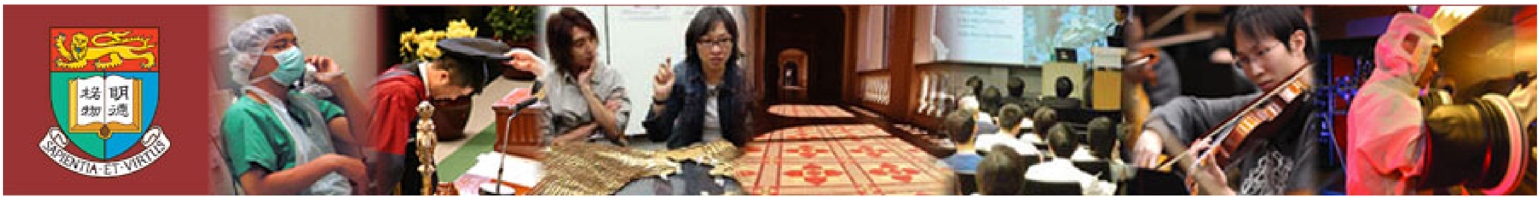

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Effects of superplasticiser on rheology and cohesiveness of CSF cement paste

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Superplasticiser (SP) is now an indispensable ingredient for the production of concrete. However, its proper use is a great concern because its over-dosage could lead to drastic reduction in the cohesiveness of concrete. Furthermore, experience has indicated that the SP demand, saturation dosage and dosage causing segregation are highly dependent on the powder content and therefore the effects of SP are not simple functions of dosage. This study aims to evaluate the effects of SP dosage on the fresh properties of cement paste containing condensed silica fume (CSF). A number of cement paste samples with different SP dosages and CSF contents were tested. The results showed that the addition of SP would improve the packing density, flowability and rheology but impair the cohesiveness of cement paste. It would also render the cement paste shear thickening. To cater for the effects of powder fineness, it is proposed that the SP dosage should be measured in terms of the quantity of SP per surface area of the powder content.

Introduction

The advent of superplasticiser (SP) is the key to the development of concrete technology (Aïtcin, 1998; Mindess *et al.*, 2003). With SP added, the cementitious materials would be dispersed to avoid the formation of agglomerates and thus increase the fluidity of the cement paste. At the same water/cementitious materials (W/CM) ratio and same paste volume, the addition of SP can improve the flowability of the concrete. Alternatively, for the same flowability requirement, the addition of SP can allow the W/CM ratio to be reduced to improve the strength and durability of the concrete, or the paste volume to be reduced to improve the dimensional stability of the concrete. In fact, with proper mix design, the use of a good SP can improve all the above performance attributes at the same time. Because of these advantages, SP has become so popular that it is now an indispensable ingredient for the production of concrete, especially high-performance concrete (HPC).

Over the decades, SP has evolved from the first generation lignosulfonate-based SP, to the second generation melamine-based or naphthalene-based SP, and then to the newest third generation polycarboxylate-based SP (ACI Committee 212, 1993; Malhotra, 2006). The earlier SPs disperse the cementitious materials mainly through the electrostatic repulsion between particles produced by imparting similar electrostatic charges to the particle surfaces, whereas the newest polycarboxylate-based SPs disperse the cementitious materials through not only the electrostatic repulsion but also the steric repulsion between particles produced by wrapping the particles with co-polymer side chains (Nawa, 2006; Neubauer *et al.*, 1998; Uchikawa *et al.*, 1997). Generally, the polycarboxylate-based SPs are more effective.

Although much effort has been devoted to studying the effects of SP on cement paste, mortar and concrete, there have been very few guidelines for the use of SP. In fact, proper use of SP is not straightforward. First, the SP demand for a given flowability requirement is dependent not only on the type of cement, but also on the types and fineness of other cementitious materials and fillers added to the cement paste (ACI Committee 234, 2006; Austin and Robins, 1994; Khatri *et al.*, 1995). In general, the higher is the fineness of the particles in the cement paste, the higher is the SP demand. Second, the effectiveness of a SP increases with the SP dosage only up to a certain saturation dosage, beyond which further addition of the SP gives minimal or even no return (Aydın *et al.*, 2009; Jayasree and Gettu, 2008; Price, 2003,). Hence, usage of a SP beyond the saturation dosage is not economical. Third, while the repulsion between particles due to addition of SP would improve the flowability of cement paste, mortar and concrete, it would also impair the cohesiveness, thus increasing the risk of bleeding, sedimentation and segregation (Kwan and Ng, 2009; Shannag, 2002). This problem of reduced cohesiveness is usually more serious when the effectiveness of the SP is high and/or the dosage of the SP is high (for instance, when a polycarboxylate-based SP is used at high dosage). To avoid such a problem, the SP dosage should be limited to not higher than the dosage causing segregation.

The SP demand, saturation dosage and dosage causing segregation are dependent not only on the SP used, but also on the types and fineness of the powder content (the cementitious materials and fillers) in the cement paste. As SP is a surface reactant adsorbed onto the particle surfaces of the powder content, its effectiveness should be dependent on the amount of SP per surface area of the powder content. Somehow, it has been a common practice to measure the SP dosage relative to the mass of the powder content (Khayat and Hwang, 2006; Uchikawa *et al.*, 1995). To cater for the difference in solid density, these authors have been measuring the SP dosage relative to the solid volume of the powder content (Kwan and Wong, 2008b; Wong and Kwan, 2008b). However, regardless of whether the SP dosage is measured by mass or by solid volume, experience has indicated that the SP demand, saturation dosage and dosage causing segregation could vary quite substantially as the powder content changes. It is thus doubtful whether it is really appropriate to specify the SP dosage by mass or by solid volume of the powder content.

With the above background, the present research project was launched to investigate the effects of a polycarboxylate-based SP at different dosage rates on the rheological properties of cement paste containing different amounts of condensed silica fume (CSF). The main aim was to evaluate how the increase in solid surface area due to the addition of CSF would affect the SP demand, saturation dosage and dosage causing segregation. A number of cement paste samples containing different dosages of SP and different amounts of CSF were produced for rheology, packing density and cohesiveness measurements. The rheology of each cement paste sample was measured in terms of flow spread and flow rate using the mini-slump cone and Marsh cone tests, and in terms of yield stress and apparent viscosity using a rheometer. On the other hand, the packing density and cohesiveness were measured using the wet packing test recently developed by the authors and a mini version of the sieve segregation test. The results so obtained were correlated to the SP dosage and CSF content for in-depth analysis of the combined effects of SP and CSF.

