

极低水胶比下含钢渣粉复合浆体和含石灰石粉复合浆体的流变性能

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摘 要: 石灰石粉和钢渣粉作为辅助性胶凝材料广泛应用于现代混凝土中, 但是极低水胶比下石灰石粉或钢渣粉对复合水泥浆体流变性能的影响仍缺乏深入研究。通过测定流动度、凝结时间、Marsh 筒流动时间、流变性能和总有机碳含量, 对石灰石粉和钢渣粉在极低水胶比下对复合浆体流变性能的影响进行了比较研究。结果表明, 添加石灰石粉的复合浆体的流动度损失率低于添加钢渣粉的复合浆体。与添加钢渣粉相比, 添加石灰石粉显著缩短了复合浆体的凝结时间和 Marsh 筒流动时间, 且添加石灰石粉的复合浆体具有更好的流变性能和更强的触变性。添加钢渣粉的复合浆体的总有机碳含量低于添加石灰石粉的复合浆体, 但添加石灰石粉的样品中自由水含量更高。与添加钢渣粉相比, 在极低水胶比下, 石灰石粉对复合浆体流变性能的影响更有利。

关键词: 复合浆体; 石灰石粉; 钢渣粉; 流变性能; 极低水胶比

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1 Introduction

The rapid development of the global science and technology contributes to an annual increase in carbon dioxide emissions, leading to severe greenhouse effects. Concrete is one of the most important civil engineering materials. However, Portland cement is a main component of concrete and a main source of carbon emissions. According to the related statistics, the annual carbon emissions of the global cement industry are as high as 2.8 billion tons, accounting for 5%–7% of the global anthropogenic CO₂ emissions^[1–2]. Nearly 60% of the world's cement is produced in China, and the carbon emissions of the cement industry account for more than half of the world's carbon emissions. Reducing the amount of cement is thus an effective approach to decrease the carbon emissions of concrete. Supplementary cementitious materials (SCMs) can be used to displace cement in part^[3–5]. Conventional SCMs are usually common industrial solid wastes, for instance, fly ash and ground

granulated blast furnace slag (GGBFS). However, the supply of fly ash and GGBFS is limited with the reduction in coal-fired power generation and iron/steel production and large-scale infrastructure construction. At present, steel slag powder (SSP) and limestone powder (LP) have attracted much attention as SCMs due to their diverse sources, low cost and economic benefits.

SSP is an industrial solid waste produced in the process of steelmaking^[6]. The main mineral constituents of SSP include C₂S, C₃S, C₄AF and C₂F^[7–8], which have gelling properties. SSP can be thus used as a typical SCM for producing concrete. To date, SSP is used to produce concrete, indicating the practicability of using SSP as SCM. Han *et al.*^[9] reported that the activity of steel slag was poor, and the hydration heat of cement containing SSP within 168 h was lower than pure Portland cement. A high dosage of SSP had a retarding effect on the hydration of Portland cement. Fan *et al.*^[10] designed a ultra-high performance concrete (UHPC) based on the high

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goodness of fit attribute predictive regression model of Doptimal design (DOD) via a numerical optimization method, and they reported that adding SSP could enhance the workability of UHPC, the maximum slump reached 268 mm. Calmon *et al.* [11] investigated the effect of converter SSP on the rheological properties of self-compacting cement paste, and found that the β_p value (i.e., the amount of water required to trigger flow in the mixture) after mixing converter SSP with water was lower than that when mixing other fillers with water, and adding converter SSP met the self-compacting concrete design standard. Han *et al.* [12] found that SSP improved the long-term performance of 5-year-old concrete at a low water/binder ratio. The increasing dosage of SSP increased the compressive strength, reduced the porosity, and improved the resistance to chloride ion penetration of 5-year-old concrete.

LP is an industrial by-product of limestone mining and processing and has abundant reserves [13-15]. LP has filling, nucleation, dilution and chemical effects, which can promote the nucleation of C-S-H gel [16-18]. Dong *et al.* [19] substituted LP and calcined clay with different replacement rates for Portland cement to prepare UHPC. They reported that the hydration of cement dominated at low replacement rates and only a single peak could appear on the hydration heat curve, whereas when the replacement rates of LP and calcined clay were higher than 40%, two heat release peaks appeared. Increasing the fineness of LP could promote the hydration of cement and form gel C-(A)-S-H. Varhen *et al.* [20] studied the effect of replacing limestone filler with cement on the rheological properties of micro-concrete used for repairing coating. They reported that the larger the volume of limestone filler in the paste was, the lower the yield stress and the higher the slump value would be. However, the plastic viscosity increased with increasing limestone filler content. Benaicha *et al.* [21] investigated the effects of LP and silica fume on the rheological and mechanical properties of self-compacting concrete and found that the compressive strength and Young's modulus of self-compacting concrete decreased, the slump increased, the V-shaped funnel flow time decreased, and the yield stress and plastic viscosity

decreased as LP content increased. Burroughs *et al.* [22] used fine ground LP to replace some or all of the cement and silica fume, and reported that the replacement of cement with LP had a notably positive effect on the workability of UHPC.

Some studies investigate the mechanical properties and microstructure of composite paste when mixing SSP or LP with other mineral admixtures. However, in UHPC and self-compacting concrete, an extremely low water/binder (W/B) ratio is a key factor in achieving ultra-high strength and ultra-low porosity of the materials, but it also leads to the deterioration of the rheological properties of the fresh paste. However, the rheological properties of the fresh paste affect the workability of cement-based materials during the later pouring and molding processes, as well as the mechanical properties and durability after hardening [23]. In the existing studies, the influence of LP and SSP on the rheological properties of cement paste at extremely low W/B ratios is rarely discussed. The mechanism of the effect of SSP or LP on the rheological properties of composite paste at low water/binder ratios is still unclear. There is little work on the influence of LP or SSP on the rheological properties of composite paste. Typically, only one method is adopted to investigate the rheological properties of composite paste. In this paper, the flow diameter, setting time, marsh cone flow time, rheological parameters and total organic carbon (TOC) content of composite paste with different dosages of LP or SSP were thus determined. The effects of contents of LP and SSP on the rheological properties of composite paste at extremely low W/B ratios were investigated and their action mechanisms were analyzed, providing a theoretical basis for the application of LP and SSP in materials such as UHPC at extremely low W/B ratios.

2 Experimental

2.1 Raw materials

PI 42.5 Portland cement (China United Cement Group Co., Ltd., China) GGBFS and SSP (Shandong JinTaicheng Construction Technology Co., Ltd., China), LP (Henan Yixiang New Material Co., Ltd., China) and silica fume (Gansu Sanyuan Silicon

Materials Co., Ltd., China) were used as raw materials. Table 1 shows the chemical compositions of the raw materials.

Fig. 1 shows the particle size distributions of raw materials. The critical pore sizes of the cement, GGBFS, LP and SSP are 30, 20, 9 μm and 7 μm , respectively. The particle size distributions of the LP

and SSP are similar, and their particles are finer than those of the cement, but there are still some coarser particles ranging from 200–300 μm in size in the SSP (see Fig. 1 (a)). However, the particle size distribution of silica fume differs from that of other materials, and its critical pore size is 545 nm (see Fig. 1 (b)). Fig. 2 shows the XRD pattern of the raw materials.

Table 1 Chemical composition of the raw materials

w/%

Composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	CaCO ₃	MgO	SO ₃	Na ₂ O _{eq}	f-CaO	Loss	Other
Portland cement	21.18	4.73	3.41	62.49		2.53	2.83	0.56	0.72	1.76	
Ground granulated blast furnace slag (GGBFS)	34.55	14.36	0.45	33.94		11.16	1.95	0.63		0.70	2.26
Limestone powder (LP)	8.43	2.39	1.41		82.85	3.28	0.10	0.8			0.74
Steel slag powder (SSP)	12.77	2.12	23.49	49.17		3.54	0.23	0.45		1.86	6.37
Silica fume (SF)	99.9										0.01

w is mass fraction.

