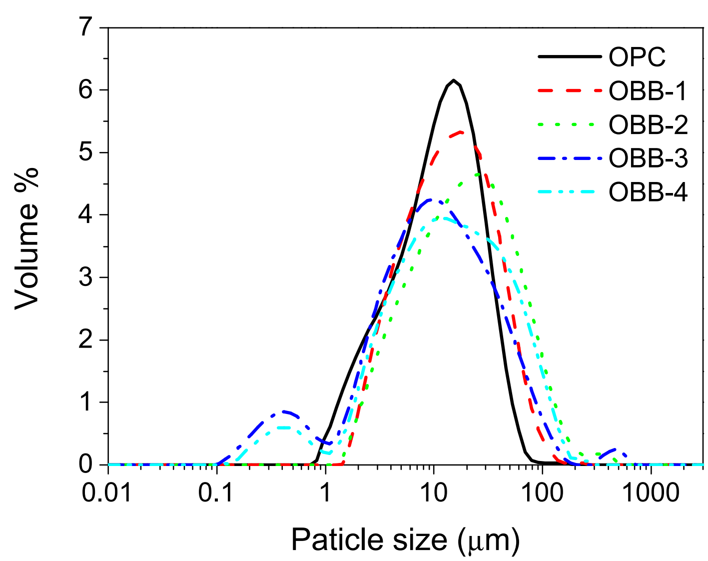

Fig. 4 Frequency distribution of samples

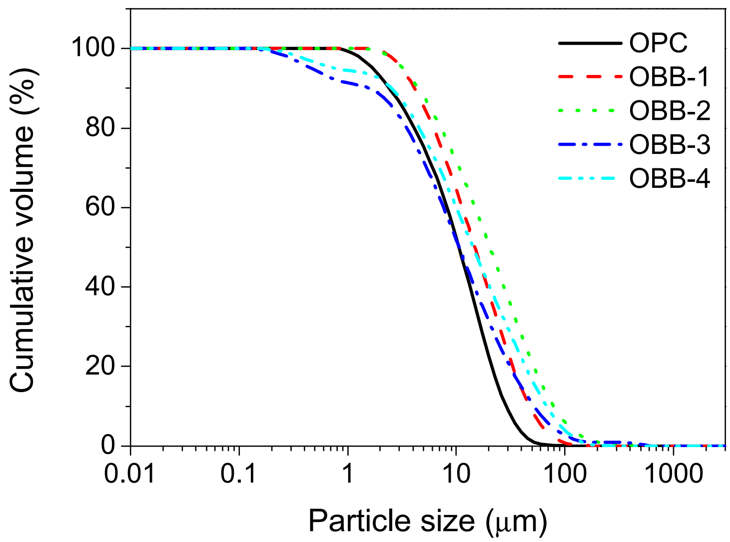

Fig. 5 Cumulative mass lager distribution of samples

3.2 Thixotropy behavior

Commonly, the definition of thixotropy is a decrease in plastic viscosity under shear rate, followed by a gradual recovery when the shear rate is removed, and producing a hysteresis loop. The area enclosed by the up and down curve of hyteresis loops were used to evaluate the structure remaining in the cement pastes as the 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 more added coarse particles like OBB-4 and OBB-2. In contrast, the hysteresis area was lager for the more added fine particles, Blaine 8300cm^2/g, indicating that the structure was comparatively broken down for more added fine particles. Because, the fine particles had a large specific surface area make rapid progress agglomerate and hydration between particles.