Rheological modelling

The rheology of a water–solid mixture is generally studied in terms of the shear stresses needed to flow at different shear rates. There are two rheological models describing the variation of the shear stress with the shear rate, namely, the Bingham model and the Herschel–Bulkley model (Nguyen *et al.*, 2006), as illustrated by the shear stress–shear rate curves plotted in Figure 1. In the Bingham model, the shear stress–shear rate relation is assumed to be linear as given by

1. $$\tau = \tau_y + \mu\,\gamma$$

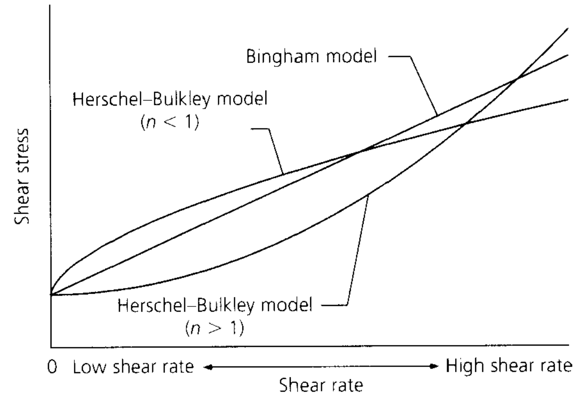

Figure 1. Bingham model and Herschel–Bulkley model

where τ (Pa) is shear stress, γ (s^{-1}) is shear rate, τ_y (Pa) is yield stress and μ (Pa s) is an empirical coefficient. On the other hand, in the Herschel–Bulkley model, the shear stress–shear rate relation is assumed to be non-linear as given by

2. $$\tau = \tau_y + K\gamma^n$$

where K (Pa s^n) and n (dimensionless) are empirical coefficients. From the shear stress–shear rate curve, the yield stress may be obtained as the shear stress at zero shear rate and the viscosity at a certain shear rate may be obtained as the shear stress to shear rate ratio at that shear rate. When $n = 1$, the Herschel–Bulkley model is the same as the Bingham model. However, for cement grout and paste, n is generally not equal to 1 and therefore the Herschel–Bulkley model is more appropriate.

When $n > 1{\cdot}0$, shear thickening (increase of viscosity with increasing shear rate) occurs. On the other hand, when $n < 1{\cdot}0$, shear thinning (decrease of viscosity with increasing shear rate) occurs. Shear thickening or thinning has great influence on the rheological behaviour of cement grout or paste. A shear thickening grout or paste has a lower viscosity when it flows slowly but a higher viscosity when it flows quickly. In contrast, a shear thinning grout or paste has a higher viscosity when it flows slowly but a lower viscosity when it flows quickly. A certain minimum viscosity at low shear rate is needed to avoid sedimentation and segregation, but a high viscosity at high shear rate would lead to difficulties in pumping (Feys *et al.*, 2009; Leemann and Winnefeld, 2007).

Whether a cement paste is shear thickening or thinning depends on several parameters, including the particle size distribution, particle shape, solid concentration of particles, interaction between particles and admixtures added (Curcio and De Angelis, 1998; Cyr *et al.*, 2000; Lachemi *et al.*, 2004; Rosquoet *et al.*, 2002). The usual practice of measuring the flowability of cement paste using the slump flow test gives only the rheological properties at low shear rate. To study the rheological properties at high shear rate, measurement of flow rate using the Marsh cone test is needed. However, these tests measure the rheological properties at either low or high shear rate. A better way of studying the full range rheological properties and shear thickening/thinning behaviour of cement paste is to use a rheometer that can be operated at a controlled and variable shear rate (this test is called the rheometer test). In the present study, the rheological properties and shear thickening/thinning behaviour of the cement paste samples were measured using all the above tests.

Experimental programme

In total, 30 cement paste samples produced by adding different SP dosages and CSF contents to ordinary Portland cement (OPC) were tested. Since it is the solid volume rather than the mass that is more relevant, the SP dosage, CSF content and the W/CM ratio were all designed on a volumetric basis. The SP dosage was expressed in terms of the liquid volume of SP per solid volume of the cementitious materials. As a norm, the standard SP dosage (denoted by $1 \times$ SP) was set to be $3{\cdot}112 \times 10^{-2}$ m^3 per m^3 of the cementitious materials (equivalent to 1% by mass of the cement when only cement is added as cementitious material). In order to study the combined effects of SP and CSF, the SP dosage was varied from $0 \times$ SP to $5 \times$ SP (from 0 to 5 times the standard SP dosage), while the CSF content was varied among 0, 5 and 10% of the total cementitious materials. On the other hand, the W/CM ratios of all the cement paste mixes were fixed at 1·0 by volume. Table 1 presents the detailed mix proportions of the cement paste samples. Each cement paste sample was assigned an identification code of C-X-Y, in which C means cement paste, X denotes the SP dosage expressed as a multiple of standard SP dosage and Y denotes the CSF content expressed as a percentage of the total cementitious materials.

Each cement paste sample was of 1000 ml size. It was produced by mixing the designed mix proportions of OPC, CSF, water and SP together, as per Table 1, using a standard mixer complying with BS EN 196: Parts 1–3 (BSI, 1995a). As the conventional method of mixing all the cementitious materials and water in one go would encounter difficulties when the CSF content is high and/or the water content is low, owing to the apparent dryness of the cement paste formed, a special mixing procedure of adding the cementitious materials in several small increments to the water was adopted. This mixing procedure has been applied in previous studies (Kwan and Wong, 2008a, 2008b; Wong and Kwan, 2008a, 2008b) and proven to be effective in ensuring thorough mixing. To minimise the influences of temperature, all sample preparation and testing procedures were carried out in a laboratory maintained at a temperature of 24 ± 2°C.

A comprehensive testing programme, in which each cement paste sample so produced was tested for its flow spread, flow rate, yield stress, apparent viscosity, shear thickening/thinning behaviour and sieve segregation index, was launched to evaluate the combined effects of SP dosage and CSF content on the rheology and cohesiveness of CSF cement paste. In addition, each designed mix of cementitious materials was tested for its packing densities at different SP dosages so as to study how the SP dosage and CSF content affect the packing density and investigate the role of packing density in the fresh properties of cement paste.

Mix no.	Volume of component materials: ml			
	OPC	CSF	Water	SP
C-0·00-0	500·0	0·0	500·0	0·0
C-0·25-0	500·0	0·0	496·1	3·9
C-0·50-0	500·0	0·0	492·2	7·8
C-0·75-0	500·0	0·0	488·3	11·7
C-1·00-0	500·0	0·0	484·4	15·6
C-1·50-0	500·0	0·0	476·7	23·3
C-2·00-0	500·0	0·0	468·9	31·1
C-3·00-0	500·0	0·0	453·3	46·7
C-4·00-0	500·0	0·0	437·8	62·2
C-5·00-0	500·0	0·0	422·2	77·8
C-0·00-5	475·0	25·0	500·0	0·0
C-0·25-5	475·0	25·0	496·1	3·9
C-0·50-5	475·0	25·0	492·2	7·8
C-0·75-5	475·0	25·0	488·3	11·7
C-1·00-5	475·0	25·0	484·4	15·6
C-1·50-5	475·0	25·0	476·7	23·3
C-2·00-5	475·0	25·0	468·9	31·1
C-3·00-5	475·0	25·0	453·3	46·7
C-4·00-5	475·0	25·0	437·8	62·2
C-5·00-5	475·0	25·0	422·2	77·8
C-0·00-10	450·0	50·0	500·0	0·0
C-0·25-10	450·0	50·0	496·1	3·9
C-0·50-10	450·0	50·0	492·2	7·8
C-0·75-10	450·0	50·0	488·3	11·7
C-1·00-10	450·0	50·0	484·4	15·6
C-1·50-10	450·0	50·0	476·7	23·3
C-2·00-10	450·0	50·0	468·9	31·1
C-3·00-10	450·0	50·0	453·3	46·7
C-4·00-10	450·0	50·0	437·8	62·2
C-5·00-10	450·0	50·0	422·2	77·8

