



Effect of compound mineral powders on workability and rheological property of HPC

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Abstract

The compounding effect of silica fume (SF) with phosphorus slag (PS) or limestone (LS) powder is investigated in this paper. It is found that such a compounding can achieve various rheological behavior of HPC. The compound powders of PS with SF lower plastic viscosity and yield stress of fresh concrete, but increase the slump and promote continuous flowability of concrete greatly. However, the compounding of LS with SF increases the yield stress, but decreases both slump and slump flow of concrete, although the viscosity remains broadly unchanged compared with the concrete containing LS only. It is demonstrated that rheological property can be highly correlated with the surface characteristic of each component of the compound powders. Based on the experimental work, the appropriate fractions of the components in these compounds, especially the optimum content of SF, have been suggested for improving rheological property of HPC. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

A concrete with an excellent rheological property should have a low internal resistance to flow and an appropriate cohesion against disintegration, so that the concrete can be formed easily without disjoining aggregate from the matrix [1]. A type of fine powders could influence workability and rheological property of HPC. Such an influence depends mainly on its surface characteristic, fineness and size distribution, etc. It has been demonstrated that silica fume (SF) and limestone (LS) play a filling effect [2,3]. In our previous investigation, the surface action of phosphorus slag (PS) has been discussed. It has been found that PS with a vitreous microstructure plays not only a filling effect but also a dispersion effect associated with the surface chemical action [5]. However, lots of questions will be proposed, if a compound of SF with any other type of powders, such as PS or LS, is used, e.g., what will be the influence on the workability and rheological property of HPC? What will be

the optimum contents of SF and the compound powder in concrete? And what will be optimum proportion of SF in the compound powders for making fresh concrete with a satisfactory rheological property. Moreover, the correlation between the fluidity of fresh concrete and rheological parameters of matrix mortar remains to be interpreted.

In order to obtain an insight into these problems, in this paper, an experimental investigation was conducted to identify the effect of compounding SF with PS or LS.

2. Experimental

2.1. Materials

Ordinary Portland cement in accordance with China National Standard, produced by Beijing Liulihe cement plant, with Blaine fineness of 3200 cm²/g, was used in this investigation. The chemical composition of the cement is given in Table 1. SF, together with either PS or LS, was used as mineral admixture. Their compositions are also given in Table 1. PS was formerly granulated industry phosphorus slag, as a by-product from the manufacture of yellow

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Table 1
Chemical analyses of cement and fine powders

	OPC	SF	PS	LS
CaO	60.85	0.31	43.75	53.60
SiO ₂	21.38	94.3	36.08	2.08
Al ₂ O ₃	5.9	1.81	4.97	1.22
Fe ₂ O ₃	2.86	0.54	2.88	0.62
MgO	3.45	0.18	1.71	1.15
Na ₂ O	0.17	—	—	—
K ₂ O	0.82	—	—	—
SO ₃	2.6	0.23	0.18	—
P ₂ O ₅	—	0.16	0.60	—
LOI	1.68	1.74	0.82	42.54

phosphorus. The previous study has demonstrated that PS was mainly composed of vitreous phase beyond 82%, similar to blast furnace slag [5]; LS was made from limestone, which was mainly composed of the crystalline calcite. PS and LS were with Blaine fineness of 8500 and 8200 cm²/g, respectively, and the size distribution is shown in Fig. 1. SF used was from Guizhou province of China, with Blaine fineness of 1.8×10^5 cm²/g, the size distribution is listed in Table 2. In this investigation, when SF together with PS or LS shorthand for the symbol PS+SF or LS+SF was used.

The fine aggregate used was river sand with fineness modulus of 2.6. The coarse aggregate was river gravel with the nominal size of 5–31.5 mm.

A powdered high range water reducer (HRWR) of naphthalene-based was used, of which the Na₂SO₄ content is <5% by mass.