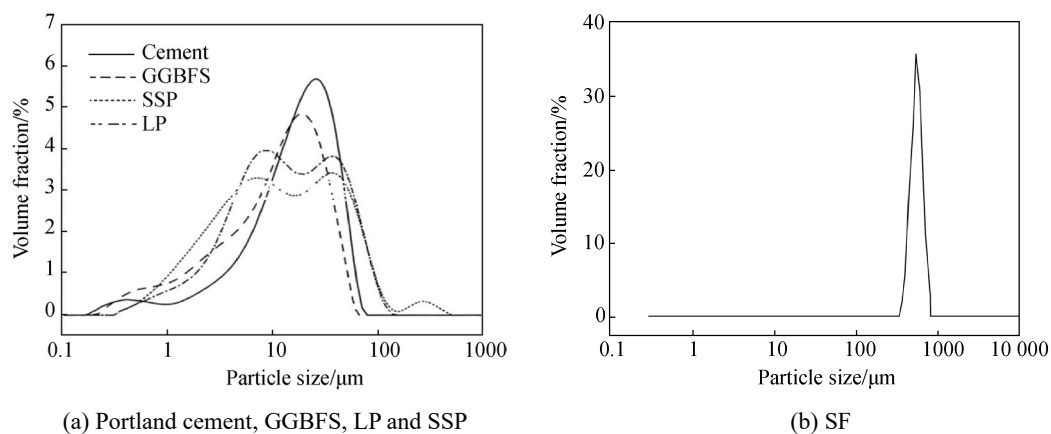


Fig. 1 Particle size distributions of the raw materials

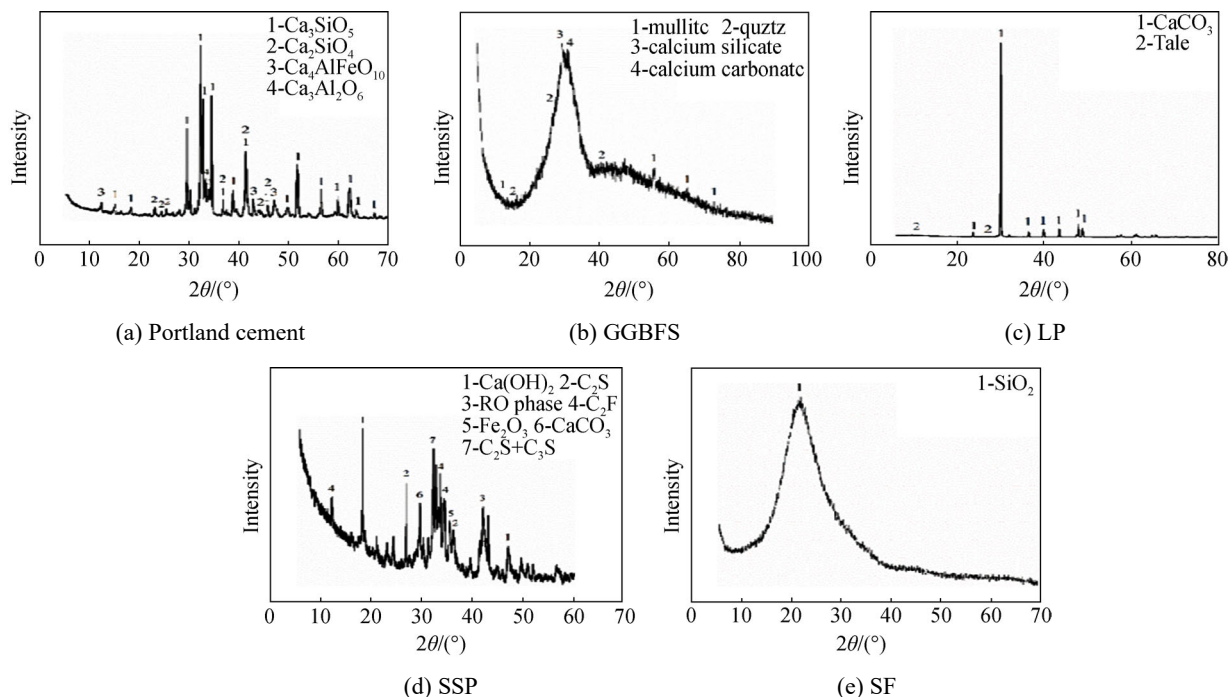


Fig. 2 XRD patterns of the raw materials

In this study, a polycarboxylate superplasticizer with a water-reducing rate of 40% (Jiangsu Subot New Materials Co., Ltd., China) was used. The dosages of the polycarboxylate superplasticizer in the test groups with W/B ratios of 0.16 and 0.21 were 2% and 0.8%, respectively. The fluidity of composite pastes was adjusted *via* adding polycarboxylate superplasticizer.

2.2 Preparation of composite pastes

Table 2 shows the mix ratios of composite pastes. The Portland cement and silica fume contents of each sample were 50% and 10%, respectively. A comparative test was conducted at different contents of SSP (or LP) and GGBFS (the total content of both is 40%). The dosages of polycarboxylate superplasticizer

were 2% and 0.8% for the composite paste at W/B ratios of 0.16 and 0.21, respectively.

For the preparation of paste, the materials were mixed in a cement paste mixer. A certain mass of polycarboxylate superplasticizer was weighed and dropped into deionized water and stirred evenly for 30 s. The cementitious materials at different mix ratios (see Table 2) were placed in the mixing pot and stirred at a low speed for 30 s to mix the different powders evenly. Afterwards, a water solution containing polycarboxylate superplasticizer was added and stirred at a low speed for 3 min. The paste was scraped on the inner wall of the stirring pot and stirred at a high speed for 3 min to ensure the homogeneity of the composite paste with a low W/B ratio.

Table 2 Mix ratios of the composite pastes

w/%

Samples	Cementitious material content					Water reducing agent	Water/binder (W/B) ratio
	Portland cement	Silica fume	GGBFS	SSP	LP		
P0	50	10	40	0	0	2.0/0.8	0.16/0.21
PL1	50	10	30	0	10	2.0/0.8	0.16/0.21
PL2	50	10	20	0	20	2.0/0.8	0.16/0.21
PL3	50	10	10	0	30	2.0/0.8	0.16/0.21
PS1	50	10	30	10	0	2.0/0.8	0.16/0.21
PS2	50	10	20	20	0	2.0/0.8	0.16/0.21
PS3	50	10	10	30	0	2.0/0.8	0.16/0.21

The composite pastes with different mix ratios were put into a plastic beaker, sealed after each test, and then placed into a standard curing box with $(20 \pm 1)^\circ\text{C}$ and 95% relative humidity for curing, respectively.

2.3 Characterizations

The flow diameters of the paste were measured at 5 (initial fluidity), 30, 60, 120, 180, 240, 360 min and 480 min, respectively. A glass plate and a mini-slump were prepared. The glass plate and the mini-slump were wiped with a wet towel to ensure a moist appearance without water droplets. The mini-slump was placed in the center of the glass plate. The evenly mixed paste was poured into the mini-slump, and the surface was subsequently smoothed with a scraper. When the mini-slump was lifted slowly and evenly, the maximum diameters of the flows to the two directions of the vertical phase were tested immediately after

30 s, and the average value was taken as the flow diameter of paste.

The prepared paste was put into the test mould, the height of sample was 40 mm. The sample was put into a standard curing box for curing. The sample was removed, and the initial setting needle was installed on a vicat apparatus after the initial setting time. The height of the initial setting needle was adjusted, so that the tip of the initial setting needle was just close to the upper surface of the paste. The initial setting needle was released, so that it fell freely, and the falling height of the vicat apparatus needle was recorded after 30 s. When the needle sank to 2–3 mm away from the bottom plate, the sample reached the initial setting state. The final setting time was measured every 15 min (or less) after the final setting time. The final setting needle was used, and the height was adjusted, so that the final setting needle was just close to the

surface of sample, and then the final setting needle was loosened. The composite paste reached the final setting state when the needle was sunken 0.5 mm into the test sample or when the ring attachment began to fail to leave traces on the sample.