Table 1 Mix proportions of the cement paste samples

Materials

The SP used for the study was a third generation polycarboxylate-based SP. Its molecule looks like a comb, with a main chain formed of an active monomer, which is basically a polycarboxylate synthesised by polymerisation, and a number of side chains formed of graft copolymers, which are synthesised from polyethylene oxide. It disperses the cementitious materials by both electrostatic repulsion and steric repulsion, and is thus more effective than earlier generation SPs. The solid mass content and relative density of this SP have been measured to be 20% and 1·03, respectively. According to the supplier, the normal dosage of this SP, measured in terms of liquid mass, should be 0·5–3·0% by mass of the cement, but a higher dosage may also be used if proven to be satisfactory by trial mixing.

Both the OPC and CSF were commonly used materials readily available from the market. The OPC was a local product of strength class 52·5N complying with BS 12: 1996 (BSI, 1996), whereas the CSF was a Norwegian product complying with ASTM C 1240-03 (ASTM, 2003). The solid densities of the OPC and CSF had been measured in accordance with BS 812: Part 2: 1995 (BSI, 1995b) as 3112 kg/m^3 and 2196 kg/m^3, respectively. Their particle size distributions were measured by a laser diffraction particle size analyser and the results obtained are plotted in Figure 2. The specific surface areas of the OPC and CSF were calculated based on their particle size distributions as $1{\cdot}40 \times 10^6$ and $13{\cdot}3 \times 10^6$ m^2/m^3, respectively. The chemical compositions of the OPC and CSF, as provided by the suppliers, are presented in Table 2.

Test methods

Measurement of packing density

The packing density was measured using the wet packing test developed by the authors' research group (Kwan and Wong, 2008a; Wong and Kwan, 2008a). Basically, this method determines the packing density of the cementitious materials as the maximum solid concentration achieved by the cementitious materials when they are mixed with water at different W/CM ratios. To measure the packing density, six to eight samples of cement paste were formed of the cementitious materials, starting at a relatively high W/CM ratio and successively reducing the W/CM ratio until the solid concentration had reached a maximum value and then decreased. The detailed procedures are summarised below.

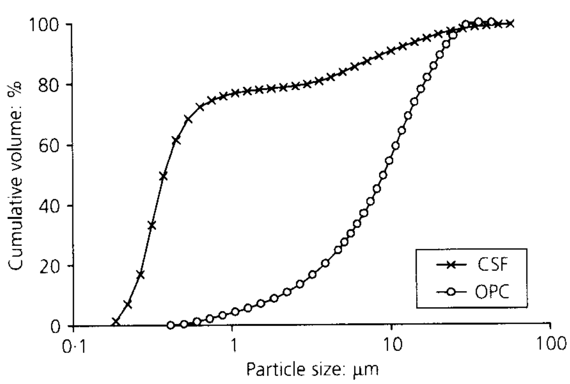

Figure 2. Particle size distributions of OPC and CSF

Chemical	OPC: %	CSF: %
Calcium oxide (CaO)	65·7	0·44
Silicon dioxide (SiO_2)	21·8	95·8
Aluminium oxide (Al_2O_3)	5·7	—
Iron oxide (Fe_2O_3)	3·6	—
Magnesium oxide (MgO)	2·2	—
Sulfuric anhydride (SO_3)	2·3	0·21
Sodium oxide equivalent ($(Na_2O)_{eq}$)	0·44	0·73
Loss on ignition	0·88	1·17

Table 2. Chemical compositions of OPC and CSF

(*a*) Set the W/CM ratio at which the test is to be carried out. Weigh the required quantities of water, cementitious materials and superplasticiser and dose each ingredient into a separate container.
(*b*) If the cementitious materials consist of several different materials blended together, pre-mix the materials in dry conditions for 2 min.
(*c*) Add all the water and half of the cementitious materials and superplasticiser into the mixing bowl and run the mixer for 3 min.
(*d*) Add the remaining cementitious materials and superplasticiser in four equal portions into the mixing bowl and each time run the mixer for 3 min.
(*e*) Transfer the mixture to a cylindrical mould, fill the mould to excess and remove the excess with a straight edge. Weigh the amount of paste in the mould to determine the wet bulk density of the paste.
(*f*) Repeat steps (*a*) to (*e*) at successively lower W/CM ratios until the maximum solid concentration has been obtained.

From the test results obtained, the solid concentration of the cementitious materials in the paste can be determined as follows. Let the mass and volume of the paste in the mould be M and V, respectively. The wet bulk density of the paste is equal to M/V. If the cementitious materials consist of several different materials denoted by α, β, γ and so forth, the solid concentration ϕ can be worked out as

$$\phi = \frac{M/V}{\rho_w u_w + \rho_\alpha R_\alpha + \rho_\beta R_\beta + \rho_\gamma R_\gamma} \quad (3)$$

where ρ_w is the density of water, ρ_α, ρ_β and ρ_γ are the solid densities of α, β and γ, u_w is the W/CM ratio by volume and R_α, R_β and R_γ are the volumetric ratios of α, β and γ to the total cementitious materials. The maximum value of ϕ so obtained is taken as the packing density of the cementitious materials.

Measurement of flowability

The flowability was measured in terms of flow spread using the mini slump cone test and in terms of flow rate using the Marsh cone test, as suggested by Aïtcin (1998). However, there are many different versions of mini slump cone and Marsh cone. In this study, the mini slump cone adopted was the same as that used by Okamura and Ouchi (2003) while the Marsh cone adopted was the same as that specified in BS EN 445: 1997 (BSI, 1997), as in previous studies (Kwan and Wong, 2008b; Wong and Kwan, 2008b).

The procedures of the mini slump cone test are as follows.

(*a*) Place the slump cone at the centre of a levelled steel plate.
(*b*) Pour the cement paste slowly into the slump cone until the slump cone is completely filled up.
(*c*) Lift the slump cone gently and allow the cement paste to spread until stoppage.
(*d*) Measure the diameters of the cement paste patty in two orthogonal directions, calculate the average diameter and determine the flow spread of the cement paste as the average diameter minus the base diameter of the slump cone.

The procedures of the Marsh cone test are as follows.

(*a*) Attach the Marsh cone to a stand and place a graduated cylinder underneath the orifice of the Marsh cone.
(*b*) Set a video recorder to take a video of the whole testing process for measuring the time of flow.
(*c*) Close the orifice and pour 900 ml of cement paste into the Marsh cone. To minimise air entrapment, pour the cement paste slowly along the inner surface of the Marsh cone.
(*d*) Start the video recorder and open the orifice to let the cement paste flow out.
(*e*) Measure the time needed for 300 ml of cement paste to flow out from the video record. Determine the flow rate as 300 ml divided by the measured time. For a dry cement paste which takes a long time to flow out, stop the test at 300 s after opening the orifice and determine the flow rate as the volume of cement paste collected underneath the Marsh cone divided by 300 s.

