



# A study on the effect of fine mineral powders with distinct vitreous contents on the fluidity and rheological properties of concrete

Yun-Xing Shi<sup>a,\*</sup>, Isamu Matsui<sup>b</sup>, Yu-Jun Guo<sup>c</sup>

<sup>a</sup>Beijing Building Research Institute of CSCEC, No. 1, Xinhua Road, Nanyuan, Beijing 100076, China

<sup>b</sup>College of Industrial Technology, Nihon University, Chiba 275-8575, Japan

<sup>c</sup>Beijing Xin Hong Guang Building Material Co., Ltd., Beijing 100076, China

Received 26 August 2003; accepted 30 December 2003

## Abstract

This paper presents an investigation on the effects of fine mineral powders on the fluidity and rheological properties of concrete. It was observed that the fluidity of concrete increased noticeably, and the plastic viscosity decreased, when vitreous powders were substituted partially for cement. It was found that vitreous powders have a strong dispersion effect on the fluidity and rheological properties of concrete, and this effect can be correlated with the vitreous content of the powders incorporated.

© 2004 Elsevier Ltd. All rights reserved.

**Keywords:** Mineral admixture; Properties of concrete; High-performance concrete; Fresh concrete

## 1. Introduction

The incorporation of mineral admixture is now an important technique in improving the properties of concrete. These properties, such as fluidity, strength, durability, etc., can be considerably influenced by both the physical and chemical characteristics of the powders. Although many researchers have reported on the latent hydraulicity or pozzolanic effect of mineral admixture, as well as the microfilling effect of the fine mineral powders [1–3], there are few studies on the effect of the surface activity of fine mineral powders. The role of the much finer powders, incorporated in high-performance concrete (HPC), involves not only their hydraulicity or pozzolanic effect and their microfilling effect, but also their surface activity. Mineral materials, especially those with a vitreous phase, can acquire a high surface activity through the process of fine grinding [4–6], which can notably influence the fluidity and rheological properties of concrete. In our previous studies [4–7], the influence of the

dispersion effects, and its interaction with other powders on fluidity, and strength of concrete have been investigated. In this paper, the effect of powders, with distinct vitreous contents, on the fluidity and rheological properties of concrete are discussed.

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Cement

The cement used in this investigation was ordinary Portland cement (OPC) of grade 42.5, produced in accordance with China National Standard GB175 by Jidong cement plant, Hebei Province. The chemical composition and physical properties of the cement are listed in Table 1.

#### 2.1.2. Fine mineral powders

Four kinds of fine mineral powders were used. Two were made from blast furnace slag (BFS) and two from phosphorous slag (PS). The chemical compositions of the powders are also given in Table 1. X-ray diffraction (XRD; CuK $\alpha$ ) shows that BFS and PS have similar XRD

\* Corresponding author. Tel.: +86-10-83982409; fax: +86-10-83982102.

E-mail address: [y\\_x\\_shi@yahoo.com](mailto:y_x_shi@yahoo.com) (Y.-X. Shi).

Table 1  
Chemical and physical analysis of cement and fine powders

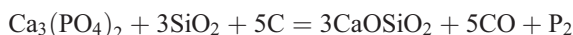
	OPC	BFS	PS	RBFS <sup>a</sup>	RPS <sup>b</sup>
<i>Chemical (percentage by mass)</i>					
CaO	63.31	36.90	43.75		
SiO <sub>2</sub>	22.04	32.32	36.08		
Al <sub>2</sub> O <sub>3</sub>	5.11	12.02	4.97		
Fe <sub>2</sub> O <sub>3</sub>	3.36	2.25	2.88		
MgO	3.25	9.14	1.71		
Na <sub>2</sub> O	0.2	—	—	—	—
K <sub>2</sub> O	0.77	—	—	—	—
SO <sub>3</sub>	2.34	0.23	0.18		
P <sub>2</sub> O <sub>5</sub>		0.16	0.60		
Loss on ignition	1.18	1.03	0.82	0.06	0.042
<i>Physical</i>					
Blaine surface area (cm <sup>2</sup> /g)	3200	6240	6129	6318	6274
Specific gravity	3.1	2.88	2.91	2.90	2.93

<sup>a</sup> Chemical composition is the same as BFS.