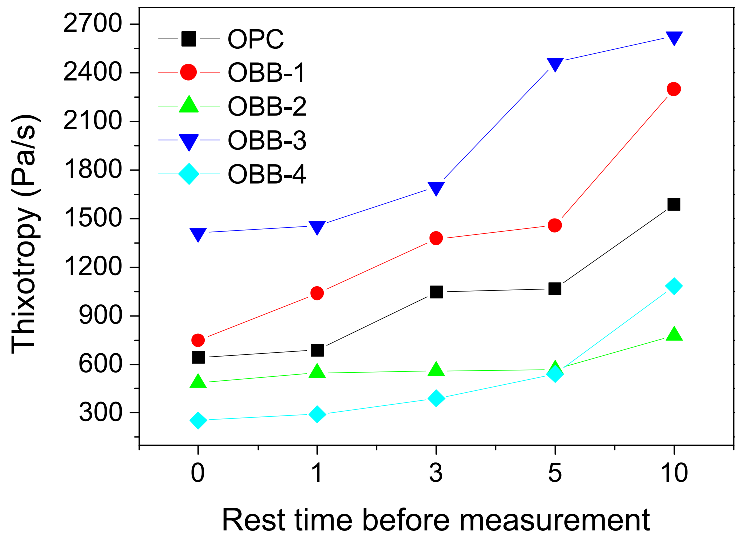

Fig. 6 Comparison of the thixtropy area

3.3 Plastic viscosity and yield stress

The result of 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 the steady time at all samples. OBB-4 that had a particle size distribution of type 5 is most good fluidity and lowest yield strength. Yield strength is getting weaker as the amount of the fine particles is less. In order words, more coarse particles led to yield strength in case of the batch with smaller amount of fine particles. This is due to more fine particles produce more contact points among the particles, and in return, stronger stress is needed for deformation.

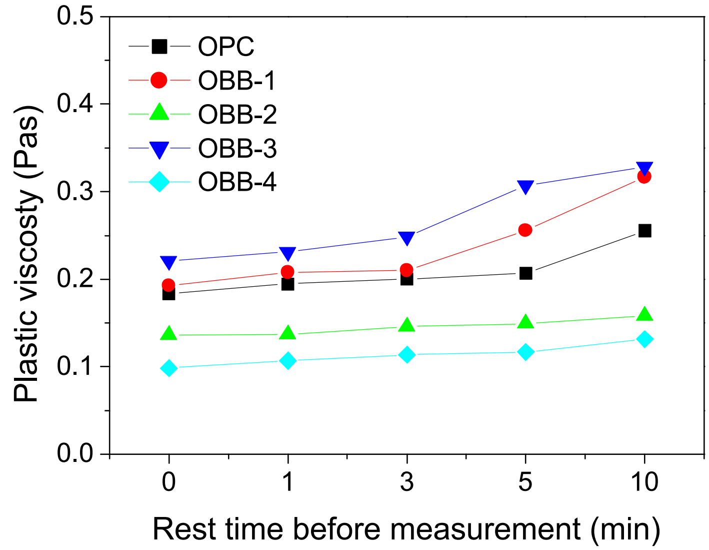

Fig. 7 Effect of the passage of steady time on the plastic viscosity

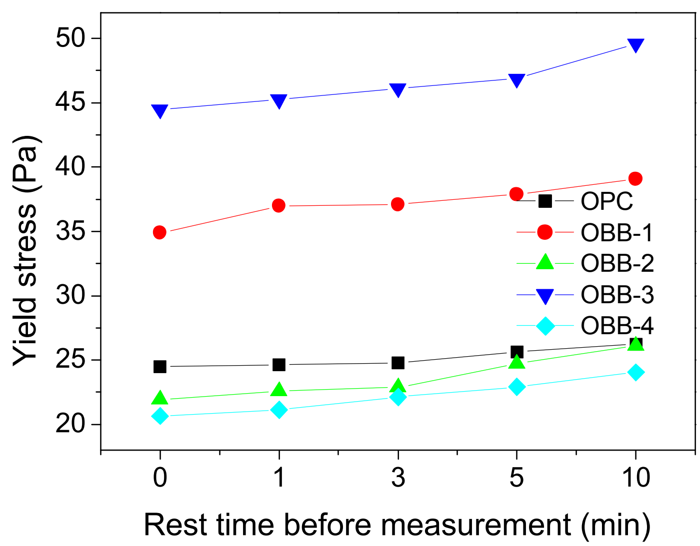

Fig. 8 Effect of the passage of steady time on the yield stress

3.4 Transition of shear stress

The transition of shear stress for 1 hour was studied, setting the shear rate at 10/s. shear stress decreased steadily to 5 min due to break down structure of agglomerated particles by external torque. And shear stress had a point of inflection which mean the steady state but shear stress increased from 5 min to 1 hour due to rise cohesion among the particles by hydrate reaction of the binder. OBB-4 starts with lowest cohesion but has more cohesion than other samples after 5 min. By cohesion, do we mean plastic viscosity.

