Pergamon

Cement and Concrete Research 31 (2001) 303–307

CEMENT AND CONCRETE RESEARCH

Communication

Simple test method to assess the relative effectiveness of plasticising chemical admixtures

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Received 2 May 2000; accepted 13 October 2000

Abstract

This paper proposes a simple test method capable of assessing the relative effectiveness of plasticising chemical admixtures. Using a hydrometer, the test measures the relative ability of different admixtures to disperse the individual particles of Portland cement (PC)/silica fume (SF) binder combinations. In a laboratory study carried out to examine the relative performance of three separate admixtures, the test clearly differentiated between PC/SF combinations containing no admixture, normal-range water-reducing admixtures, and high-range water-reducing admixtures. The two high-range water-reducing chemical admixtures considered showed identical dispersive effectiveness in the hydrometer test. This trend was mirrored in a concrete workability study, which indicated that equal dosages of each high-range water-reducing admixture were required to achieve 75-mm nominal slump for mixtures prepared using a range of SF dosages.

Keywords: Dispersion; Workability; Particle size distribution; Admixture; Silica fume

1. Introduction

Due to the demanding performance levels essential for concrete structures built in harsh environments, high-performance concrete containing supplementary cementitious materials, such as silica fume (SF), is frequently specified. Because of their individual physical and chemical properties, supplementary cementitious materials can offer considerable mechanical and durability performance improvements [1–3]. It is well documented in Refs. [4,5], however, that in order to optimise the potential benefits of SF in concrete, it should always be used in conjunction with a plasticising chemical admixture. This is because it is essential to thoroughly disperse the material throughout a given mix, as flocculates of the material may otherwise be formed, and also to compensate for the potential high water demand of the mixture caused by its high surface area. Ensuring dispersion is particularly the case for SF, which is purposely preconditioned to form particle agglomerates as its inherent high particle fineness may otherwise cause handling problems. If plasticising admixtures are not used, it is likely that desired workability, mechanical, and durability performance levels will not be achieved, owing to the higher water demand of the mixture, poor material dispersion, or both [6].

It is important to realise, however, that all plasticising chemical admixtures do not behave similarly in concrete, with performance levels often depending on Portland cement (PC) and admixture compatibility [4]. Therefore, until such time as compatible suites of admixtures and binder materials are available commercially, a major design requirement when proportioning concrete containing supplementary cementitious materials is to choose an admixture that provides optimum levels of particle dispersion. Against this background, this paper proposes a quick and simple test method that is capable of assessing the relative effectiveness of plasticising chemical admixtures for use in concrete containing supplementary cementitious materials.

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0008-8846/01/$ – see front matter
PII: S0008-8846(00)00448-8

2. Proposed test method

2.1. Theory of hydrometer test [7]

Mainly used for soil analysis, the hydrometer test determines the particle size distribution (PSD) of fine-grained materials. In the test, material is mixed in water, and a deflocculating agent is added to ensure full particle dispersion. When this specimen is agitated in a graduate filled with water, the various particles settle at rates according to their size. As a result, an initially uniform density of the suspension will begin to vary, becoming denser at the bottom of the graduate where the particles are accumulating and less dense at the top. At any level in the suspension, the density will be time-dependent due to the size of the particles present. A hydrometer inserted at intervals measures the specific gravity or density of the fluid at a given depth. Stokes' Law relates the particle size, specific gravity of particles and fluid, and particle velocity, and can be applied to hydrometer readings with time to calculate the percentage of particles by mass smaller than a certain given size. In the simple test method proposed in the following sections, this theory has been applied to binder combinations of PC and SF in order to examine the dispersive effectiveness of various deflocculating agents (i.e., water-reducing chemical admixtures). As SF inherently contains particle agglomerates, the dispersive effectiveness of the chemical admixtures considered will be reflected in the resulting PSD.

2.2. Apparatus and hydrometer calibration

Apparatus required for the test includes: (1) hydrometer; (2) balance (0.1-g sensitivity); (3) two graduated cylinders (1-l capacity); (4) timer; (5) spatula; (6) syringe; (7) distilled water supply; and (8) plasticising admixture(s).