A marsh cone was used to measure the flow time of paste at 5, 60, 120 min and 240 min, respectively. All the pastes were fully mixed according to the same stirring process. The lower outlet of the marsh cone was plugged tightly, and the inner wall was wiped with a wet cloth. 300 mL paste was poured into the marsh cone, and the lower outlet was opened. The time required for the paste to finish flowing in the marsh cone was recorded.

The rheological properties of the composite paste were measured by a model R/S rheometer (Brookfield Co., USA). For the pre-shearing experiment, the shear rate was firstly changed from 0 to 5 s^{-1} at the shear time of 25 s, and then the shear rate was diminished from 5 s^{-1} to 0 at the shear time of 25 s. Afterwards, the static time was 10 s. The purpose of pre-shearing was to fully disperse the particles in the paste. The static time was 10 s to ensure the stability of the paste before the test. In the formal experiment, the shear rate was set from 0 to 60 s^{-1} at the shear time of 60 s. The shear rate was reduced from 60 s^{-1} to 0 at the shear time of 60 s. The test time was 5, 60, 120 min and 360 min after the contact between the water and cementitious material. The rheological data were collected by a software named Rheo3000 (Brookfield Co., USA). The yield stress (τ_0) and plastic viscosity (μ) of the paste were obtained via the Herschel-Bulkley model.

TOC is the total amount of organic matter in the system expressed by the carbon content, and the result is expressed by the mass concentration of carbon (C) (mg/L). The molecular structure of polycarboxylate superplasticizer contains the polar group, which can be adsorbed on the surface of cementitious material particles through electrostatic adsorption, chemical bonds or hydrogen bonds. When the polycarboxylate superplasticizer in the fresh paste is transferred from the liquid phase (*i.e.*, aqueous solution) to the solid phase (*i.e.*, the surface of cementitious particles),

which leads to a decrease in the content of organic carbon in the liquid phase. The adsorption capacity of the water reducing agent on the surface of the cementitious material is thus obtained *via* measuring the difference in the organic carbon contents of the solution before and after adsorption. For the preparation of samples, the cementitious materials were prepared according to different mixing ratios (see Table 2). The cementitious materials were thoroughly stirred with the water solution containing a water reducing agent and then stood for 20 min to simulate the adsorption process of the water reducing agent. The paste was centrifuged at 3000 r/min for 3 min, and then diluted the supernatant by 10 times with deionized water to obtain the test solution. The organic carbon content (C_1 , mg/L) in the solution was determined by a model TOC instrument. Also, 0.4 g of polycarboxylate superplasticizer was dropped into 100 g of deionized water. The initial value of the superplasticizer before adsorption (C_0 , mg/L) was tested, and the adsorption capacity of the superplasticizer was calculated by

$$\text{TOC} = \frac{(C_0 - C_1) \times V}{m} \quad (1)$$

where V is solution volume (L) and m is the quality of cementitious materials (g).

Moreover, a centrifugal free water content test was conducted to confirm the adsorption effect of the water reducing agent. The water centrifuged out in a centrifuge is the free water content in the paste.

3 Results and discussion

3.1 Flow diameter

Fig. 3 shows the flow diameter of the composite paste with LP or SSP at a W/B ratio of 0.16. In Fig. 3(a), the initial flow diameter of the paste increases with increasing LP content. When the dosages of LP are 10%, 20% and 30%, the loss rates of the flow diameter of the composite paste within 480 min are 2.14%, 4.14% and 5.94%, respectively. The LP particles are smaller than the cement particles. At a low dosage, LP mainly plays a role in physical filling and releasing free water to improve the fluidity of the paste. At the dosage of 30%, a great deal of LP can

reduce the nucleation barrier and promote cement hydration^[24-25], due to the growth of the loss rate of the flow diameter.

In Fig. 3(b), adding SSP significantly improves the initial flow diameter of the composite paste. When SSP contents are 10%, 20% and 30%, the loss rates of flow diameters within 480 min are 6.23%, 7.37% and 7.64%, respectively, which are significantly higher than the loss rates of the control group (*i.e.*, 2.46%). SSP hydrates more slowly in the early stage, but the water requirement content of SSP is larger. SSP has an irregular shape, a rough surface and contains large

particles of RO (CaO-FeO-MnO-MgO solid solution) phase, which increase the frictional resistance between particles, leading to a decrease in fluidity. Moreover, it is not conducive to the accumulation and filling of voids by particles to release free water. Therefore, the content of free water in the paste containing SSP is relatively low, resulting in a rapid decrease in the fluidity of the paste containing SSP. As the dosage of SSP and the rest time increase, the free water in the paste consumes in significant quantities, resulting in a notable increase in the loss rate of the flow diameter.

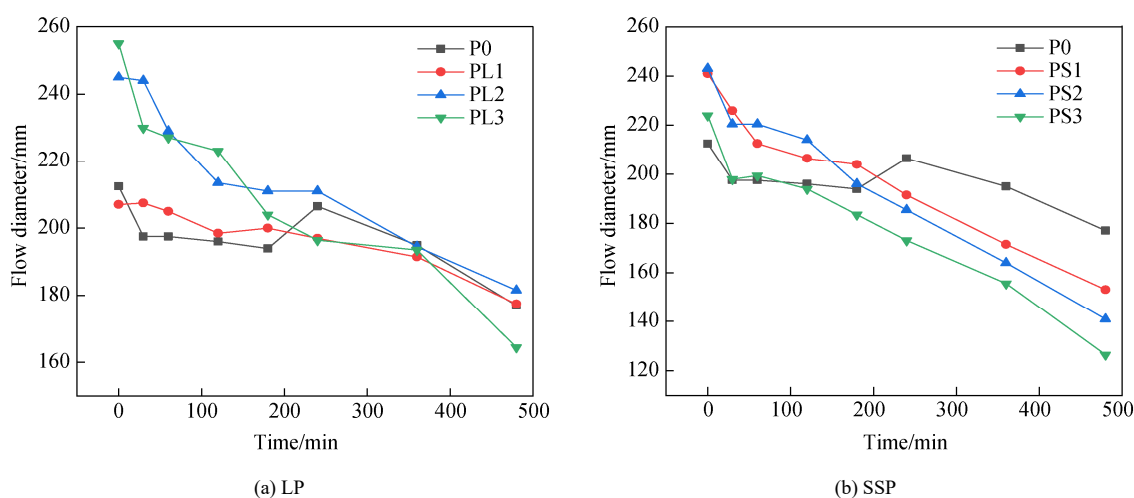


Fig. 3 Flow diameters of the composite pastes at a W/B ratio of 0.16

Fig. 4 shows the flow diameters of the composite pastes with LP or SSP at a W/B ratio of 0.21. In Fig. 4(a), adding LP increases the initial flow diameter of the paste, and the loss rate of the flow diameter reduces with increasing the LP dosage. The flow diameters loss rates of samples with different additions of 10%, 20% and 30% within 480 min are 11.87%, 10.70% and 8.46%, respectively. A high water content increases the initial flow diameter of the composite paste at a W/B ratio of 0.21, and the filling effect of LP is conducive to the release of free water in the voids. Moreover, LP possesses a water-reducing effect^[26], which can slightly reduce the flow diameter loss rate.

In Fig. 4(b), the initial flow diameter of the composite paste is slightly improved *via* adding 10% and 20% SSP. The flow diameter decreases with increasing SSP dosage and prolonging time. When the

contents of SSP are 10%, 20% and 30%, the loss rates of the flow diameter within 480 min are 9.81%, 10.56% and 11.80%, respectively, which are higher than the loss rate of the control group (*i.e.*, 7.16%). The water requirement of SSP gradually increases as the content of SSP increases. The increased frictional resistance between particles and the loose packing structure all lead to a decrease in the content of free water. Therefore, the fluidity of the paste reduces more rapidly.

LP can improve the initial fluidity of paste, the improvement of SSP on the initial flow diameter of composite paste is not as great as that of LP. In general, the reducing effect of LP on the flow diameter of composite paste is lower than that of SSP at the same dosage for a long rest time. In Figs. 3 and 4, the initial flow diameter of the composite paste at a W/B ratio of 0.16 is basically smaller than that of the

composite paste at a W/B ratio of 0.21 at the same dosage. The composite paste at a W/B ratio of 0.16 contains more water reducing agents, which has an

effect of retarding coagulation. Therefore, the loss of flow diameter of the composite paste at a W/B ratio of 0.16 is relatively slow.