2.2. Experimental method and procedure

The mix proportion of concrete with SF or compound powders are shown in Tables 3 and 4. In order to clarify the influence of the powder contents, the binder amount of 500 kg/m³ was kept constant for each batch, but with the powder content changing only. For each batch, slump flow (spread diameter of concrete) during 15 and 30 s after the slump cone lift up and slump was measured, respectively. Then samples were sieved on a 10-mm sieve. The throughs (regarded as matrix mortar comparatively)

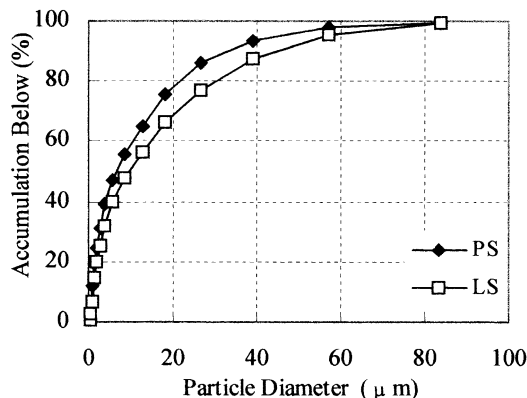


Fig. 1. Size distribution of PS and LS.

Table 2
Size distribution of SF

Particle size (μm)	Accumulation below (%)
<0.04	26
<0.08	57.3
<0.16	87.1
<0.28	99.6

were used on sample taken from the mixture for the rheological measurement.

The rheological apparatus used in the experiment was called Model 35 Viscometer, with the coaxial outer cylinder and rotating inner bob. Both inner bob and outer cylinder can be chosen from the four diameter sizes, respectively, and 12 rotating speeds of bob are available with respect to sample condition.

In the present experiment, rotational torques applied to the matrix mortar were measured for various rotational speeds, 0.9, 1.8, 3, 6 and 30 rpm, etc. Since fresh cement mortar coincides with Bingham flow, the plastic viscosity (η) and the yield stress (τ_0) can be obtained by working out the slope and the intercept of the shear stress–shear rate curve, with the linear regression calculation.

For a clear understanding of the interaction between powder and cement, the morphology of the paste samples (regarded as paste comparatively) at 20 min after initial contract of cement with mixing water were collected for microscope observation by means of a scanning electron microscope (SEM) of model HITACHI S-450.

From each batch, three of 150-mm cubes were prepared in accordance with Chinese National Standard GB50204-92 for test of compressive strength at curing age of 28 days, the cubes were demoulded at 24 h after moulding, then put in the standard curing room (temperature 20 ± 2 °C, higher than 95% of relative humidity).

3. Experimental results and discussion

3.1. Workability of the concrete

The measured slump values of concrete with SF or compound powders as partial replacement for cement are given in Fig. 2. It can be seen that an appropriate content of SF can increase concrete fluidity; the SF content for maximum fluidity is 6% and that for secondly higher fluidity is

Table 3
Mix proportion of concrete with SF, kg/m³

No.	Cement	SF	Water	Fine aggregate	Coarse aggregate	HRWR	R ₂₈ (MPa)
1	500	0	170	650	1110	4.5	72.4
2	485	15	170	650	1110	4.5	75.8
3	470	30	170	650	1110	4.5	77.9
4	455	45	170	650	1110	4.5	81.1
5	440	60	170	650	1110	4.5	83.6

Table 4

Mix proportion of concrete with compound powders, kg/m³

No.	Cement	PS or LS	SF	Water	Fine aggregate	Coarse aggregate	HRWR	R_{28} (MPa)	
								PS or PS + SF	LS or LS + SF
1	350	150	0	170	650	1110	4.5	74.8	55.2
2	350	135	15	170	650	1110	4.5	76	57.9
3	350	120	30	170	650	1110	4.5	78.5	62.4
4	350	105	45	170	650	1110	4.5	82.9	68.1
5	350	90	60	170	650	1110	4.5	85.1	72.7

9% (Fig. 2a). Fluidity was also promoted by PS 27%+SF 3% and PS 24%+SF 6% (Fig. 2b). However, concrete with various contents of LS+SF have lower slump than that containing LS alone (Fig. 2b). This result indicates that SF did not favor the fluidity effect of LS.