<sup>b</sup> Chemical composition is the same as PS.

patterns (Fig. 2a and c), and that their vitreous contents are greater than 90% and 85%, respectively, as indicated in the previous investigation [5].

The phosphorus slag used in this investigation is a by-product from the production of yellow phosphorus. During the manufacturing of phosphorus, quartz sand and coke are added to the powdered calcium phosphate mineral, to be heated at 1450 °C and to produce phosphorus through the following reaction:



The 3CaOSiO<sub>2</sub>, with some impurities, was quenched with cool water to form a pale-colored granulated slag with diameters of 0.5–5 mm.

In addition, to identify the influence of the vitreous content of the powders on the properties of concrete, two other powders were made from retreated blast furnace slag (RBFS) and phosphorous slag (RPS), with approximately the same Blaine fineness to BFS and PS. The retreating process is then discussed.

First, BFS and PS were heated to 1100 °C in a melting furnace and kept at that temperature for 30 min. Then, the temperature was lowered to 500 °C, with a drop of about 130 °C/30 min. Finally, they were cooled down naturally to room temperature in the furnace, with its shutter open. The temperature change curve before the natural cooling is shown in Fig. 1. XRD patterns in Fig. 2c and d show that the diffraction peaks in RBFS and RPS rose noticeably. This indicated that the crystalline C<sub>3</sub>MS<sub>2</sub> (in RBFS) and C<sub>2</sub>AS (in RPS) formed as the vitreous phase degenerated, as compared with BFS and PS in Fig. 2a and b.

According to the peak strength analysis method suggested in the literature [8], the vitreous contents of RBFS and RPS were evaluated, and the contents determined are both about 40%. Hence, the two of them will be called semicrystalline powders in this report. A SEM observation

of the grain features of the four powders is shown in Fig. 3. It can be seen that the grains of each type are of irregular shape and have a wide size distribution. In addition, the grains of RBFS and RPS seem to have rougher surface as compared with that of BFS and PS.

### 2.1.3. Aggregates

The coarse aggregate used in the experiment was river gravel, with a nominal maximum size of 31.5 mm. The fine aggregate was river sand, with a fineness modulus of 2.6.

### 2.1.4. Chemical admixture

A powdered, high-range naphthalene-based water reducer (HRWR) was used, of which the amount of Na<sub>2</sub>SO<sub>4</sub> was less than 5% by mass.

## 2.2. Experimental methods and apparatus

The mix proportions of concretes with various contents of the powders are shown in Table 2. For each type of powder, five batches of concrete with powder contents of 10%, 20%, 30%, 40% and 50% were prepared.

The concrete batches were prepared by using a forced mixer with a nominal capacity of 0.05 m<sup>3</sup>, and slump flow at 30 s, as well as slump, was measured in accordance with China National Standard GBJ80. Then, a part of fresh concrete for each batch was wet sieved on a vibrating sieve with a mesh size of 5 mm. The matrix mortar separated out was taken as the sample for rheological measurements.

The rheological apparatus used in the experiment was a Model35 viscometer (Fig. 4), with a coaxial outer cylinder and rotating inner bob. Either the inner bob or the outer cylinder can be chosen from the four diameter sizes, and 12 rotating speeds of the bob are available with respect to the sample condition.

In the present experiment, the rotational torques applied to the sample were measured for various rotational speeds: 0.9, 1.8, 3, 6, 30 rpm, etc.

Since fresh cement mortar displays Bingham flow, the plastic viscosity ( $\eta$ ) and the yield stress ( $\tau_0$ ) can be obtained

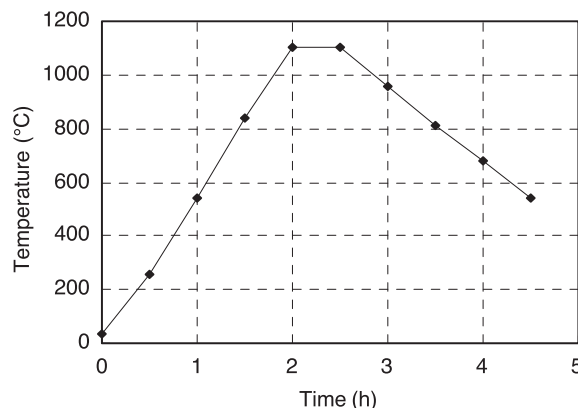


Fig. 1. Temperature change during retreatment.