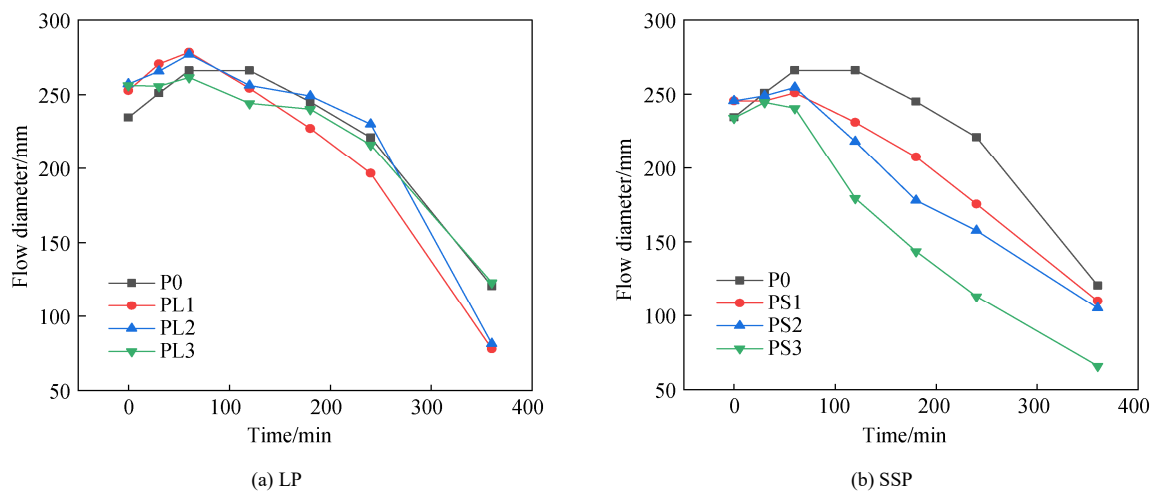


Fig. 4 Flow diameter of the composite paste at a W/B ratio of 0.21

3.2 Setting time

Fig. 5 shows the setting time of the composite paste with LP or SSP at a W/B ratio of 0.16. In Fig. 5(a), the initial setting time decreases with increasing LP dosage. Compared with the control group, the initial setting time is decreased by 67, 130 min and 136 min at the LP contents of 10%, 20% and 30%, respectively, elucidating that the hydration rate of binder can be accelerated with increasing LP content. Compared with the control group, the initial setting time is diminished by 43 min and 32 min at the SSP contents of 10% and 20%, respectively. However, at the SSP content of 30%, the initial setting time is

increased by 40 min, which demonstrates that the high dosage of SSP delays the early hydration of binder, which is in agreement with the previous results^[27], indicating that the presence of $C_{12}A_7$ in SSP delays the early hydration of C_3S in cement, leading to a prolonged setting time.

In Fig. 5(b), the final setting times of the paste are reduced by 71, 137 min and 102 min at the LP contents of 10%, 20% and 30%, respectively. The nucleation of the LP accelerates the hydration of the binder, resulting in the generation of a greater quantity of hydration products. The final setting time thus reduces.

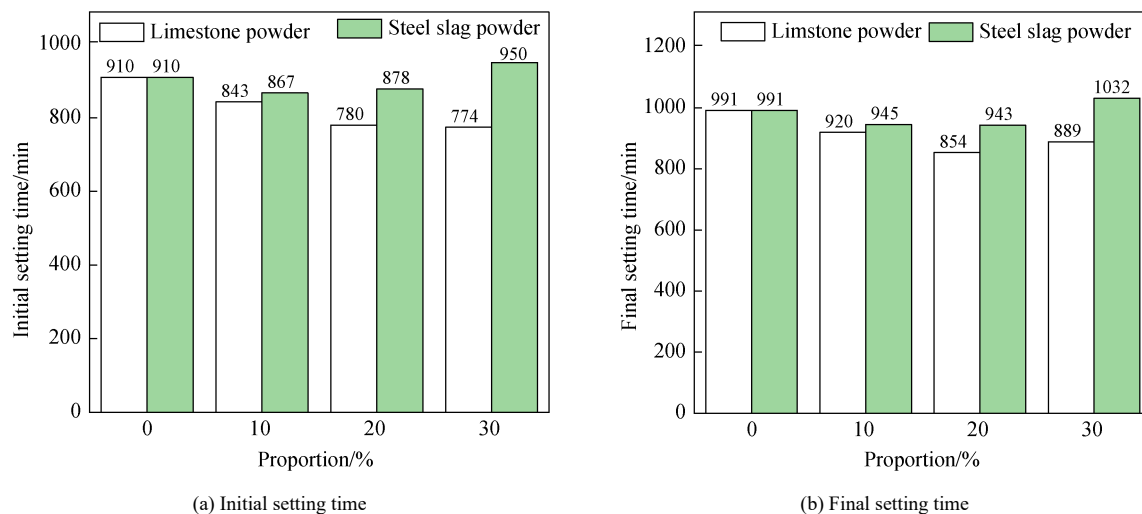


Fig. 5 Setting time of the composite paste containing LP or SSP at a W/B ratio of 0.16

The final setting times of the paste are reduced by 46 min and 48 min at the SSP contents of 10% and 20%, respectively. The $\text{Ca}(\text{OH})_2$ (CH) can be produced by the hydration of SSP, which promotes the reaction of GGBFS, and the two reactions have a synergistic effect. At the SSP content of 30%, the final setting time is increased by 41 min. Adding SSP reduces the early hydration rate of cement, and the higher dosage results in the greater retarding effect. Moreover, the low content of GGBFS weakens the synergistic effect, resulting in the inhibition of cement hydration and thus an increase in the final setting time^[27-29].

Fig. 6 shows the setting time of the composite paste with LP or SSP at a W/B ratio of 0.21. In Fig. 6(a), the initial setting time is decreased by 32, 46 min and 61 min at the LP contents of 10%, 20% and 30%, respectively. LP exerts a more pronounced enhancing effect on the hydration of binder with increasing content. Compared to the control group, the initial setting time is increased by 6 min at the SSP dosage of 10%. However, at the SSP contents of

20% and 30%, the initial setting time are decreased by 2 min and 23 min, respectively. Substantial SSP is beneficial to shortening the initial setting time at a W/B ratio of 0.21. A greater amount of CH generated by cement hydration can stimulate the activity of SSP. The reaction between CH from the hydration of SSP and GGBFS is also beneficial to improving the degree of hydration and shortening the initial setting time.

In Fig. 6(b), compared to the control group, the addition of LP at different dosages of 10%, 20% and 30% results in the reductions of 33, 34 min and 85 min, respectively. At a high LP dosage, the shortening effect of LP on the final setting time is more distinct. SSP also can shorten the final setting time of composite paste. The SSP contents of 10%, 20% and 30% lead to reductions in the final setting time of 23, 36 min and 81 min, respectively. SSP promotes the cement hydration in later stage^[27]. A higher SSP dosage contributes to a better promoting effect, thus shortening the final setting time of paste.