To initially calibrate the hydrometer being used, the distance (Z) from the center of the bulb to a range of readings (R) on the hydrometer stem (between 1.000 and 1.040) must first be taken. A plot of R vs. Z should then be made, as shown in Fig. 1(a). In addition, the volume of the hydrometer bulb and the plan area of the graduated cylinder must be recorded. It should be noted that these calibration procedures are only necessary if PSD determinations are required.

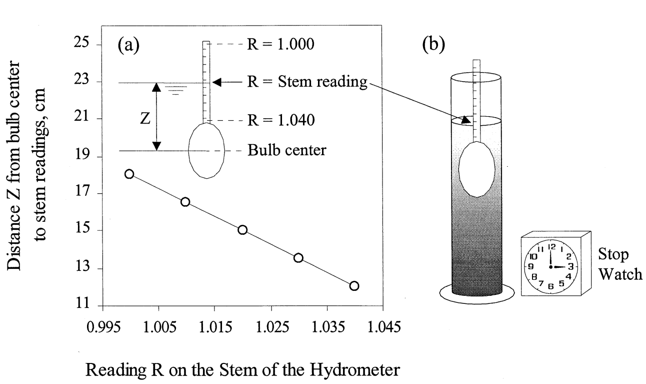

Fig. 1. (a) Example of initial hydrometer calibration plot (as required if establishing PSD). (b) Typical apparatus setup during hydrometer test.

2.3. Proposed test procedure

(1) Fill a graduated jar with distilled water and use this to store the hydrometer initially and between subsequent readings.

(2) Place 50 g of dry binder material in a beaker and add sufficient distilled water to allow thorough mixing. As 10% by mass SF replacement is the maximum dosage typically used in structural concrete, a PC/10% SF by mass binder combination is recommended. If alternative binder combinations are considered, then the constant in Eq. (3), Table 1 must be revised accordingly (see Ref. [7]).

(3) If not a control mixture, then add a dosage of plasticising chemical admixture (preferably within the specified dosage range) to the paste using a syringe. The admixture dosage chosen must remain constant throughout a series of tests to enable direct comparisons between various mixtures. Using a spatula, mix the suspension thoroughly in the beaker until any visible binder flocculates are dispersed (approximately 2 min).

(4) After mixing, wash the specimen into a graduated cylinder and add enough distilled water to bring the level to the 1-l mark.

(5) Mix the specimen and water in the graduate by placing the palm of the hand over the open end and turning the graduate upside down and back. This process should be continued until no material is stuck to the base of the graduate when inverted. Be careful not to spill any of the solution during this process.

(6) After shaking for approximately 30 s, place the graduate on a smooth, horizontal surface, insert the hydrometer, and start the timer [see Fig. 1(b)].

(7) Take hydrometer readings at total elapsed times of 15, 30 s, 1, and 2 min without removing the hydrometer. The suspension should then be remixed and these four readings repeated until consistent sets of results have been obtained.

(8) Having satisfied the above requirement, remove the hydrometer after the 2-min reading, remix the solution, and restart the test. In this case, take no reading until the 2-min one. For this reading and all the following ones, insert the hydrometer just before reading, being sure to dry the stem before doing so.

(9) Take hydrometer readings at total elapsed times of 2, 5, 10, 20 min, etc., approximately doubling the previous time interval. The hydrometer should be removed from the suspension and stored in the graduated jar of distilled water after each reading. Continue readings until the hydrometer reading indicates a value of around 1 (i.e., stem value of 1.001).