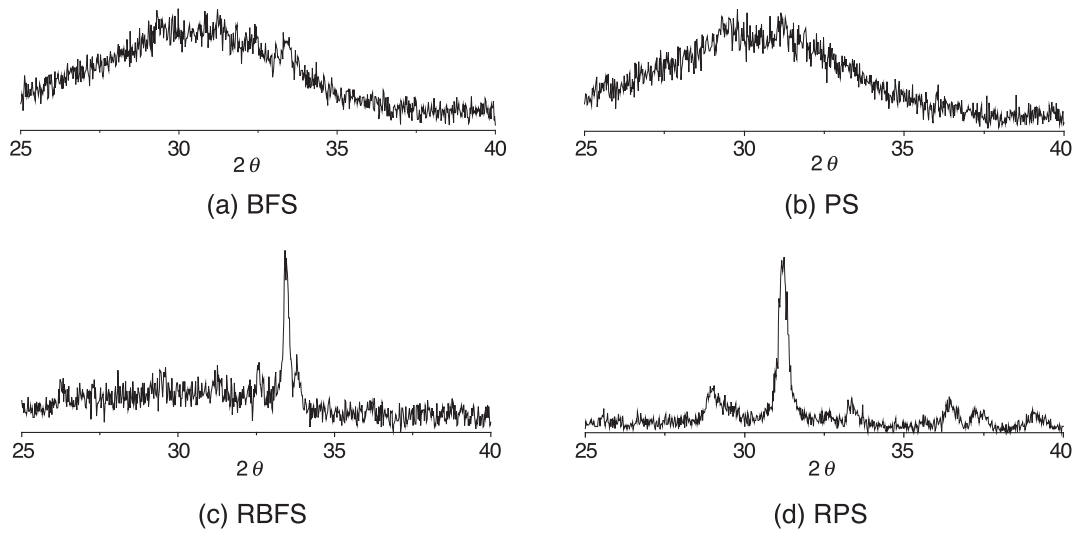


Fig. 2. XRD patterns of the powders.