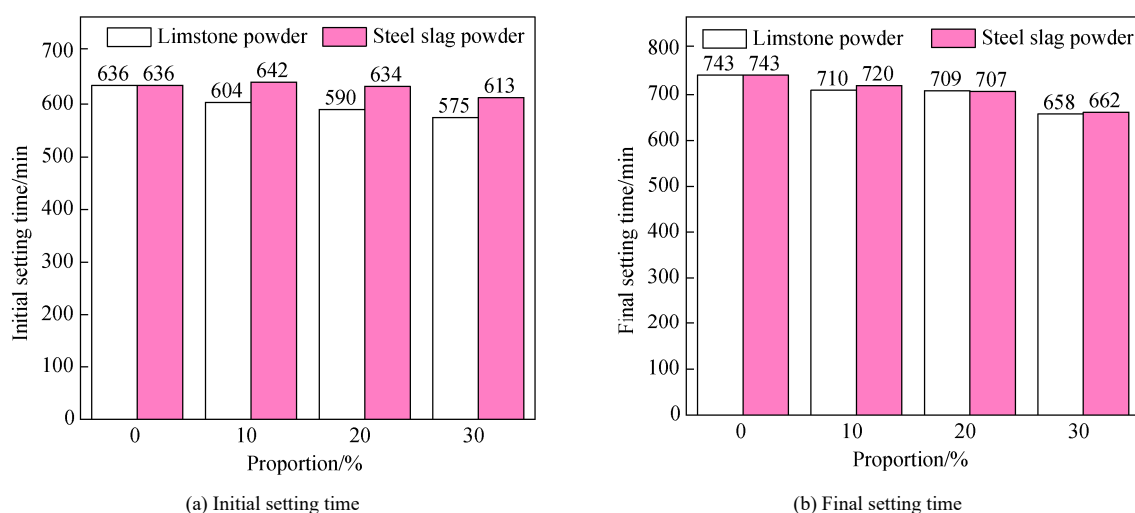


Fig. 6 Setting time of the composite paste containing LP or SSP at a W/B ratio of 0.21

The setting time of the composite paste with LP is obviously lower than that of the composite paste with SSP at the same dosage. The setting time of the composite paste at a W/B ratio of 0.21 is distinctly lower than that of the composite paste at a W/B ratio of 0.16. The polycarboxylate superplasticizer has a retarding effect. It is demonstrated that the polycarboxylate superplasticiser has a potential to form a hydrophobic protective film with the binder

particles. This hinders the process of cement hydration and prolongs the hydration induction period^[30-31], which in turn leads to the prolonged setting time. The amount of water reducing agent in the paste at a W/B ratio of 0.21 is small and the free water content is higher, which is conducive to promoting the synergistic reaction among cement, GGBFS and SSP or LP, generating more reaction products, which accelerates the coagulation and hardening of the

composite paste. Therefore, the final setting time of paste at a W/B ratio of 0.21 is shorter.

3.3 Marsh cone flow time

Fig. 7 shows the marsh cone flow time of the composite paste with LP or SSP at a W/B ratio of 0.16. In Fig. 7(a), the initial flow time of paste is increased by 25.1 s and 13.9 s at the LP contents of 10% and 20%, respectively, compared to the control group. However, the flow time is decreased by 2.6 s at the LP content of 30%. This may be related to the viscosity of paste. At the LP contents of 10% and 20%, the marsh cone flow time increases due to the increased plastic

viscosity of the composite paste, which makes the paste viscous and difficult to flow. Similarly, at the LP content of 30%, the plastic viscosity of the composite paste decreases, the fluidity of paste improves, and the marsh cone flow time shortens. In Fig. 7(b), clearly, adding SSP prolongs the flow time of paste, the flow time increases rapidly with increasing SSP dosage and prolonging rest time. SSP has a retarding effect on cement hydration. The yield stress and the plastic viscosity of the paste further increase with increasing the SSP content, resulting in a rapid increase in the flow time.

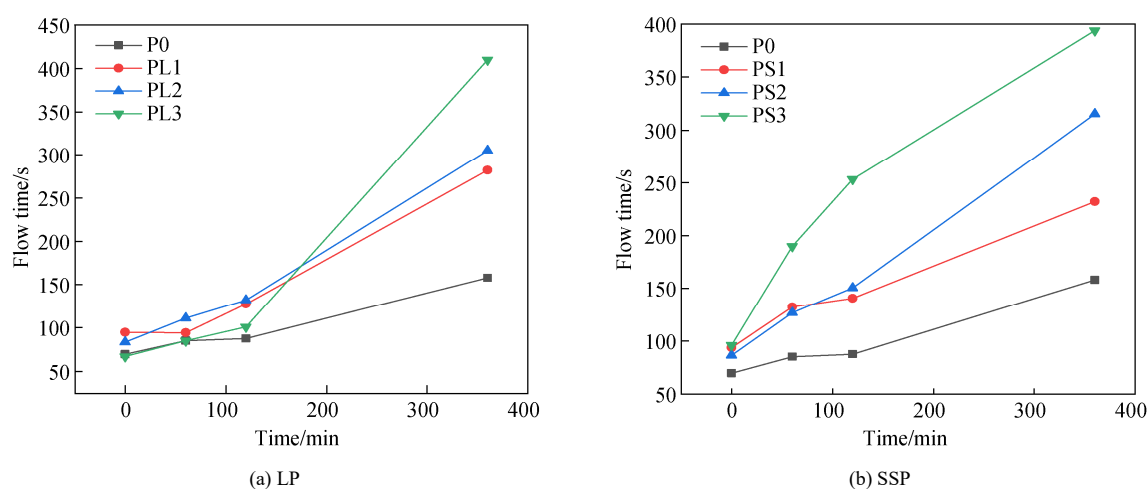


Fig. 7 Marsh cone flow time of the composite paste at a W/B ratio of 0.16

Fig. 8 shows the marsh cone flow time of the composite paste with LP or SSP at W/B ratio of 0.21. In Fig. 8(a), adding LP slightly diminishes the initial flow time of the composite paste. The paste with 20% and 30% LP stops flowing after 120 min. LP has a nucleation effect, which can effectively promote cement hydration and shorten the flow time. Compared to the control group, the flow time of the composite paste containing the LP content of 10% is increased by 14.2 s at 360 min. This may also be due to the increased viscosity. In Fig. 8(b), the flow time of the composite paste containing SSP increases with increasing SSP content within 120 min. After 120 min, the pastes with SSP contents of 20% and 30% stop flowing. Compared to the control group, the flow time of the paste with the SSP content of 10% is decreased by 8.2 s at 360 min. The flow time can be reduced

because the retarding effect is weak at a low SSP content. The hydration of SSP and synergistic reaction with GGBSF are significant as the SSP content increases, resulting in a reduction in the flow time of the paste.

LP and SSP both increase marsh cone flow time of the composite paste at a W/B ratio of 0.16. Nevertheless, the increase in the flow time of the composite paste with LP is lower than that of the composite paste with SSP. The flow time of the composite paste with LP is essentially shorter than that of the composite paste with SSP at a W/B ratio of 0.21. The marsh cone flow time of the sample at a W/B ratio of 0.21 is markedly shorter than that of the sample at a W/B ratio of 0.16. This is due to the higher free water content in the sample with a high W/B ratio, which is conducive to improving the fluidity of the composite paste.

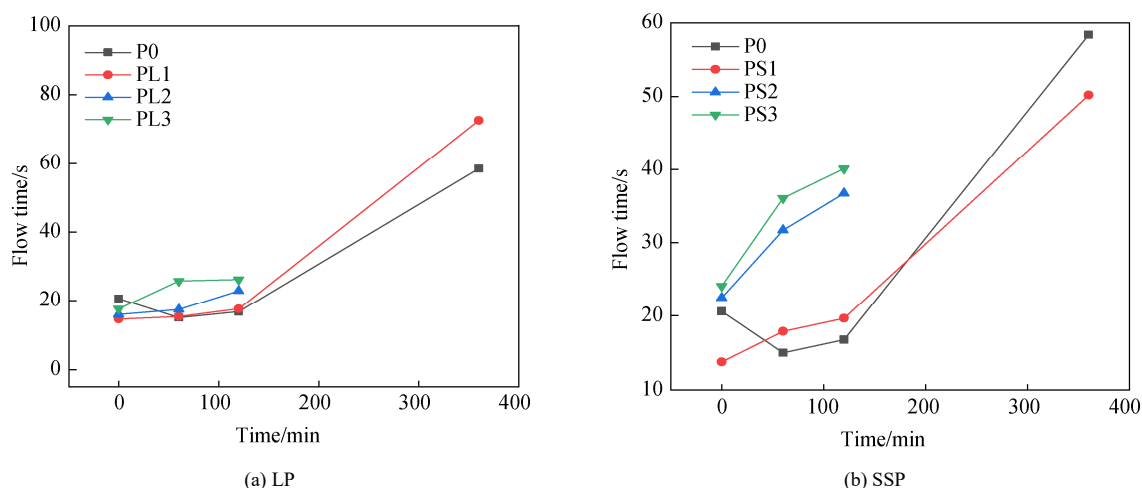


Fig. 8 Marsh cone flow time of the composite paste at a W/B ratio of 0.21

3.4 Rheological properties

Fig. 9 shows the rheological curve of the composite paste containing LP at a W/B ratio of 0.16. In Figs. 9(a)–9(d), the shear stress and thixotropic ring areas of all the samples gradually increase with increasing rest time. Table 3 shows the thixotropic ring areas. The thixotropic ring area of samples reduces with increasing LP content at 5 min. At 1, 2 and 4 h, the thixotropic ring area of the paste with LP content of 10% is larger than that of the control group, whereas the thixotropic ring areas of the pastes with LP contents of 20% and 30% are smaller than those of the control group, manifesting that a high content of LP can effectively improve thixotropic properties of paste. This is because the formation of hydration products is diminished at a high LP content, which is conducive to restoring the paste to the initial state. These results indicate that the thixotropic properties of paste can be enhanced via adding LP.