Fig. 3 shows the slump flow of concrete with SF or compound powders. Similar to the case in Fig. 2a, concrete achieved the highest slump flow value at the content of 6% SF (Fig. 3a). Although slump flow with SF did not enlarge typically, it showed the clear change tendency with SF content. In Fig. 3b, the compound PS+SF increases slump flow obviously at the contents of 27%, 24% and 21% of PS with 3%, 6% and 9% of SF, respectively. On the other hand, the concrete with LS+SF has a less slump flow value than that with LS only (Fig. 3b). This result also confirmed that

either combination of SF and LS, or LS alone, did not help the continuous flow of concrete.

3.2. Influence of SF and compound powders on rheological property of matrix mortar

Based on the rheological measurement results of matrix mortar, relationship between shear stress (Pa) to shear rate (1/s) was shown in Fig. 4. From the comparison of curves in Fig. 4a, it is clear that the curves of the mortars shift downwards with the increase of SF content, except the curve with SF content of 12% is over the control. In Fig. 4b for PS+SF, the curves with the compound powders are all below the control, however, the curve with PS 18%+SF 12% is the most close to the control one. In Fig. 4c for

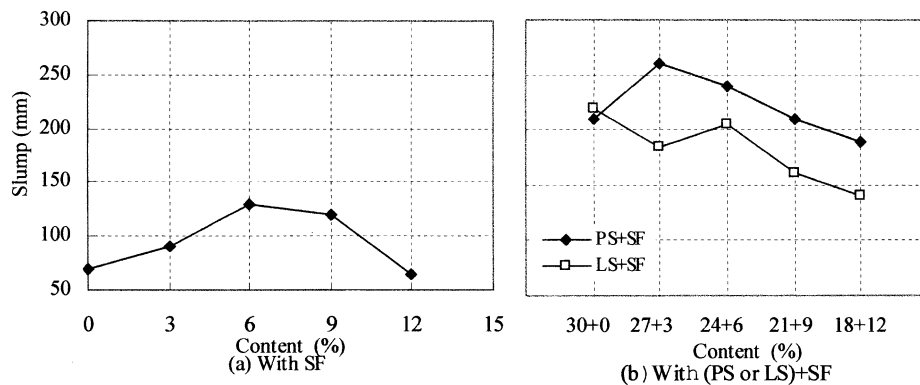


Fig. 2. Slump of concrete with SF or compound powder.

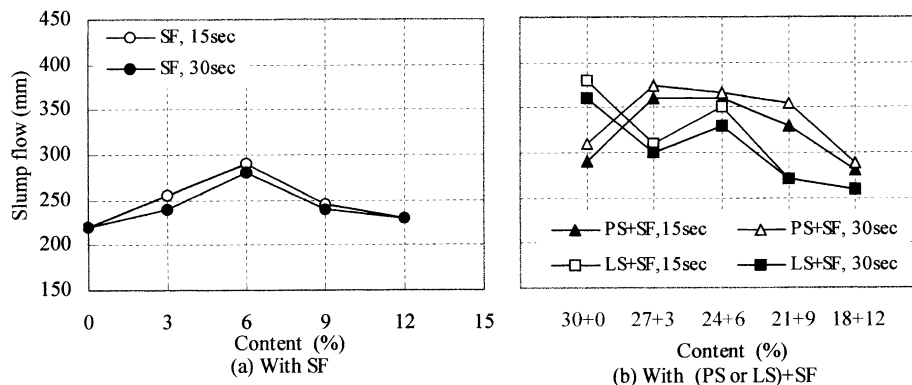


Fig. 3. Slump flow of concrete with SF or compound powder.