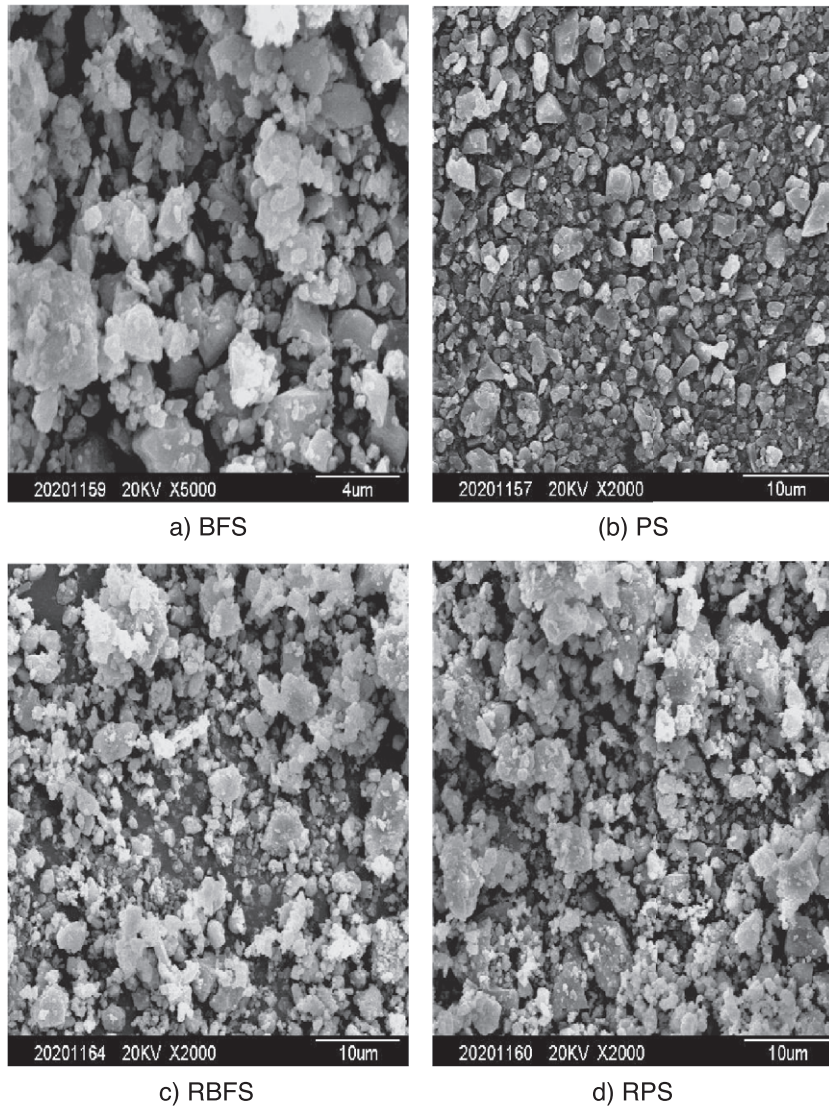


Fig. 3. SEM observations of the powders.

Table 2

Mix proportion of concrete (kg/m<sup>3</sup>)

No.	Cement	Powder	Powder content (%)	Water	Fine aggregate	Coarse aggregate	HRWR	$R_{28}$ (MPa)			
								With BFS	With PS	With RBFS	With RPS
1	460	0	0	170	685	1100	4.2	59.2			
2	414	46	10	170	685	1100	4.2	60.9	59.5	58.4	56.9
3	368	92	20	170	685	1100	4.2	58.8	58.0	57.2	55.6
4	322	138	30	170	685	1100	4.2	57.5	56.9	55.7	53.8
5	276	184	40	170	685	1100	4.2	56.1	54.8	54.3	51.6
6	230	230	50	170	685	1100	4.2	53.9	52.4	51.1	48.7

by determining the slope and the intercept of the shear stress–shear rate curve, using the least squares.

### 3. Experimental results and discussion

#### 3.1. Concrete fluidity

The slump and slump flow of the concrete with varied contents of each type of powder are given in Figs. 5 and 6. It can be seen that the changes in the fluidity of the concretes were closely related to the types of powders and their contents.

With BFS or PS, the slump and slump flow increased in proportion to the powder content. At 50% powder content, the former reached 252 or 260 mm, and the latter reached 465 or 472 mm. In the case of RBFS and RPS, although slump and slump flow also exhibited an increase, the increase was obviously smaller as compared with BFS or PS.

The results suggest that the fluidity of the concrete depends on the types of powders and their vitreous contents. As discussed in our previous reports [4,5,7], vitreous

powders can have a dispersion effect and increase greatly the fluidity of concrete through adsorbing superplasticizer on their surfaces and forming electric double layers. Therefore, the fluidifying effect of fine mineral powders should not be considered to be only a microfilling effect. These experimental results confirm also that the dispersion effect is more effective in enhancing the fluidity of concrete in some cases, and is related to the vitreous phase.

#### 3.2. Rheological properties of matrix mortars

As soon as the fluidity measurement was conducted, a part of each batch of fresh concrete was wet sieved on a vibratory sieve. Then, the rheological properties of the matrix mortar just separated out were measured promptly with the viscometer. The shear stress–shear rate curves obtained are shown in Fig. 7. It can be seen that the curves shift downwards as the powder contents increase. Notably, the curves for BFS and PS shifted more than those for RBFS and RPS, even at the same powder contents.

The plastic viscosity and yield stress of the mortars were determined using the least squares, and are shown in Figs. 8 and 9. It can be seen from Fig. 8 that for each type of powder, the plastic viscosity decreased progressively as the powder content increased. However, in the case of RBFS and RPS, the decrement of plastic viscosity was smaller than



Fig. 4. Picture of Model35 viscometer.