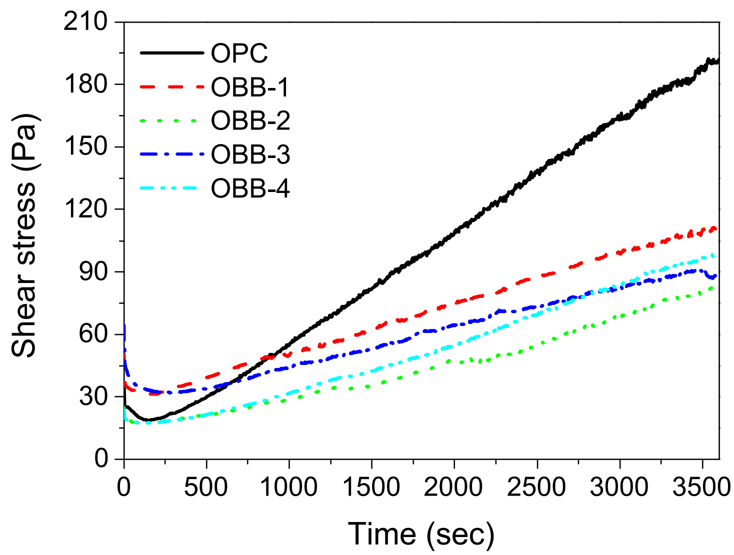

Fig. 9 The transition of the shear stress for I hour at constant shear rate 10/s

3.5 The relationship between fluidity and RR function

In order to study for the relationship between particle size distribution and fluidity, the n-value of Rosin-Rammler function and plastic viscosity were compared. Many report that particle size distribution of those grinded materials such as cement is relatively better generated by Rosin-Rammler function. In this experiment, the particle size distribution of each samples were measured employing the laser particle size analyzer as well as the n-value of Rosin-Rammler function via nonlinear square fitting. RR function is presented as following:

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

Where R(Dp) is cumulative mass percentage, Dp is particle diameter, De is characteristic size parameter for which cumulative mass percentage is

0.47 and n is index indicating dispersion of size. Besides, as the n-value decreases, the particle size distribution becomes wider.[15]

The plastic viscosity rises, as the n-value decreases, most of samples indicated that the n-value had a correlation with plastic viscosity. However, in case of sample which excess over 30 vol% of fine particles, plastic viscosity decreased though the n-value decreased.

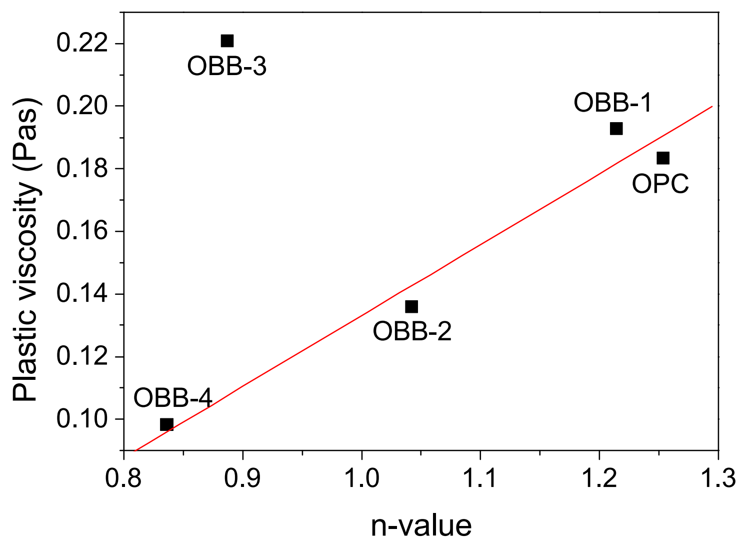

Fig. 10 Relationship between n-value and plastic viscosity

4. Conclusion

This work is carried out to study the relationship between rheological properties and fluidity in cement pastes containing blast furnace slag.

- All of the measured flow curves represented thixotropy behavior. And the hysteresis area was smaller for the more added coarse particles like OBB-4 and OBB-2.
- When the combination was based on a ratio of 20~25 vol% fine particles, 30~40 vol% OPC and 40~45 vol% coarse particles of the total volume, a high fluidity and low yield stress was achieved. Furthermore the cohesion and hydration rate of particles were lowest at the early measurement, but it dramatic increased after measuring 5min, except OPC
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indicated that the n-value had a correlation with plastic viscosity. However, in case of sample which excess over 30 vol% of fine particles, plastic viscosity decreased though the n-value decreased.

In conclusion, we achieved high fluidity and low yield stress though controlled particle size distribution of cementitious materials.

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