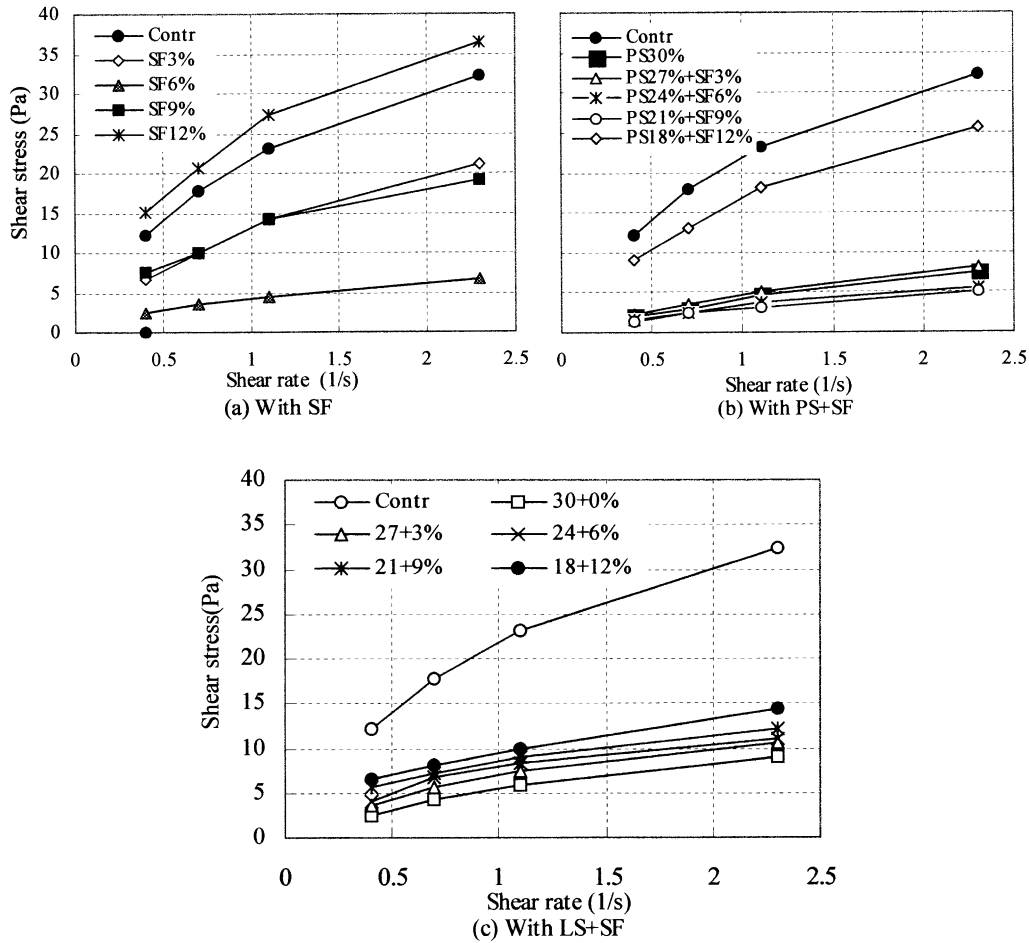


Fig. 4. Rheological property of matrix mortar.

LS + SF as well as the control, the curves with the compound powders shift downward obviously. All these results show that rheological property of the mortars was characterized by blending distinct powders.

With the linear regression calculation, plastic viscosity and yield stress of the mortars calculated from Fig. 4 are shown in Figs. 5 and 6. From Fig. 5a, it is found that the viscosity of the mortar decreased with increase of SF

content, and to the minimum value at SF content as 9%, but increased suddenly at the content of SF up to 12%. This phenomenon indicated that there was an optimum content of SF for the lowest viscosity, which should depend on the space between cement particles. The results in Fig. 5b suggested that compounding of SF with PS was beneficial to lowering the viscosity. In other words, SF promoted the dispersion effect of PS. However, in the case of LS + SF, the