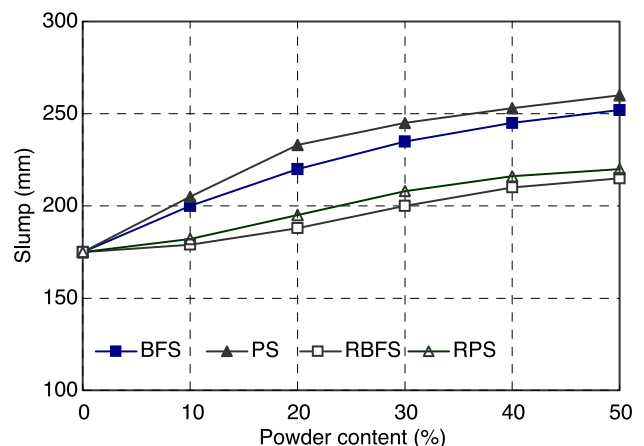


Fig. 5. Effect of powder content on slump.



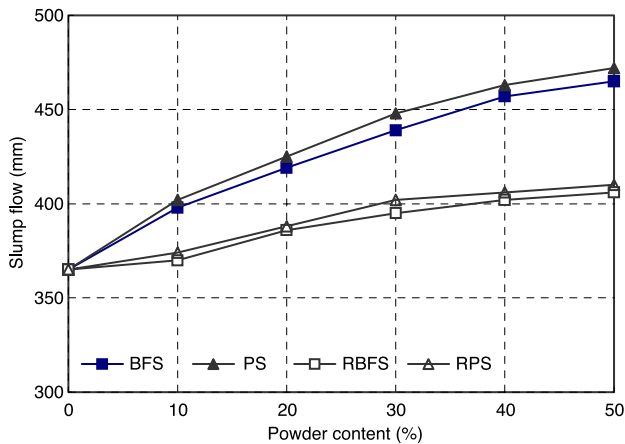


Fig. 6. Effect of powder content on slump flow.

with BFS and PS. It can be postulated from the results that the vitreous content can be a contributing factor to decreasing the viscosity.

The yield stresses of the mortars are shown in Fig. 9. The mortar with PS had the lowest yield stress at each replacement level, and the second lowest was with BFS. However, those with either RBFS or RPS exhibited a significant increase in yield stress as compared with BFS or PS.

These experimental results suggest that the rheological properties of the mortar can also be related to the vitreous contents of the powders. The decrease of the vitreous content can weaken the effect in lowering the viscosity and yield stress.

### 3.3. Correlation between rheological parameter and fluidity of concrete

#### 3.3.1. Slump versus plastic viscosity and yield stress

The correlations between slump and viscosity, as well as yield stress, are shown in Fig. 10. It can be seen that as the viscosity decreases, concretes with BFS or PS showed a higher increment in slump than those with RBFS or PS. That is, the concrete with BFS or PS had a higher fluidity than that with RBFS or RPS, even if they had the same plastic viscosity. The slump of concrete incorporating BFS or PS seems also have had a tendency to decrease with increasing yield stress, but in the case of RBFS or RPS, a clear relation between slump and the yield stress cannot be seen.

#### 3.3.2. Slump flow versus plastic viscosity and yield stress

The correlations between slump flow and plastic viscosity, as well as yield stress, are shown in Fig. 11. Such