Measurement of rheological properties

The rheometer and test method used were the same as those used in previous studies (Wong and Kwan, 2008b). As the details have been presented before, only the basic features are given herein. The shearing sequence applied to each cement paste sample consisted of two shearing cycles. The first shearing cycle (pre-shearing cycle) was to ensure that each cement paste sample had undergone the same shearing history before measurement. The second shearing cycle (data-logging cycle) was the cycle in which actual measurement was carried out by a data logger. During each shearing cycle, the shear rate was increased from 0 to 50 s^{-1} in 75 s and then decreased to 0 s^{-1} in another 75 s. Two shear stress–shear rate curves were obtained, one at increasing shear rate and the other at decreasing shear rate. The shear stress–shear rate curve at decreasing shear rate, which is generally more consistent and repeatable, was used for evaluating the rheological properties of the cement paste sample.

The shear stress–shear strain curve may be described in terms of either the Bingham model or the Herschel–Bulkley model. Herein, the Herschel–Bulkley model, which generally agrees better with the experimental results, was adopted. Its shear stress–shear rate equation is given by Equation 2. For each cement paste sample, the best-fit curve based on this equation was obtained by regression analysis. From the best-fit curve so obtained, the shear stress at a shear rate of 0 s^{-1} and the ratio of shear stress to shear rate at a shear rate of 50 s^{-1} were taken as the yield stress and the apparent viscosity, respectively, of the cement paste sample tested. Furthermore, the n-value of the best-fit curve was taken as a measure of the degree of shear thickening of the cement paste sample ($n > 1{\cdot}0$ implies shear thickening whereas $n < 1{\cdot}0$ implies shear thinning).

Measurement of cohesiveness

Until now there has been no established test method for measuring the cohesiveness of cement paste. In this study, the possibility of using a mini version of the sieve segregation test to measure the cohesiveness of cement paste in terms of a sieve segregation index (SSI) was explored. Two sieves, one of 0·15 mm aperture size and the other of 0·3 mm aperture size, were employed for the sieve segregation tests. These two sieves are just those normally used for sieve analysis of fine aggregate. Apart from the aperture size, they are similar to each other and both have an overall diameter of 200 mm.

The procedures of the sieve segregation test are as follows.

(*a*) Put the base receiver underneath the 0·15 mm or 0·3 mm sieve.
(*b*) Pour gently about 150 ml of cement paste onto the sieve from a height of 300 mm.
(*c*) Let the cement paste in the sieve drip through the apertures of the sieve into the base receiver and wait for at least 2 min until the dripping stops.
(*d*) Measure the weight of cement paste poured onto the sieve as the increase in weight of the sieve and base receiver assembly, and record this weight as W_p.
(*e*) Measure the weight of cement paste collected by the base receiver as the increase in weight of the base receiver, and record this weight as W_b.
(*f*) Determine the SSI as $(W_b/W_p) \times 100\%$.

The SSI so determined above is used as an indication of the cohesiveness of the cement paste. A low SSI represents a high cohesiveness whereas a high SSI represents a low cohesiveness.

Experimental results

Quantification of SP dosage

Up to now, the SP dosage recommended by the supplier is generally expressed in terms of the quantity of SP per mass of cement. However, this way of quantifying the SP dosage is not really appropriate when supplementary cementitious materials are present because in such cases the cement is only a part of the cementitious materials. Hence, the SP dosage should be quantified relative to the cementitious materials content rather than the cement content.

Furthermore, since the various supplementary cementitious materials do not have the same density as the cement and it is the solid volume rather than the mass that is more relevant, quantifying the SP dosage relative to the solid volume of the cementitious materials should be more appropriate. In theory, however, since SP is a surfactant and its effects take place on the particle surfaces, its effectiveness should be dependent on the quantity of SP per surface area of the cementitious materials rather than the quantity of SP per solid volume of the cementitious materials. In the present study, in order to find out which is a better way of quantifying the SP dosage, the effects of SP were correlated first to the SP dosage expressed in terms of the quantity of SP per solid volume of cementitious materials and then to the SP dosage expressed in terms of the quantity of SP per surface area of cementitious materials.

Effects of SP on packing density

The packing density results of the cement paste samples with different SP dosages and CSF contents are tabulated in the second column of Table 3. From these results, it can be seen that with no CSF added, the packing density increased with the SP dosage from 0·578 at 0 × SP to 0·654 and 0·668 at 3 × SP and 5 × SP, respectively. With 5% CSF added, the packing density increased from 0·601 at 0 × SP to 0·681 and 0·683 at 3 × SP and 5 × SP, respectively. With 10% CSF added, the packing density increased from 0·604 at 0 × SP to 0·707 and 0·696 at 3 × SP and 5 × SP, respectively. Hence, the dispersion effect of the SP had significantly improved the packing density of the cementitious materials. It was only when the SP dosage was increased to beyond 3 × SP that there was little further improvement in packing density.

It can also be seen that at 0 × SP, the packing density increased with the CSF content from 0·578 at 0% CSF to 0·601 and 0·604 at 5% CSF and 10% CSF, respectively. At 3 × SP, the packing density increased from 0·654 at 0% CSF to 0·681 and 0·707 at 5% CSF and 10% CSF, respectively. Hence, the filling effect of

Mix no.	Packing density	Flow spread: mm	Flow rate: ml/s	Yield stress: Pa	Apparent viscosity: Pa s	*n*-value
C-0·00-0	0·578	0·0	0·00	17·06	1·22	0·80
C-0·25-0	—	130·8	7·22	5·21	0·88	0·86
C-0·50-0	0·622	200·8	11·65	1·15	0·58	1·16
C-0·75-0	—	283·8	13·76	0·44	0·66	1·34
C-1·00-0	0·634	339·3	14·95	0·45	0·47	1·41
C-1·50-0	—	366·8	14·68	0·48	0·61	1·51
C-2·00-0	0·651	371·0	14·82	0·07	0·42	1·51
C-3·00-0	0·654	395·0	13·84	0·05	0·43	1·61
C-4·00-0	0·667	397·8	15·82	0·01	0·46	1·66
C-5·00-0	0·668	413·5	13·97	0·00	0·50	1·77
C-0·00-5	0·601	0·0	0·00	50·85	1·81	0·69
C-0·25-5	—	65·0	2·57	30·25	1·42	0·83
C-0·50-5	0·645	147·5	7·04	5·34	1·16	0·95
C-0·75-5	—	199·0	9·82	3·41	1·23	1·04
C-1·00-5	0·662	271·3	10·55	1·86	0·92	1·38
C-1·50-5	—	342·3	11·66	1·44	0·90	1·44
C-2·00-5	0·687	400·0	11·16	1·43	0·68	1·57
C-3·00-5	0·681	407·5	13·09	0·60	0·61	1·63
C-4·00-5	0·689	421·3	10·37	0·44	0·60	1·68
C-5·00-5	0·683	444·5	9·80	0·04	0·73	1·61
C-0·00-10	0·604	0·0	0·00	70·02	1·97	0·97
C-0·25-10	—	0·0	0·00	48·29	1·81	0·97
C-0·50-10	0·648	151·0	6·87	26·27	1·68	0·99
C-0·75-10	—	171·5	8·57	10·44	1·28	1·11
C-1·00-10	0·689	257·8	10·01	8·92	1·07	1·32
C-1·50-10	—	299·3	11·18	6·52	0·94	1·50
C-2·00-10	0·696	394·0	10·32	0·14	0·68	1·63
C-3·00-10	0·707	402·5	10·96	0·17	0·69	1·63
C-4·00-10	0·703	399·0	11·38	0·04	0·66	1·68
C-5·00-10	0·696	406·5	11·11	0·21	0·74	1·64