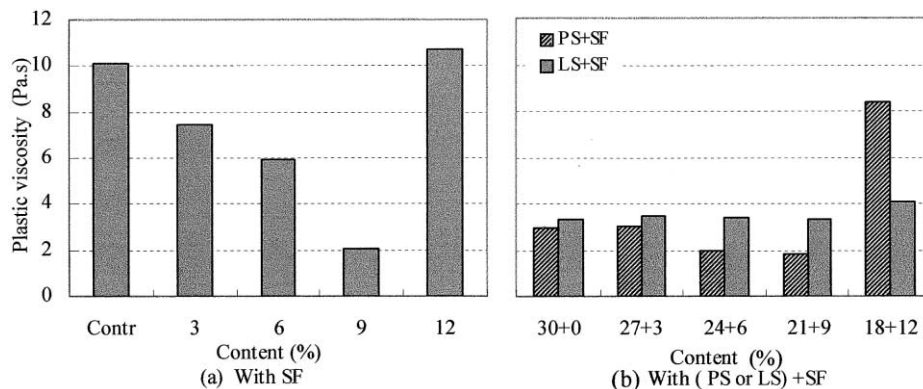


Fig. 5. Plastic viscosity of matrix mortar.

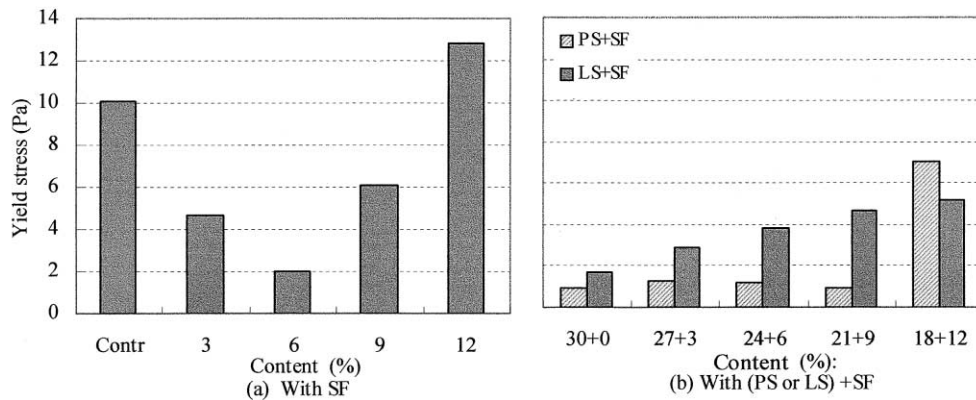


Fig. 6. Yield stress of matrix mortar.

viscosity values of the mortars with various contents of LS + SF were approximately unchanged compared with that of the mortar containing individual LS, except for a slight increase at the content LS 18% + SF 12%. Fig. 6 shows the influence of SF and the compound powders on yield stress of the mortar. SF could lower yield stress, the most remarkably lowering occurred at 6% of SF. But the stress value increases abruptly at the content up to 12% (Fig. 6a). It can be seen from Fig. 6b that the combination of SF with PS has only a slight effect on yield stress, as compared to the case of PS employed only; but for the combination LS + SF, yield stress increases gradually with an increase of SF fraction. These results also confirmed that the influence of the combination of SF with vitreous powders is quite different from that with nonvitreous powders.

3.3. Correlation between plastic viscosity and slump of concrete

Taking reciprocal of plastic viscosity as fluidity parameter ($1/\eta$), the correlation between fluidity parameter and slump, drawn out from Figs. 2 and 4, was shown in Fig. 7. The concrete with SF had a maximum slump at fluidity parameter about 0.17 (viscosity value 6 Pa s), while either with

PS + SF or with LS + SF, maximum slump appeared at fluidity parameter 0.33 (viscosity 3 Pa s). Apart from this maximum, fluidity of concrete with SF or PS + SF decreases gradually with an increase of the viscosity. Nevertheless, no regular correlation can be found from concrete with LS + SF. The behaviors of concrete with distinct compound powders can be related to the interaction between cement and powder particles. It has been found in our previous reports that vitreous PS can adsorb superplasticizer molecules and form the electrical double layer on its surface, thus, produces a dispersion effect in the paste [5], which gives a driving force to concrete for flowing. Furthermore, spherical SF particles can produce a lubricating effect to minimize the mechanical interlock between cement particles as well as that between aggregates [4,6] and enable the fresh concrete to flow. Thus, the combination of SF with a vitreous powder leads to a doubling effect on concrete fluidity. But the particles of nonvitreous LS, unlike to PS, without adsorbing superplasticizer, are joined to surface of cement particles and form a coagulation state, which is not easier to flow automatically. Actually, the flow of concrete depends both on the positive effect and the negative effect, the former promote fluidity, such as dispersing, filling and lubricating, the latter restricts fluidity, such as formation of particle coagulation, an increase of wettable surface and mechanical interlock, etc. Therefore, it is speculated that, although with a lower viscosity, positive effect in fresh concrete with LS + SF is not enough to overcome the negative effect for flowing.