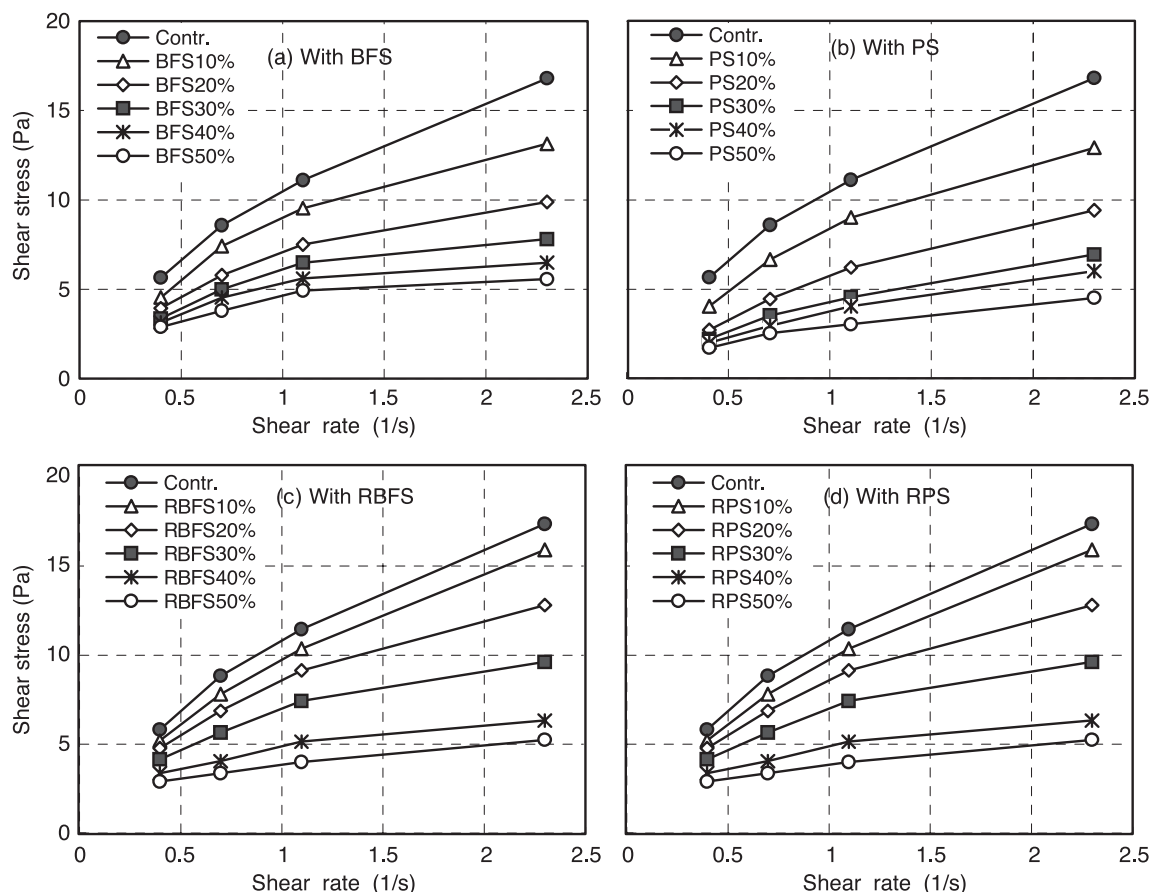


Fig. 7. Shear stress–shear rate curve of matrix mortar.

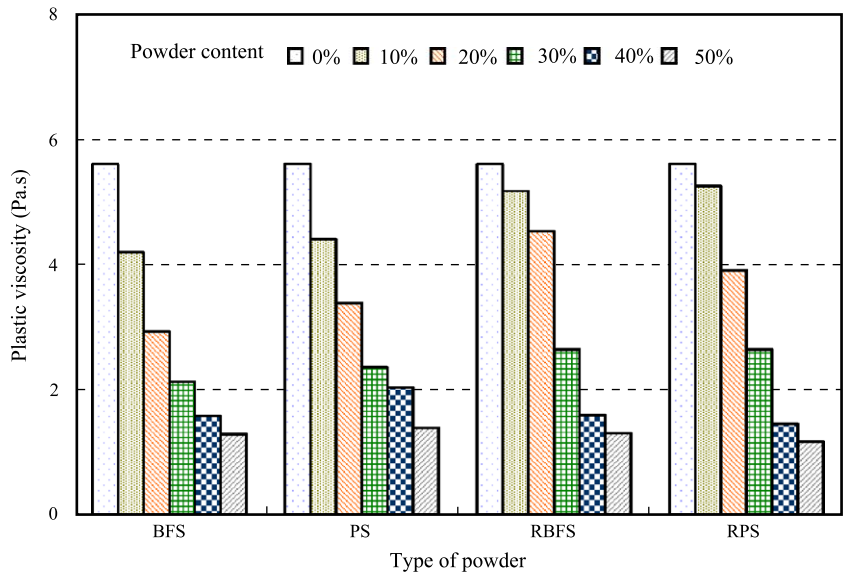


Fig. 8. Plastic viscosity of matrix mortar.