Table 3. Packing density and rheological properties results

the CSF can significantly improve the packing density of the cementitious materials. With 10% CSF added, the increase in packing density at 0 × SP was about 4·5% whereas the increase in packing density at 3 × SP was about 8·1%. These results reveal that the improvement in packing density was greater at a higher SP dosage or, in other words, the dispersion effect of the SP had enhanced the filling effect of the CSF.

Effects of SP on flow spread

The flow spread results are tabulated in the third column of Table 3. To present graphically how the flow spread varied with the SP dosage, the flow spread is plotted against the SP dosage in terms of the quantity of SP per solid volume of cementitious materials in Figure 3(a) and against the SP dosage in terms of the quantity of SP per surface area of cementitious materials in Figure 3(b). It can be seen that as SP was added, initially when the SP dosage was relatively small, the flow spread increased almost linearly with the SP dosage. Then, when the SP dosage had become relatively high, the flow spread increased with the SP dosage at a decreasing rate until eventually, the flow spread increased only marginally with the SP dosage. Hence, for each cement paste, there existed a saturation SP dosage, beyond which further addition of SP gave little further increase in flow spread.

From Figure 3(a), it is evident that the saturation SP dosage relative to the solid volume of cementitious materials was dependent on the CSF content, being higher at a higher CSF content. However, from Figure 3(b), it can be seen that the saturation SP dosage relative to the surface area of cementitious materials was not dependent on the CSF content. Furthermore, the various flow spread–SP dosage per surface area curves for different CSF contents are very close to each other at SP dosage lower than the saturation SP dosage, indicating that the effectiveness of the SP should be dependent more on the SP dosage per surface area than on the SP dosage per solid volume. Based on the flow spread results, the saturation dosage per surface area is determined as $0{\cdot}025 \times 10^{-6}$ m³/m².

Effects of SP on flow rate

The flow rate results are tabulated in the fourth column of Table 3 and plotted against the SP dosage per solid volume in Figure 4(a) and against the SP dosage per surface area in Figure 4(b). It

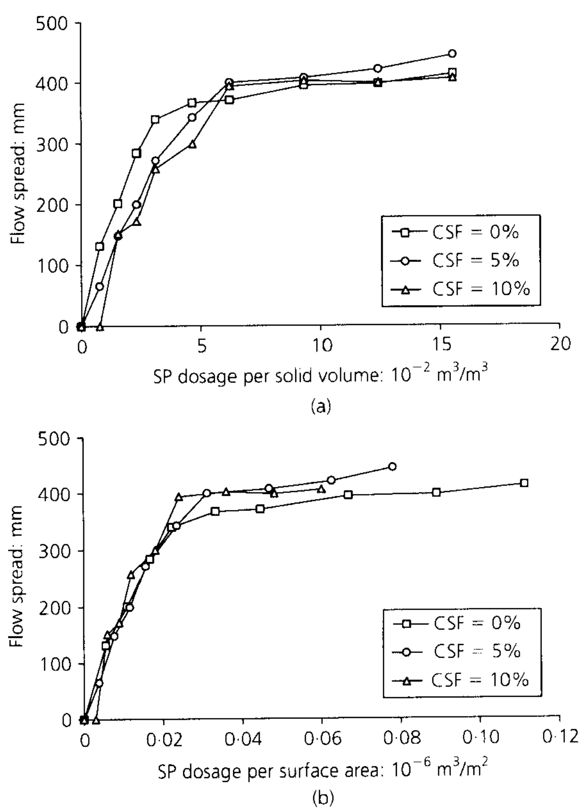

Figure 3. Flow spread plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

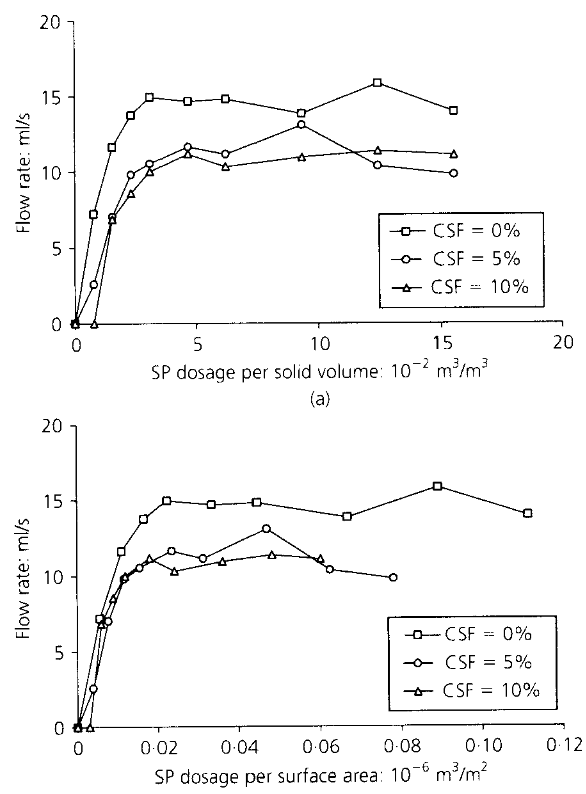

Figure 4. Flow rate plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

is seen that the flow rate increased with the SP dosage until a certain saturation dosage was reached and then increased only marginally with the SP dosage. As in the case of flow spread, the flow rate curves reveal that the saturation SP dosage relative to the solid volume of cementitious materials was quite sensitive to the CSF content, whereas the saturation SP dosage relative to the surface area of cementitious materials was not sensitive to the CSF content. Based on the flow rate results, the saturation dosage per surface area is determined as $0{\cdot}025 \times 10^{-6}$ m^3/m^2.

The flow rate curves plotted in Figure 4 also reveal that when the SP dosage was lower than the saturation dosage, the flow rate was dependent mainly on the SP dosage per surface area (note that at low SP dosage, the various flow rate–SP dosage per surface area curves merge into one single curve). However, when the SP dosage was higher than the saturation dosage, the flow rate was dependent mainly on the CSF content, being higher at lower CSF content and lower at higher CSF content. This was because at higher CSF content, the surface area of the cementitious materials was larger and thus for the same W/CM ratio, the water film thickness (the thickness of water films coating the solid particles) was smaller (Kwan *et al.*, 2010).