3.4. Correlation between viscosity and slump flow

Fig. 8 shows the correlation between fluidity parameter and slump flow. Fig. 8a is the case with slump flow of 15 s, and Fig. 8b is with slump flow of 30 s. The correlation with the former is similar to the case of slump shown in Fig. 7. With a comparison between Fig. 8a and b, it can be seen that the fluidity of concrete containing PS + SF has a higher increase at 30 s over that at 15 s, and there is a clear correlation between fluidity parameter and slump flow value, which confirms that a combination of dispersion

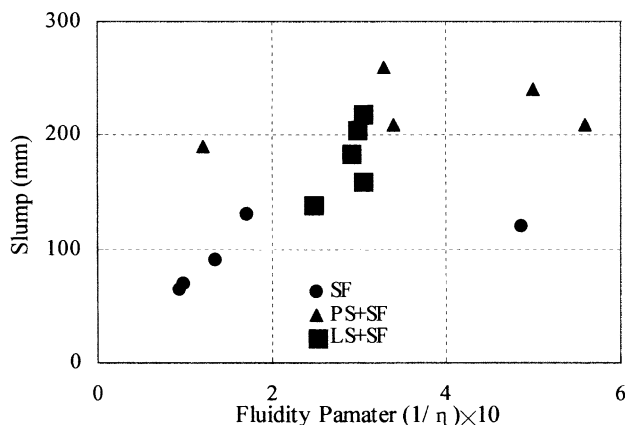


Fig. 7. Correlation between fluidity parameter and slump.

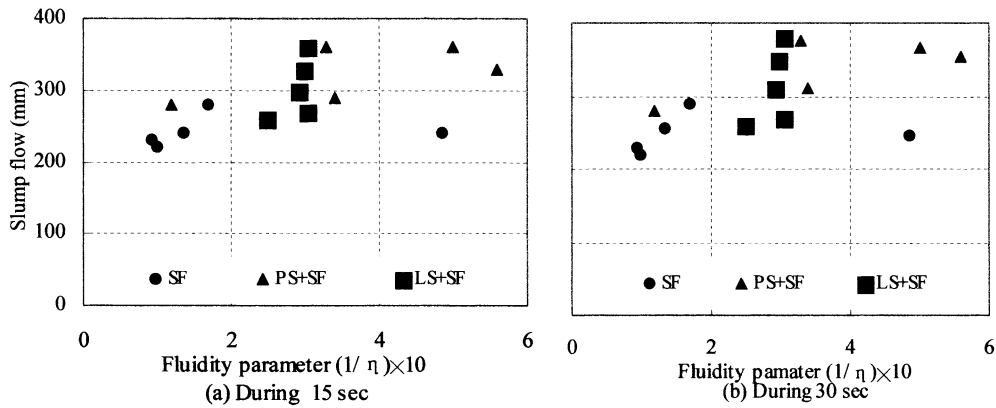


Fig. 8. Correlation between fluidity parameter and slump flow.

effect (PS) with lubricating effect (SF) can give concrete a continuous flow. While a combination of filling effect (LS) with lubricating effect (SF) gives concrete too small driving force to overcome coagulation structure. In this case, initial flow at 15 s is mainly due to the own gravity of concrete rather than the dispersion effect. As a result, fluidity of concrete during the interval from 15 to 30 s was very low, because the own gravity became smaller than that at the initial 15 s.

From the present experiment results, a clear correlation between the yield stress of the matrix mortar and the slump flow remains to be proved further.

3.5. Microstructure of the paste samples

In Fig. 9 are morphology observations of SF, PS and LS. It can be seen that SF (a) are very small spherical grain and some of them present in a gathering state. However, PS (b) and LS (c) are with irregular shapes and the sizes are in a wide range. PS seems to be with smoother surface than LS.