2.4. Analysis of test results

Testing times and corresponding hydrometer readings should be recorded in columns 1 and 2 of Table 1,

Table 1
Data analysis from hydrometer test

1	2	3	4	5	6
Time t (s)	Hydrometer reading, R_t	Distance Z_t, (cm)[a] [see Eq. (1)]	Velocity x, (cm/s)[a] [see Eq. (2)]	Diameter D, (cm)[a] [see Eq. (3)]	% less than size D by mass[a] [see Eq. (4)]
15	1.0364[b]	11.4045	0.760	0.00813	100.0
30	1.0332[b]	11.8845	0.396	0.00587	91.2
60	1.0262[b]	12.9345	0.216	0.00433	72.0
120	1.015[b]	14.6145	0.122	0.00326	41.2
300	1.001[b]	16.7145	0.056	0.00220	2.7
600	–	–	–	–	–

(1) $Z_t = Z(\text{obtained from Fig. 1}) - [(\text{volume hydrometer bulb}) \div (2 \times \text{plan area of cylinder})]$
(2) $v = Z_t\,(\text{column 3}) \div t\,(\text{column 1})$
(3) $D = [v\,(\text{from column 4})/11,490\,(\text{constant value – see Ref. [7]})]^{0.5}$
(4) $\%\text{ less than by mass} = [(R_t - 1) \div (R_{\text{initial}} - 1)] \times 100$
[a] Data only required if calculating PSD of mixture.
[b] Example results shown are for the PC/10% SF control mixture.

respectively (this table contains example data to be discussed in following sections). The recommended means of comparing the relative dispersive influence of various chemical admixtures is then simply to plot hydrometer readings vs. time. An example of this approach is discussed in the following sections and shown in Fig. 2(a). Although not the primary aim of this test, if calculating sample PSD, values of distance Z_t, velocity v, particle diameter D, and percentage of material less than size D are also required. These values are calculated as described by Eqs. (1)–(4) in Table 1 and entered into columns 3–6 of the table, respectively. PSD curves may then be constructed by plotting particle diameter values (column 5) vs. the percentage less than size D by mass values (column 6). An example of a PSD curve constructed in this way is shown in Fig. 2(b). The limitations of obtaining a PSD in this way are discussed in more detail in the following sections.

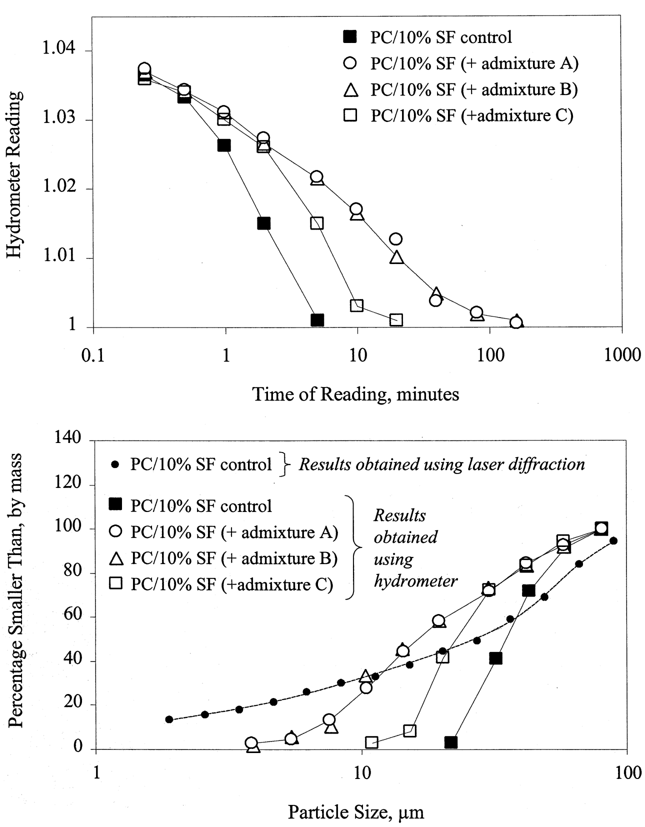

Fig. 2. (a) Decay of hydrometer readings with time (recommended data analysis approach). (b) PSD of material combinations using hydrometer method and laser diffraction analysis (the latter for PC/10% control mixture only).