Effects of SP on yield stress

The yield stress results are tabulated in the fifth column of Table 3 and plotted against the SP dosage per solid volume in Figure 5(a) and against the SP dosage per surface area in Figure 5(b). It is seen that regardless of the CSF content, the yield stress decreased dramatically to a very small value as the SP dosage increased to a certain saturation dosage and then decreased to nearly zero as the SP dosage further increased. Comparing the yield stress curves in Figures 5(a) and 5(b), it is evident once again that the saturation SP dosage was related more to the surface area than to the solid volume of the cementitious materials. Based on the yield stress results, the saturation dosage per surface area is determined as $0{\cdot}025 \times 10^{-6}$ m^3/m^2. It is also evident that when the SP dosage was lower than the saturation dosage, the yield stress was dependent not only on the SP dosage, but also on the CSF content (note that the various yield stress curves do not merge into one curve).

Effects of SP on apparent viscosity

The apparent viscosity results are tabulated in the sixth column of Table 3 and plotted against the SP dosage per solid volume in Figure 6(a) and against the SP dosage per surface area in Figure 6(b). It is seen that regardless of the CSF content, the apparent viscosity decreased by more than half as the SP dosage increased to a certain saturation dosage and then remained more or less constant as the SP dosage further increased. As in the case of yield stress, the saturation SP dosage was related more to the surface area than to the solid volume of cementitious materials. Based on the apparent viscosity results, the saturation SP dosage per surface area is determined as $0{\cdot}025 \times 10^{-6}$ m^3/m^2. However, regardless of the SP dosage, the apparent viscosity was dependent not only on the SP dosage, but also on the CSF content (note that the various apparent viscosity curves do not merge into one curve).

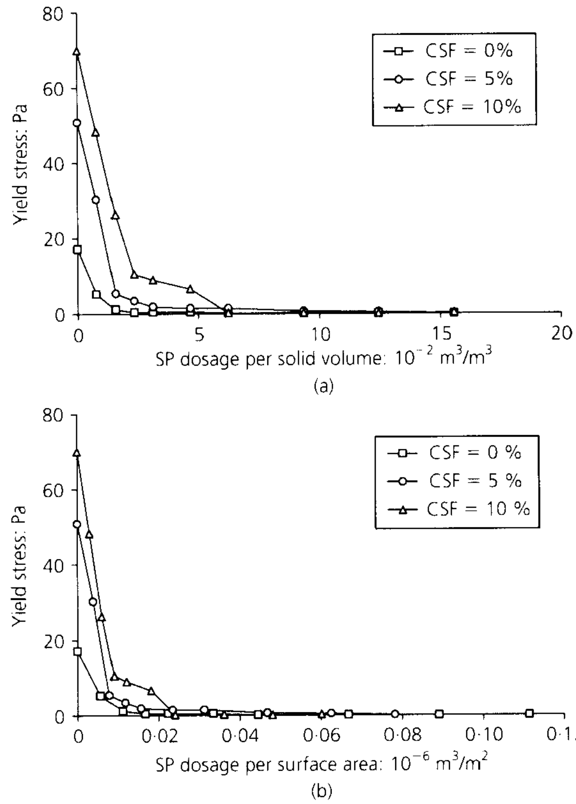

Figure 5. Yield stress plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

Effects of SP on shear thickening/thinning

The shear stress–shear rate curves for the cement paste samples with SP dosages of $0 \times$ SP, $0{\cdot}5 \times$ SP and $4 \times$ SP and CSF contents of 0% and 5% are plotted in Figure 7. It can be seen from the figure that the position and gradient of the shear stress–shear rate curve are both dependent on the SP dosage. The position of the curve moved downwards as the SP dosage increased because of the decreases in yield stress and apparent viscosity with increasing SP dosage. However, the effect of the SP dosage on the gradient of the curve was fairly complicated. At a SP dosage of $0 \times$ SP, the gradient of the curve decreased as the shear rate increased, indicating that the cement paste was shear thinning. At a SP dosage of $0{\cdot}5 \times$ SP, the gradient of the curve remained almost constant as the shear rate increased, indicating that the cement paste was neither shear thickening nor shear thinning. At a SP dosage of $4 \times$ SP, the gradient of the curve increased as the shear rate increased, indicating that the cement paste was shear thickening.

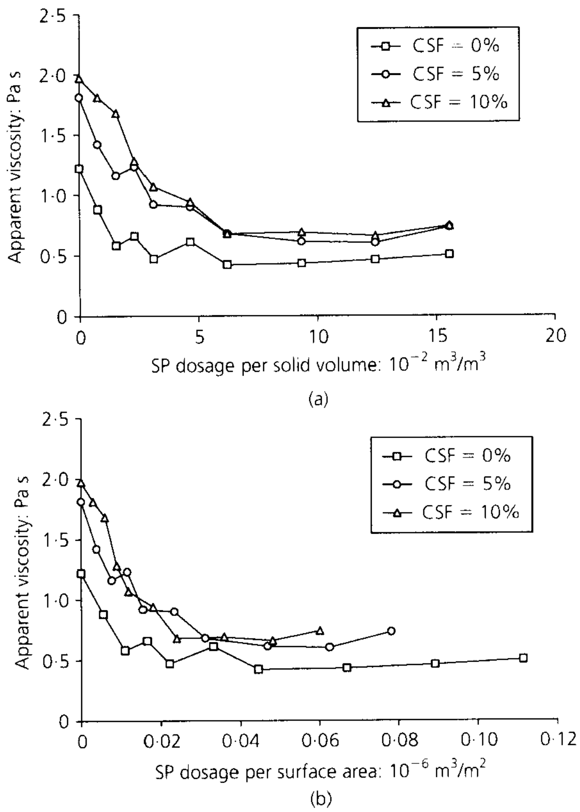

Figure 6. Apparent viscosity plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

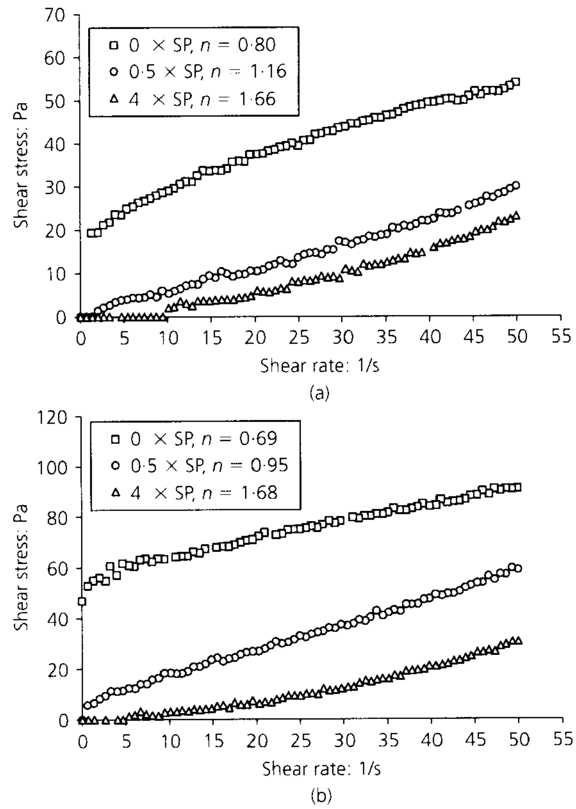

Figure 7. Shear stress–shear rate relations at different SP dosages and CSF contents: (a) CSF = 0%; (b) CSF = 5%