The SEM observations on the samples with or without powders are shown in Fig. 10. Fig. 10a for the control paste,

the cement particles with irregular shapes and various sizes were almost in mechanical interlocking state against each other. Obviously, such interface hinders cement particles from removal. As there was still considerable space between cement particles, some mix water could thus be detained in this space, lowering fluidity of cement paste. Fig. 10b shows the paste sample containing SF 6%. In this case, numerous small spherical particles of SF were distributed within the spaces between cement particles, eliminating the interlocking of cement grains and making the grain “mobile.” This action can be regarded as a lubricating effect of SF. Meanwhile, the filling of SF could release the captured water, consequently favoring the fluidity of the paste (or concrete). As for the paste with PS 24%+SF 6% shown in Fig. 10c, cement particles were separated more significantly by the dispersion effect of PS with filling effect of SF, leading to cement particles more liable to freely moving. For LS 24%+SF 6% shown in Fig. 10d, powder particles seemed to cause a flocculation state, which might limit flow of cement particles to some degree. This phenomenon seemed to be correlated with the morphology of nonvitreous LS. These SEM observations revealed that the microstructure of

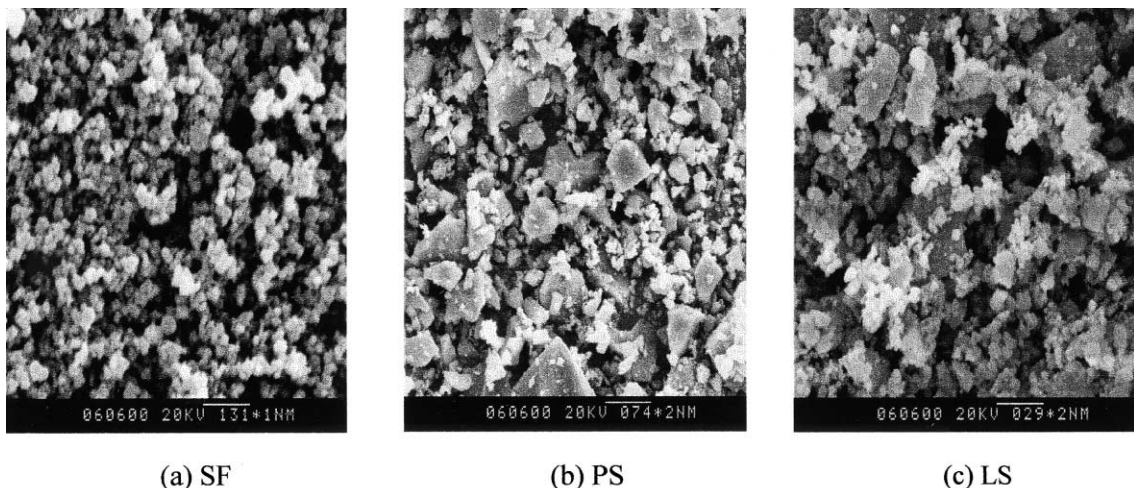


Fig. 9. SEM observation of the powder samples.