Fig. 10 shows the shear stress–shear rate curve of the composite paste containing SSP at a W/B ratio of 0.16. In Figs. 10(b) and 10(c), the final maximum shear stress reaches approximately 900 Pa at the SSP content of 10% or 20%. However, the shear stress rapidly increases to approximately 1300 Pa with increasing shear rate at SSP content of 30%. The area of the thixotropic ring increases gradually with increasing SSP content and prolonging rest time. In Table 3, the thixotropic ring area of the experimental group with SSP content of 10% is increased by

1131 mm², compared to that of the control group at 5 min. Conversely, the thixotropic ring areas of the experimental groups with SSP contents of 20% and 30% are decreased by 546 mm² and 1629 mm², respectively. The thixotropic ring areas of the paste increase significantly at 1, 2 h and 4 h. At a low SSP content, it is not easy to return to the initial state after shearing because of the uneven particle size [32], and the thixotropic properties become poor. It can delay the early hydration of cement as the SSP content increases. Thus, the thixotropic ring area decreases. An increase in rest time can promote the hydration of cement and the reaction of SSP in the subsequent stage, resulting in a notable alteration in the thixotropic ring area.

Table 3 Thixotropic ring area of the composite paste with LP or SSP at a W/B ratio of 0.16

Sample	A/mm ²			
	5 min	1 h	2 h	4 h
P0	4510	5670	5374	10 043
PL1	4084	7560	6666	12 274
PL2	2198	2993	3893	8778
PL3	1966	4170	5037	8007
PS1	5641	10 258	9097	13 105
PS2	3964	12 835	16 913	13 009
PS3	2881	27 232	44 456	26 770

A is area.

The composite paste is a multiphase mixture that exhibits complex non-Newtonian fluid characteristics. Cement-based materials at very low W/B ratios typically exhibit dilatant fluid characteristics. Therefore,

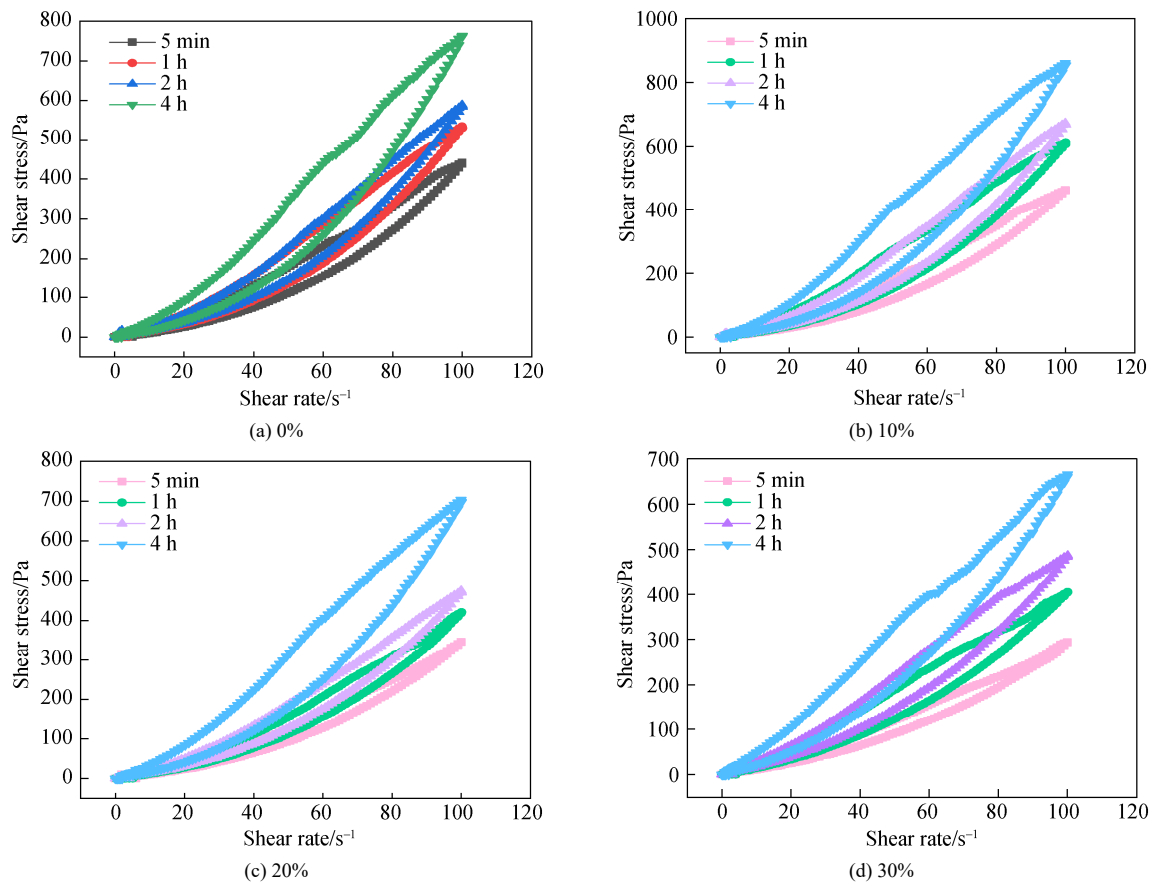


Fig. 9 Shear stress–shear rate curves of the composite pastes with different LP contents at a W/B ratio of 0.16