3. Experimental study

3.1. Phase 1: hydrometer test series

Using the procedure outlined above, a series of hydrometer tests was carried out in Phase 1 to assess the dispersive effectiveness of three separate plasticising chemical admixtures (labelled A, B, and C). Meeting the recommended dosages for each, the quantity of admixture used in each case was 0.75% by mass of the total binder content. Admixtures A and B were high-range water-reducing admixtures, whereas admixture C was an ordinary water reducer. All materials were of sulfonated naphthalene formaldehyde form. A control series was also included where no plasticising chemical admixture was used. The four material combinations considered were: (1) PC/10% SF control (i.e., no chemical admixture used), (2) PC/10% SF + admixture A, (3) PC/10% SF + admixture B, and (4) PC/10% SF + admixture C. In each instance, material combinations were tested eight times using the proposed method, and final hydrometer readings were calculated by taking an average of these.

3.2. Phase 2: concrete workability study

In Phase 2, a concrete workability study was undertaken to establish the dosage of admixtures A and B required to

Table 2
Concrete mixture proportions

SF dosage (% by (% by mass of binder)	Constituent materials, kg/m^3							W/B ratio
		Binder				Aggregate		
	Water	PC	SF	Total	Fine[a]	Coarse[b]		
0	175	350	0	350	851	1040		0.50
1	175	346	3.5	350	850	1040		0.50
2	175	343	7.0	350	848	1040		0.50
3	175	339.5	10.5	350	846	1040		0.50
4	175	336	14.0	350	845	1040		0.50
5	175	332.5	17.5	350	844	1040		0.50
6	175	329	21.0	350	842	1040		0.50
7	175	325.5	24.5	350	841	1040		0.50
8	175	322	28.0	350	839	1040		0.50
9	175	318.5	31.5	350	838	1040		0.50
10	175	315	35.0	350	836	1040		0.50

[a] Pit-sourced fine aggregate.
[b] Greywacke coarse aggregate.

maintain constant workability for concrete containing a range of SF quantities. As shown in Table 2, all concrete mixtures were prepared with a fixed total binder content, coarse aggregate content, and water–binder ratio of 350 kg/m^3, 1040 kg/m^3, and 0.50, respectively. SF dosages were varied in 1% increments between 0% and 10% by mass of binder, and fine aggregate contents were varied accordingly to maintain unit volume. These proportions were chosen to provide a 75-mm slump for the control concrete (i.e., 0% SF replacement) without using any chemical admixtures. As the SF replacement level increased, the dosages of admixtures A and B required to maintain a nominal 75-mm slump were then recorded.

4. Results

4.1. Hydrometer test

Decay of final hydrometer readings with time and PSD curves are given in Fig. 2(a) and (b), respectively. Original results obtained and the data steps undertaken to enable the construction of these curves are additionally given in Table 1 for the PC/10% SF control mixture.

As expected, the general trends in Fig. 2(a) indicate decreasing hydrometer readings with time, reflecting the gradual settlement of the PC and SF particles. Also, apparent from Fig. 2(a) is the influence of using plasticising chemical admixtures, with significantly slower decay rates noted for these mixtures in comparison to the control. Using the time until completion of the test (i.e., when a reading of 1.001 was obtained) as a means of comparison, mixtures containing admixtures A and B behaved very similarly, with full particle settlement taking around 35 times longer (175 min) than for the control (7 min). In contrast, full-particle settlement for mixtures containing admixture C took only four times longer (20 min) than the control. The increased settlement times obtained for mixtures containing plasticising chemical admixtures may be attributed to their dispersive influence on the PC and SF particles. This increased dispersion has clearly been reflected in decreased velocities of settlement. This is in comparison to the control paste, where flocculates of the binder materials were most likely present. Varying settlement times noted between mixtures using the different admixtures may be attributed to the fact that materials A and B were high-range, whereas material C was a normal-range water-reducing admixture. As expected, the high-range water-reducing admixtures provided improved levels of particle dispersion, resulting in prolonged particle settlement times. This effect was almost identical for both materials, suggesting that their influence in concrete performance may be similar.