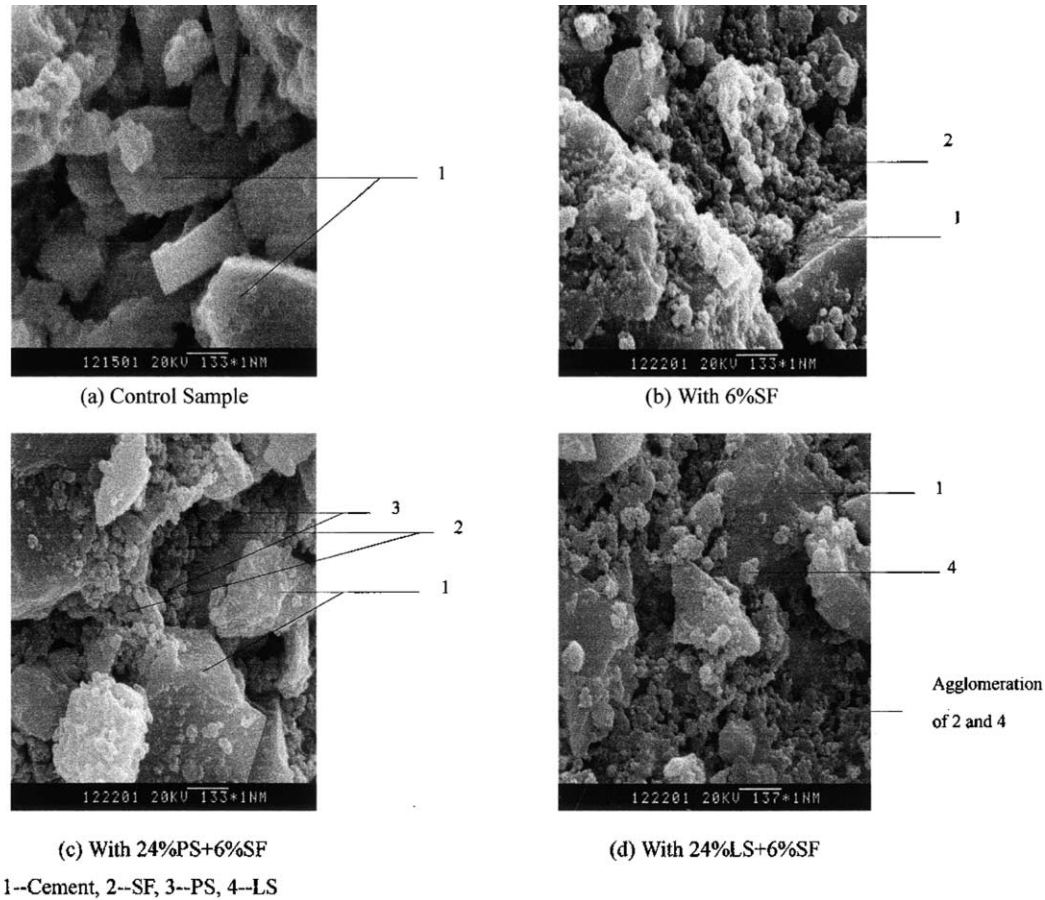


Fig. 10. SEM observation of the paste samples (1, cement; 2, SF; 3, PS; 4, LS).

paste could be characterized by different interface actions (the physical and the chemical) as well as morphology effect between cement and powder particles.

The role of the fine powders in fresh mortar or concrete is a synthesis of the surface chemical and physical effects. It should be noted that SF/PS/LS also affect water/HRWR demand by different rate of adsorption of water and HRWR; so it is not only physical effect. The behavior of LS is also dependent on the types of HRWR. There are still many things about the interactions between the fine powder and cement as well as HRWR that deserve further attention.

4. Conclusions

(1) Partial replacement of cement with SF can improve the fluidity and rheological property of HPC. In this investigation, the optimum content of SF to decrease the plastic viscosity of matrix mortar was 9%, while that to give the lowest yield stress and the highest fluidity was 6%. However, when SF content was up to 12%, plastic viscosity and yield stress became the maximum, and concrete had lowest fluidity.

(2) The combination of SF with the dispersing effect of PS generated a doubling effect, which can greatly increase

the fluidity of concrete, and help concrete flow continuously, i.e., enhance homogeneity, thus improved the rheological property of HPC more significantly. Nevertheless, although the combination of SF with LS decreased the plastic viscosity of matrix mortar, such a type of compound powders did not increase fluidity of concrete. This phenomenon was correlated to distinct interface action (the physical and the chemical) between cement and the powder.

(3) The SEM observations revealed that microstructure of the pastes was characterized by blending distinct powders. SF could eliminate the mechanical interlock between cement particles, while the compound PS + SF could separate the cement particles more effectively than the PS alone. But, the compound LS + SF might form an agglomeration over the spaces between cement particles. This distinct interaction could lead to a different fluidity or rheological property of HPC.

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