Based on the Herschel–Bulkley model, the shear thickening/thinning behaviour may be studied in terms of the n-value of the shear stress–shear rate curve obtained by regression analysis. The n-values of all the cement paste samples are tabulated in the last column of Table 3 and plotted against the SP dosage per solid volume in Figure 8(a) and against the SP dosage per surface area in Figure 8(b). The data points plotted clearly show that, regardless of the CSF content, the n-value increased with the SP dosage from lower than 1·0 at a relatively low SP dosage to higher than 1·0 at a relatively high SP dosage. Since $n < 1 \cdot 0$ indicates shear thinning and $n > 1 \cdot 0$ indicates shear thickening, it may be said that the addition of SP up to a certain dosage would render the cement paste shear thickening.

It should, however, be noted that the addition of SP would not thicken the cement paste. In fact, from Figure 7, it can be seen that the addition of SP had substantially reduced the yield stress and apparent viscosity of the cement paste. It was only that the SP was more effective in reducing the shear stress at low shear rate than in reducing the shear stress at high shear rate. The cement paste appeared to be shear thickening solely because of the proportionally larger reduction in the shear stress at low shear rate than the reduction in the shear stress at high shear rate.

The above phenomenon may be explained as follows. There are two major factors affecting the shear thickening/thinning behaviour of cement paste: thixotropy and dilatancy. When the SP dosage is low, the thixotropy would render the yield stress relatively high and the continuous breakdown of the particle structure during shearing would lead to shear thinning (Salem, 2002). On the other hand, when the SP dosage is high, the thixotropy would be reduced by the dispersive effect of the SP and the dilatancy caused by attrition among the closely packed particles in the cement paste would lead to shear thickening (Curcio and De Angelis, 1998).

Effects of SP on cohesiveness

The 0·15 mm SSI (SSI measured by a 0·15 mm sieve) and 0·3 mm SSI (SSI measured by a 0·3 mm sieve) results are respectively presented in the second and third columns of Table 4 and plotted against the SP dosage in Figures 9 and 10. These results reveal that as the SP dosage increased from zero, the SSI values first remained at absolute zero and then, after the SP dosage had exceeded a certain value, increased significantly with

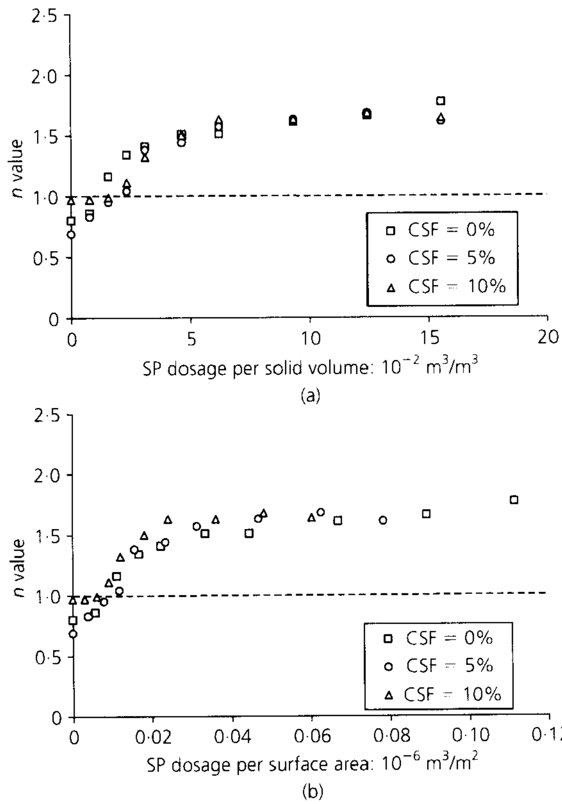

Figure 8. n-value plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

Mix no.	SSI: %		Bleeding width: mm
	0·15 mm	0·3 mm	
C-0·00-0	0·0	0·0	0·0
C-0·25-0	0·0	0·0	0·0
C-0·50-0	1·4	1·8	0·0
C-0·75-0	5·7	16·2	0·0
C-1·00-0	9·9	39·6	7·1
C-1·50-0	12·7	47·1	9·6
C-2·00-0	10·4	40·0	16·5
C-3·00-0	11·1	42·2	30·8
C-4·00-0	10·2	47·8	26·6
C-5·00-0	13·0	40·0	15·3
C-0·00-5	0·0	0·0	0·0
C-0·25-5	0·0	0·0	0·0
C-0·50-5	0·0	0·0	0·0
C-0·75-5	0·0	0·0	0·0
C-1·00-5	2·9	4·2	0·0
C-1·50-5	5·1	20·1	0·0
C-2·00-5	6·0	26·1	2·8
C-3·00-5	6·1	23·0	11·3
C-4·00-5	6·7	27·3	3·9
C-5·00-5	7·5	28·1	16·3
C-0·00-10	0·0	0·0	0·0
C-0·25-10	0·0	0·0	0·0
C-0·50-10	0·0	0·0	0·0
C-0·75-10	0·0	0·0	0·0
C-1·00-10	0·0	0·0	0·0
C-1·50-10	1·3	5·4	0·0
C-2·00-10	4·7	14·3	4·5
C-3·00-10	4·1	14·5	5·0
C-4·00-10	4·1	13·0	4·8
C-5·00-10	5·4	15·5	6·5

Table 4. Sieve segregation index and bleeding width results

the SP dosage. This may be attributed to the gradual reduction in cohesiveness of the cement paste due to the dispersion effect of the SP added.

Comparing the SSI–SP dosage per solid volume curves plotted in Figures 9(a) and 10(a) to the SSI–SP dosage per surface area curves plotted in Figures 9(b) and 10(b), it can be seen that the SP dosage at which the SSI started to increase was related more to the surface area than to the solid volume of cementitious materials. Moreover, the various SSI–SP dosage per surface area curves are quite close to each other before they diverge at high SP dosage. Overall, at a relatively low SP dosage, the SSI was dependent mainly on the SP dosage per surface area while at a relatively high SP dosage, the SSI was dependent mainly on the CSF content.