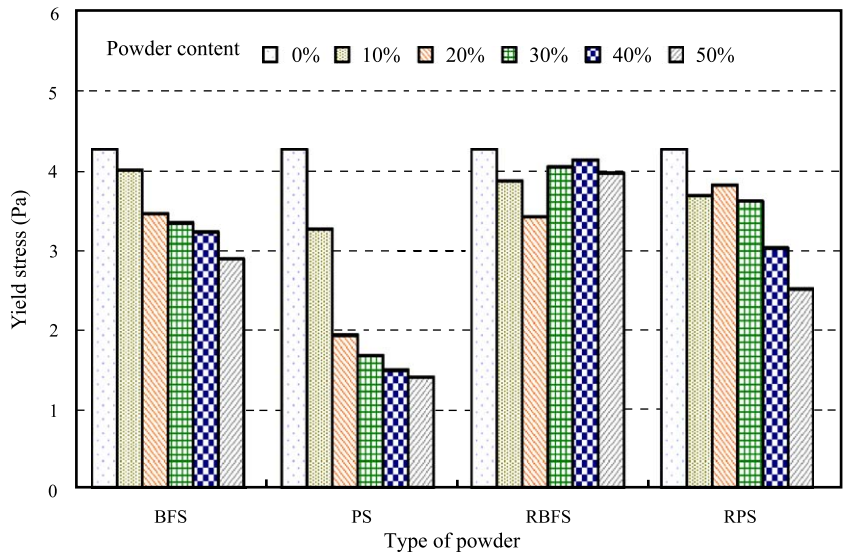


Fig. 9. Yield stress of matrix mortar.

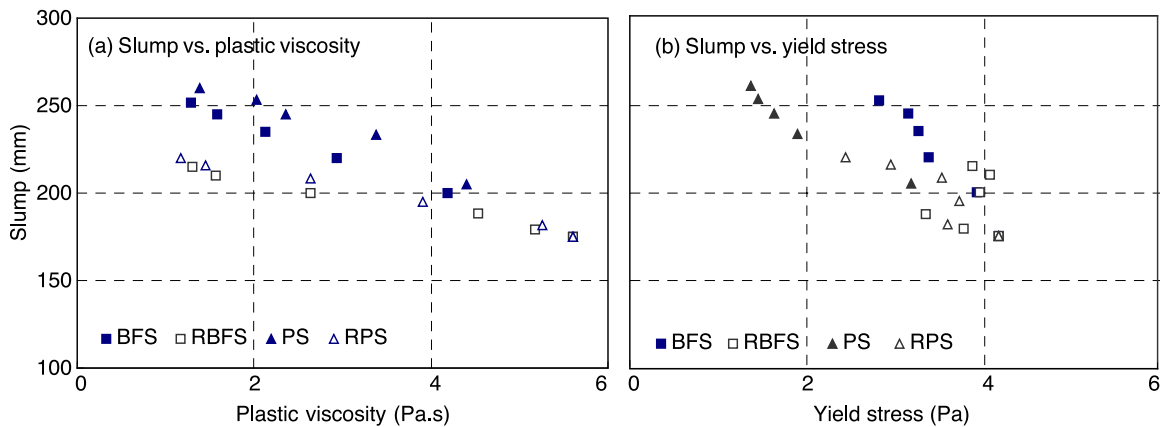


Fig. 10. Correlation between rheological parameter and slump.

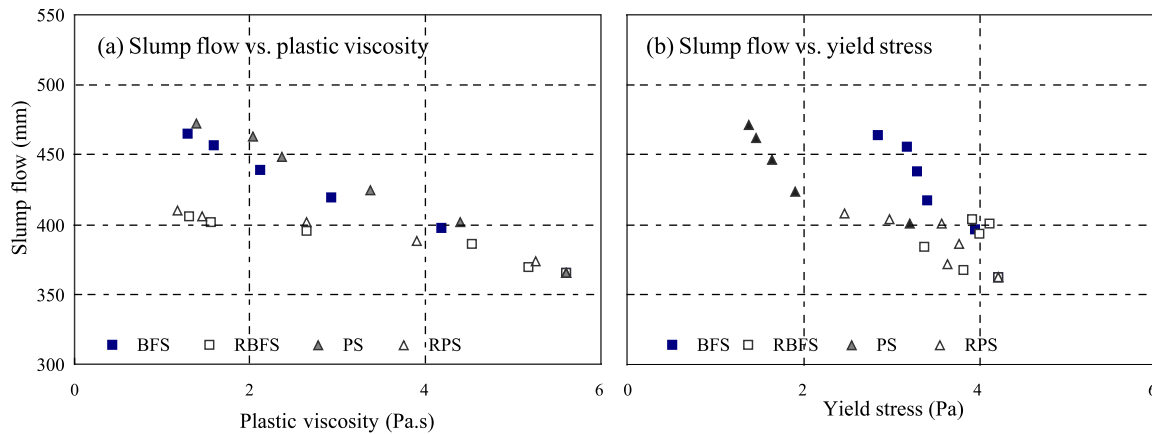


Fig. 11. Correlation between rheological parameter and slump flow.