PSD curves for each material combination are additionally given in Fig. 2(b). As would be expected, the trends shown in this figure clearly reflect those in Fig. 2(a), with slower hydrometer decay rates corresponding to finer PSDs. It should be realised, however, that the accuracy of PSDs obtained from hydrometer analysis is limited. Previous research [8] has shown that hydrometer analysis commonly predicts coarser PSDs in comparison to more advanced analytical techniques, such as laser diffraction or scanning electron microscopy. Such discrepancies have been attributed to dispersion problems rendering extremely accurate measurements in the 0.3–30-μm size range difficult. Indeed, the inaccuracy of the hydrometer test for providing PSDs was confirmed in the current study by additionally assessing the PSD of the PC using laser diffraction analysis. The composite PSD of the PC/10% SF mixture was then calculated by assuming the SF to be fully dispersed with all particles less than 0.5 μm. As shown in Fig. 2(b), the PSD curve constructed using this approach deviates considerably from those obtained using the hydrometer method, highlighting, in particular, the inaccuracy of the latter in the < 10-μm size range. As PSD determinations are not the primary aim of this test, however, this disparity does not detract from the ability of the method to distinguish between the relative dispersive effectiveness of different plasticising chemical admixtures.

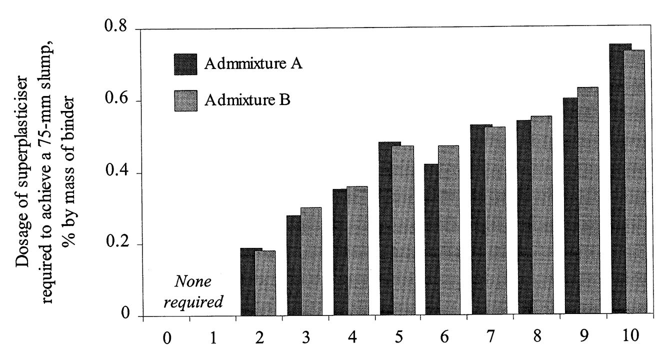

Fig. 3. Dosage of superplasticiser required to achieve a 75-mm slump.

4.2. Concrete workability study

Given in Fig. 3 is the relationship between SF replacement and the dosage of plasticising chemical admixtures A and B required to achieve a nominal concrete slump of 75 mm (70–85 mm). As can be seen, at SF dosages of 0% and 1%, the required slump was achieved without the use of any admixture. Between SF replacements 2% and 10%, however, the dosages required for both admixtures A and B were very similar, with amounts increasing at a fairly constant rate from approximately 0.18% to 0.76% by mass of binder.

Based on the premise that a relationship between particle dispersion (as established from the hydrometer test) and the ability of a given chemical admixture to impart workability in concrete exists, the results obtained from Phases 1 and 2 are in close correlation. In other words, the similar hydrometer decay rates noted for mixtures containing admixtures A and B in Fig. 2(a) are reflected in the fact that similar dosages of these admixtures were required to maintain constant workability in a range of PC/SF concrete mixtures. These results suggest, therefore, that the hydrometer test may provide an effective tool for assessing the relative performance of different water-reducing chemical admixtures in concrete. It is realised, however, that as the current study considered only three chemical admixtures, additional research using a wider variety of admixtures and concrete mixtures is certainly merited to further validate this approach.

5. Conclusions

(1) A simple test method to assess the relative effectiveness of plasticising chemical admixtures for use in concrete has been proposed. Using a hydrometer, the test measures the capability of admixtures to disperse the individual particles of PC/SF combinations.

(2) From a study carried out on three different admixtures, the proposed test method clearly differentiated between PC/SF combinations containing no admixture, normal-range water-reducing admixtures, and high-range water-reducing admixtures. Full-particle settlement for these admixtures occurred after approximately 8, 20, and 175 min, respectively.

(3) The similar dispersive effectiveness of admixtures A and B from the hydrometer test correlated strongly with a concrete performance study, which indicated that equal dosages of each admixture were required to achieve a nominal slump of 75 mm.

Acknowledgments

The South African Foundation for Research Development and Alpha are gratefully acknowledged for supplying materials and funding the reported research project. Thanks are also given for the technical guidance and contributions made by Alpha representatives Mr. V. Blackbeard and Mr. P. Pretorius.

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