There is still no generally adopted acceptance criterion for the SSI. Actually, obvious signs of segregation were frequently observed when the SSI of the cement paste sample was relatively high. One easy way of detecting segregation was found to be the observation of the presence of bleeding water along the edge of the cement paste patty after the mini slump cone test. After the mini slump cone test, a ring of bleeding water was sometimes found along the edge of the cement paste patty. The width of such ring of bleeding water, if found, was measured at four equidistant locations, which were then averaged to yield the bleeding width of the cement paste sample. For correlation with the SSI values, the bleeding width results so obtained are tabulated in the last column of Table 4. The correlation reveals that all the cement paste samples showing obvious signs of segregation (bleeding width > 0) have 0·15 mm SSI $\geqslant$ 4·1% and 0·3 mm SSI $\geqslant$ 13·0% and all the cement paste samples showing no obvious signs of segregation (bleeding width $= 0$) have 0·15 mm SSI $\leqslant$ 5·1% and 0·3 mm SSI $\leqslant$ 20·1%. There is no definite SSI above which segregation would occur and below which segregation would not occur. Nevertheless, as a rough guide, a 0·15 mm SSI of 5% or a 0·3 mm SSI of 10% may be taken as the acceptable limit.

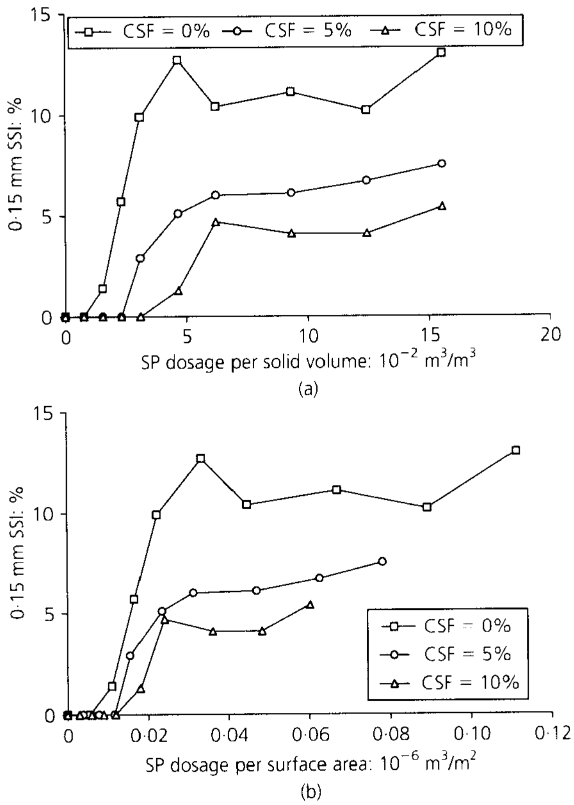

Figure 9. 0·15 mm SSI plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

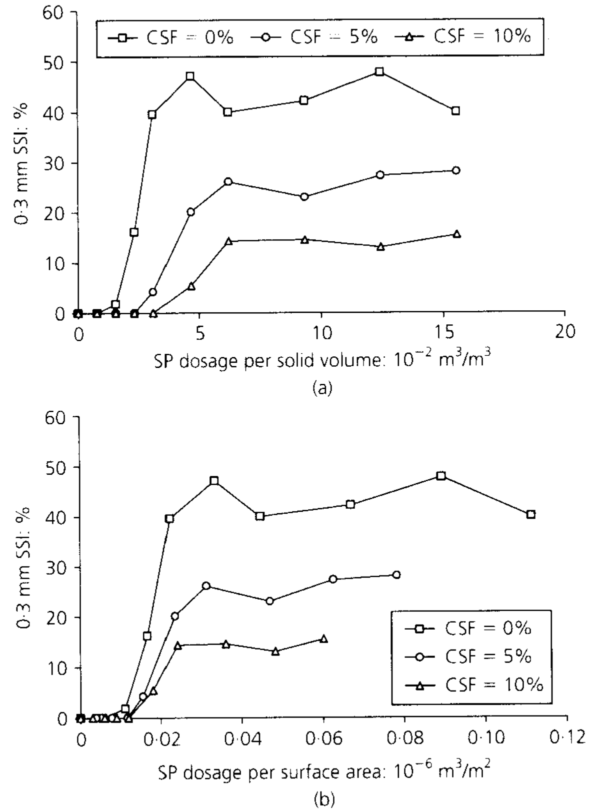

Figure 10. 0·3 mm SSI plotted against SP dosage: (a) SP dosage per solid volume of cementitious materials; (b) SP dosage per surface area of cementitious materials

Taking a 0·15 mm SSI of 5% or a 0·3 mm SSI of 10% as the acceptable limit, it can be found from Figures 9(b) and 10(b) that the SP dosage per surface area causing segregation is within $0{\cdot}015 \times 10^{-6}$ to $0{\cdot}025 \times 10^{-6}$ m^3/m^2, being slightly lower at a lower CSF content and slightly higher at a higher CSF content. In actual practice, the SP dosage should not exceed the minimum of the saturation dosage and the dosage causing segregation. In this particular case, since the dosage causing segregation is lower than the saturation dosage, the maximum allowable SP dosage is governed by the dosage causing segregation. Regarding this issue, it is noteworthy that the maximum dosage recommended by the supplier of the polycarboxylate-based SP has actually exceeded both the saturation dosage and the dosage causing segregation. Therefore, it would be prudent always to determine the saturation dosage and dosage causing segregation by trials before using any SP.

Conclusions

Altogether, 30 cement paste samples having different SP dosages and CSF contents were tested to study the effects of a polycarboxylate-based SP on the flowability, rheology, packing density and cohesiveness of CSF cement paste. The flowability and rheological properties were measured using the mini slump cone test, Marsh cone test and rheometer test, whereas the packing density and cohesiveness were measured using the wet packing test and a mini version of the sieve segregation test. Based on the test results obtained, the following conclusions are drawn.

(*a*) Both the dispersion effect of the SP and the filling effect of the CSF can significantly improve the packing density of cementitious materials.
(*b*) Up to a certain saturation dosage, the addition of the SP can effectively increase the flow spread and flow rate and decrease the yield stress and apparent viscosity of the cement paste. The effectiveness of the SP and the saturation SP dosage are highly related to the surface area of cementitious materials and therefore the SP demand and SP dosage should be quantified relative to the surface area of solid particles.
(*c*) The SP is more effective in reducing the shear stress at low shear rate than in reducing the shear stress at high shear rate. As a result, upon addition of the SP up to a certain dosage, the cement paste would become shear thickening.
(*d*) The addition of SP would decrease whereas the addition of CSF would increase the cohesiveness of cement paste. A mini

version of the sieve segregation test has been employed to measure the cohesiveness of the cement paste samples. Based on the experience gained, it is recommended that to avoid segregation, the acceptance criteria for the 0·15 mm SSI and 0·3 mm SSI should be set as 5% and 10%, respectively. Furthermore, it is found that, within the range of mix parameters covered in this study, the SP dosage causing segregation is lower than the saturation SP dosage.

In general, the SP dosage to be used should not exceed the minimum of the saturation dosage and the dosage causing segregation. Although the saturation dosage and dosage causing segregation have been found in this study to be dependent mainly on the surface area of cementitious materials, they may also vary with the other mix parameters, especially the W/CM ratio. The fixed maximum dosage recommended by the supplier of the SP may not be appropriate. Hence, it would be prudent always to determine both the saturation dosage and dosage causing segregation by trial mixing and testing before using any SP.

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