correlations are similar with those of slump versus viscosity, as well as yield stress, shown in Fig. 10. It can be seen in Fig. 11a that with BFS and PS, the relationships between slump flow and plastic viscosity were more linear as compared with the case of RBFS and RPS. In other words, slump of concrete incorporating BFS or PS decreased more significantly with an increase of the viscosity than those of concrete with RBFS and RPS. In Fig. 11b, the slump flow of concretes with BFS and PS showed a steep drop as the yield stress increased; however, they did not seem to be closely correlated in the case of RBFS and RPS.

From the results above, we can suggest that the relationship between the fluidity of concrete and the rheological parameter can be notably influenced by incorporating varied powders with different vitreous contents. This may be attributed to the surface chemical and physical effects, which are also associated with the vitreous contents of the powders.

The effects of fine mineral powders on the properties of fresh concrete depend on various factors such as surface activity, specific surface area, size distribution, surface feature, and particle shape of the powders, as well as the types of cement, HRWR, etc. Therefore, there are many things about the behavior of the powders that remain to be interpreted further.

#### 4. Conclusions

- (1) Vitreous powders can play a dispersion effect, which can effectively enhance the fluidity of the concrete incorporating the powder. The effect can be associated with the vitreous content of the powders. A decrease of the vitreous content results in a corresponding weakening of the dispersion effect.
- (2) The plastic viscosity and yield stress of the matrix mortar can be significantly decreased by partially substituting the cement with vitreous powders. The semicrystalline powders, although only at the higher

substitution levels, can also achieve a noticeable effect in lowering the viscosity.

- (3) The correlation between the fluidity of the concrete and the rheological parameters of the mortar can also be changed by incorporating powders with different vitreous contents. The correlation with the vitreous content was found to be more linear as compared with the case of the semicrystalline content.
- (4) The behavior of fine mineral powders in concrete depends not only on their own surface activity, but also on the cement chemical composition, as well as the admixture types, etc. Therefore, the physiochemical behavior of the mineral powders in concrete has not yet achieved a thorough explanation.

#### References

- [1] L. Kucharska, M. Moczko, Influence of silica fume on the rheological properties of the matrices of high-performance concretes, *Adv. Cem. Res.* 6 (24) (1994) 139–145.
- [2] T. Nawa, Development and state of super workable concrete with emphasis on mechanism of self-compaction, *Cem. Concr.* 578 (1995) 10–17 (in Japanese).
- [3] M. Nehdi, S. Mindess, P.-C. Aïtcin, Rheology of high-performance concrete: effect of ultrafine particles, *Cem. Concr. Res.* 28 (5) (1998) 687–697.
- [4] Y. Shi, Y. Tanigawa, H. Mori, Y. Kurokawa, A study on effect of superfine powder on fluidity of cement paste, *Japan Concrete Institute Annual Convention, Tokyo*, vol. 7, 1997, pp. 223–228.
- [5] Y. Shi, I. Matsui, Y. Tanigawa, Effect of fine powders from distinct minerals on rheological property of fresh concrete, *Concr. Res. Tech.* 13 (2002) 11–18 (in Japanese).
- [6] Y. Shi, I. Matsui, N. Feng, Effect of compound mineral powders on workability and rheological property, *Cem. Concr. Res.* 32 (2002) 71–78.
- [7] N. Feng, Y. Shi, T. Hao, Influence of ultrafine powder on the fluidity and strength of cement paste, *Adv. Cem. Res.* 12 (3) (2002) 89–95.
- [8] H. Lian, L. Tong, E. Chen, *Micro-Analysis of Building Materials*, Tsinghua University Publishing House, Beijing, 1996, pp. 25–32 (in Chinese).