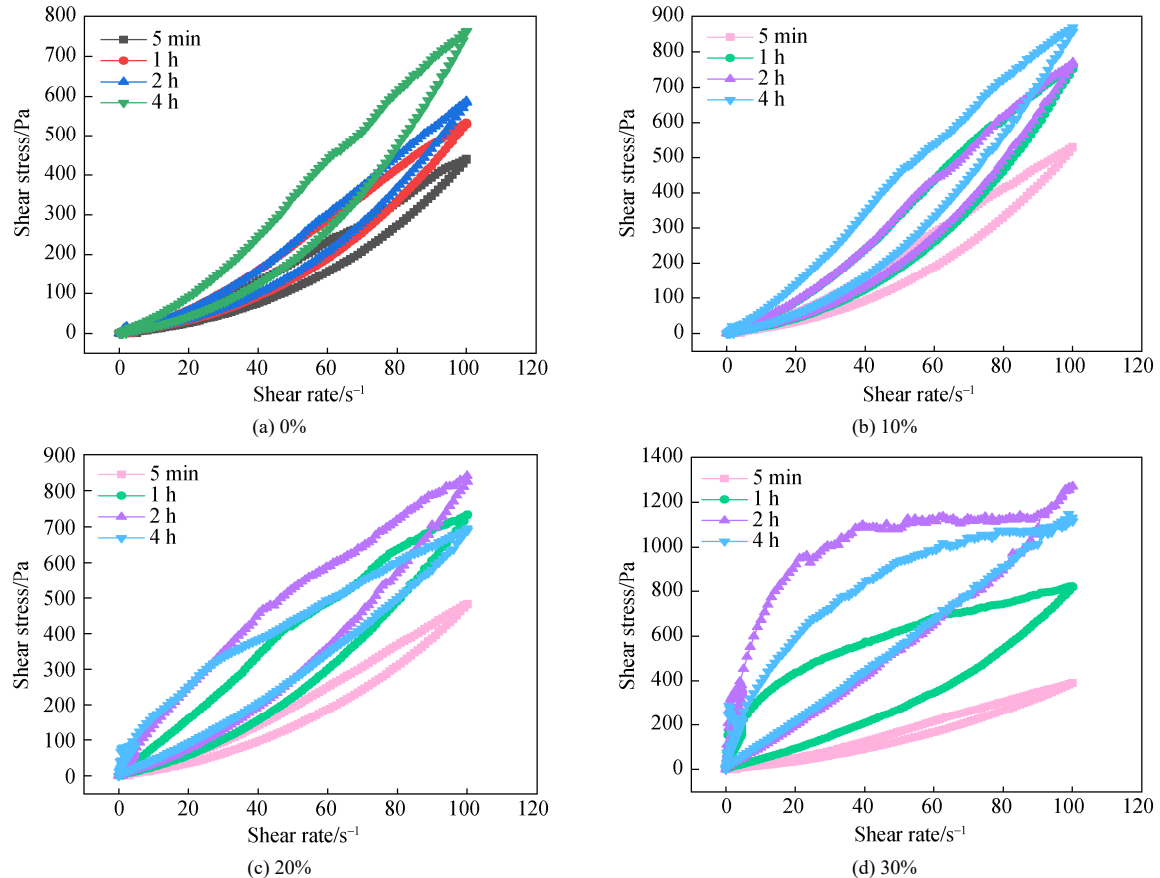


Fig. 10 Shear stress–shear rate curves of the composite paste with different SSP contents at a W/B ratio of 0.16

the rheological parameters are calculated *via* the Herschel-Bulkley model^[33–35]. The deceleration shear test results of the rheological curve of the paste are used to fit the Herschel-Bulkley model. Table 4 shows the rheological parameters of the composite paste containing LP or SSP.

Table 4 Rheological parameters of the composite paste with LP or SSP at a W/B ratio of 0.16

Sample	Test time	Yield stress (τ_0)/Pa	Plastic viscosity (μ)/(Pa·s)
P0	5 min	10.55	3.09
	1 h	13.14	3.77
	2 h	13.67	4.15
	4 h	16.09	5.34
PL1	5 min	10.63	3.27
	1 h	11.99	4.34
	2 h	12.99	4.77
	4 h	15.93	6.07
PL2	5 min	9.12	3.12
	1 h	10.75	3.24
	2 h	11.98	3.41
	4 h	14.37	5.01
PL3	5 min	7.65	2.22
	1 h	10.00	3.07
	2 h	13.23	3.63
	4 h	13.07	5.10
PS1	5 min	12.09	3.04
	1 h	14.90	5.27
	2 h	14.96	5.57
	4 h	15.40	6.49
PS2	5 min	12.12	3.53
	1 h	15.22	5.65
	2 h	16.14	5.87
	4 h	16.42	6.64
PS3	5 min	9.44	3.76
	1 h	36.10	6.11
	2 h	41.32	10.90
	4 h	45.39	11.09

In Table 4, adding LP reduces the yield stress (τ_0) and plastic viscosity (μ) of the composite paste, compared to those of the control group, indicating that the rheological properties of the composite paste are enhanced *via* increasing the LP content. This phenomenon can be attributed to the fact that the LP is capable of optimising the particle size distribution of the binder particles^[36], thereby facilitating the release of a greater quantity of free water and consequently

enhancing the fluidity of the paste. The τ_0 and μ of composite paste increase with prolonging rest time due to the hydration of the binder.

Compared to the control paste, the τ_0 and μ increase after adding SSP. In particular, the τ_0 of the composite paste with SSP content of 30% increases rapidly at 1 h and 2 h. These results indicate that adding SSP is not beneficial to the development of rheological properties of the paste. The rheological parameters increase markedly with prolonging rest time. SSP contains large particles (see Fig. 1(a)). Thus, its filling effect is not as good as that of LP, and the amount of free water released is lower. Furthermore, the hydration of SSP itself results in the consumption of free water, in turn leading to the weakening of the rheological properties of the paste.

LP can significantly reduce the thixotropic ring area and enhance the thixotropic properties of composite paste. The τ_0 and μ of the paste with LP are lower than those of the paste with an equal dosage of SSP. Furthermore, the change rule of the μ is consistent with the result of Marsh cone flow time (see Fig. 6), that is, the reduction of the μ improves the fluidity of the composite paste. Therefore, the rheological properties of the paste with LP are better than those of the paste with SSP.

3.5 TOC content

Fig. 11 shows the content of TOC adsorbed by binder particles in the composite paste at a W/B ratio of 0.16. Adding LP increases the content of TOC adsorbed by binder particles in the paste. A great deal of LP results in a greater adsorption amount. These results indicate that LP can improve the water reducing agent adsorption capacity of binder particles in the composite paste. The smaller particles and higher specific surface area of LP result in the enhanced adsorption capacity. Compared to the addition of LP, the incorporation of SSP markedly increases the TOC content adsorbed by the binder particles, thereby demonstrating that SSP can notably improve the adsorption capacity of water reducing agent of the binder particles in the paste. The adsorption state of polycarboxylate superplasticizer on SSP particles is toothed adsorption, which has a better adsorption

effect than LP. The molecular structure of polycarboxylate superplasticisers contains ether bonds, which result in the formation of a thicker hydrophilic stereo protective film. This film provides a dispersion stability for SSP particles, thereby enhancing the adsorption of polycarboxylate superplasticisers on SSP particles^[37].

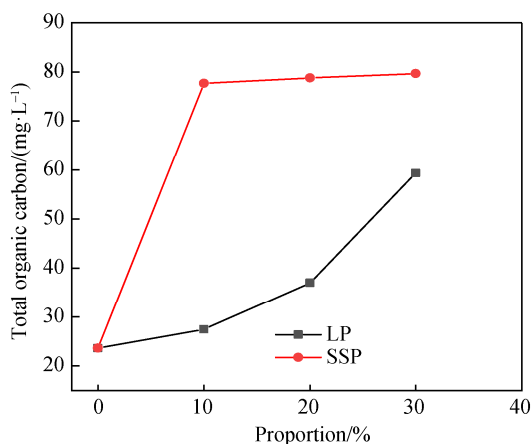


Fig. 11 Contents of TOC adsorbed by binder particles in composite paste at a W/B ratio of 0.16

Fig. 12 shows the free water content of composite paste at a W/B ratio of 0.16 after centrifugation. Adding LP increases free water content in the paste. The adsorption effect of the water reducing agent is also confirmed (see Fig. 11). A high content of LP can reduce the cement nucleation barrier and promote cement hydration, which in turn leads to a reduction in the free water content. The water-reducing effect of polycarboxylate superplasticizer is highly correlated to its dosage. The free water content is related to the amount and the water demand of LP or SSP. At the content of 10%, the free water content of the composite paste with SSP is higher than that of the composite paste with LP due to the smaller particle size of LP. The free water content of the composite paste with SSP is significantly lower than that of the composite paste with LP when the contents of SSP are 20% and 30%. This is due to the fact that the water demand of SSP is greater than that of LP. At the content of 30%, the dosage of free water in the composite paste with SSP changes little. However, the high content of LP leads to an increased water demand, thus free water content in the composite paste

with LP content of 30% is significantly reduced. Note that the adsorption capacity of polycarboxylate superplasticizer of the binder particles with SSP is greater than that of the binder particles with LP (see Fig. 11), but the free water content of the paste with SSP is lower than that of the paste with LP at a high replacement ratio (see Fig. 12). It is due to the rough and porous surface of SSP and the uneven particle shape^[38]. The polycarboxylate superplasticizer molecules are physically adsorbed on the SSP particles, reducing the content of effective dispersants. Although the content of polycarboxylate superplasticizer adsorbed by the composite paste containing SSP is high, the content of polycarboxylate superplasticizer adsorbed on the surface of cement particles is relatively low. Therefore, the flocculation structure formed by cement is not easily destroyed. Ultimately, the content of free water decreases significantly. On the contrary, the ability of LP to adsorb polycarboxylate superplasticizer is weak, the content of polycarboxylate superplasticizer adsorbing on cement is high, the flocculation structure formed by cement is easy to be destroyed, and the free water content significantly increases. Nevertheless, at the excessive content, LP also absorbs a greater quantity of polycarboxylate superplasticizer, which results in a reduction in the free water content.

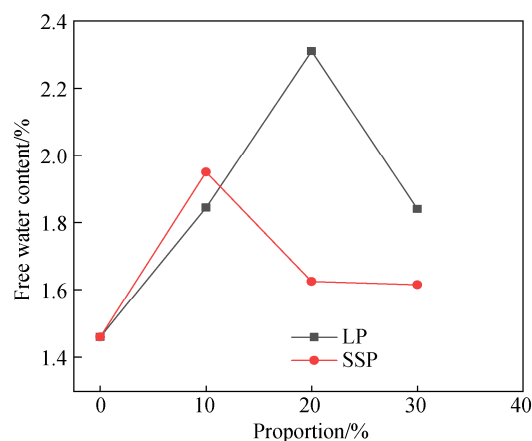


Fig. 12 Free water content of the composite paste at a W/B ratio of 0.16 after centrifugation

4 Conclusions

1) The initial flow diameter of composite paste can be increased *via* incorporating LP or SSP at a W/B

ratio of 0.16. The flow diameter of paste decreased with increasing addition amount and extension of the rest time. In general, the reduction in the loss rate of flow diameter caused *via* adding LP was lower than that caused *via* adding SSP.

2) The setting time of composite paste was shortened *via* adding an appropriate amount of LP or SSP at a W/B ratio of 0.16. At the SSP content of 30%, SSP had a retarding effect, prolonging the final setting time. The effect of LP on shortening the setting time could be greater than that of SSP.

3) The marsh cone flow time of the composite paste could extend with adding LP or SSP at a W/B ratio of 0.16. An increase in the mixing amount and extension of the rest time significantly prolonged the flow time of the composite paste. The increase in the flow time of the composite paste with the addition of LP was less pronounced than that with the addition of SSP.

4) The thixotropic properties of composite paste could be enhanced *via* adding an appropriate amount of LP at a W/B ratio of 0.16, whereas the thixotropic properties of paste could be weakened *via* adding SSP. The τ_0 and μ of paste decreased with increasing the content of LP, whereas the τ_0 and μ of the composite paste increase with increasing the content of SSP. The rheological properties of the composite paste with LP were better than those of the composite paste with SSP.

5) Adding LP or SSP could be advantageous for improving the adsorption capacity of binder particles for polycarboxylate superplasticizer. The adsorption effect was more pronounced at a high dosage. The adsorption effect of binder particles with SSP was more dominant than that of the binder particles with LP. The free water content of the composite paste with LP or SSP was greater than that of the control group.

6) The flow diameter and the setting time of the composite paste reduced and the marsh cone flow time prolonged when the W/B ratio increases to 0.21 due to the decrease in the content of polycarboxylate superplasticizer.

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Rheological Properties of Composite Pastes with Steel Slag Powder or Limestone Powder at Very Low Water/Binder Ratio

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Extended Abstract

Introduction The widespread use of cement in concrete leads to increased carbon emissions, so the demand for supplementary cementitious materials increases significantly. Limestone powder and steel slag powder are widely used as supplementary cementitious materials in modern concrete. However, for UHPC and self-compacting concrete, an extremely low water/binder (W/B) ratio is on one hand a key factor in achieving ultra-high strength and ultra-low porosity of the materials, on the other hand, also leads to the deterioration of the rheological properties of the fresh paste. Meanwhile, the existing researches focus on the influence of single limestone powder or steel slag powder on the mechanical properties or microstructure of cement-based materials. Little work on the influence of steel slag powder or limestone powder on the rheological properties of composite paste at an extremely low water/binder ratio has been investigated quantitatively. The mechanism of the effect of steel slag powder or limestone powder on the rheological properties of composite paste at extremely low water/binder ratios is still unclear. In this work, the effects of steel slag powder and limestone powder on the rheological properties of composite paste at different low water/binder ratios were analyzed via determining the flow diameter, setting time, marsh cone flow time, rheological parameters, and total organic carbon content.

Methods A composite paste was prepared with P.I 42.5 ordinary Portland cement, steel slag powder, limestone powder, blast furnace slag and silica fume as raw materials in a certain proportion. To achieve the preparation of composite paste with extremely

low water/binder ratios, a polycarboxylate superplasticizer with a water/reducing rate of 40% (Jiangsu Subot New Materials Co., Ltd., China) was used. The dosage of polycarboxylate superplasticizer for the composite paste with different water/binder ratios of 0.16 and 0.21 was 2% and 0.8%, respectively.

Composite pastes with different proportions of steel slag powder or limestone powder at water/binder ratios of 0.16 and 0.21 were prepared. The flow diameter (*i.e.*, the larger the flow diameter, the better the fluidity), setting time (*i.e.*, the time when the fluidity is lost) and marsh cone flow time (*i.e.*, the shorter the flow time, the better the fluidity) were determined to analyze the fluidity of the composite pastes. The rheological properties of composite paste at a water/binder ratio of 0.16 for rheological properties tests were determined, and the rheological parameters were obtained by the H-B model. The adsorption performance of the polycarboxylate superplasticizer was analyzed by testing the TOC content.

Results and discussion When the ratio of water/binder is 0.16, both limestone powder and steel slag powder initially increase the flow diameter of the composite paste. However, the fluidity of the composite paste decreases over time, and the reduction is more pronounced with steel slag powder. This is because the nucleation and hydration promotion effect of limestone powder can reduce the loss rate of flow diameter, while the rough particles of steel slag powder increase the internal friction force, resulting in a decrease in the flow diameter of composite paste. Adding limestone powder and steel slag powder both shortens the setting time and marsh cone flow time of the composite paste. However, the steel slag powder addition of 30 % delays the final setting time due to its delaying effect. Also, limestone powder can enhance the thixotropy and reduce the yield stress and plastic viscosity, thereby improving the rheological properties. In contrast, steel slag powder can increase the yield stress and plastic viscosity, thereby weakening the rheological properties and thixotropy. Steel slag powder and limestone powder both can enhance the adsorption effect of polycarboxylate superplasticizer. Steel slag powder has a stronger adsorption effect. The composite paste containing limestone powder has a higher free water content. This is because the rough and porous surface of steel slag itself and the uneven particle shape lead to the physical adsorption of polycarboxylate superplasticizer molecules on the surface of steel slag particles, thereby reducing the effective content of the water reducer dispersion. Increasing the water/binder ratio to 0.21 results in a decrease in the flow diameter of the composite paste. Furthermore, the setting time and marsh cone flow time can prolong due to the reduction in the dosage of polycarboxylate superplasticizer.

Conclusions The results showed that the fluidity loss rate of composite paste with limestone powder could be lower than that of composite paste with steel slag powder. Compared to steel slag powder, the addition of limestone powder reduced the setting time and marsh cone flow time of the composite paste. The addition of limestone powder could shorten the yield stress and plastic viscosity of the composite paste. Therefore, the composite paste with limestone powder had better rheological properties and stronger thixotropy rather than that with steel slag powder. The addition of limestone powder could improve the rheological properties of the composite paste. Compared to composite paste with limestone powder, a better adsorption effect of polycarboxylate superplasticizer on the surface of the composite binder with steel slag powder could be obtained. The free water content of the composite paste with limestone powder was still higher than that of the composite paste with steel slag powder. The fluidity and rheological properties of the composite paste with limestone powder could be better. The comprehensive analysis indicated that a positive effect of limestone powder on rheological properties of composite paste at an extremely low water/binder ratio could be more dominant than that of steel slag powder.

Keywords composite paste; limestone powder; steel slag powder; rheological properties; extremely low water/binder ratio