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WORKABILITY ADJUSTMENT AND SENSITIVITY OF DIFFERENT FINE CEMENT MIXTURES TO POLYCARBOXYLATE ETHER-BASED SUPERPLASTICIZER

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From previous studies, the most effective superplasticizer on workability was the polycarboxylate ether-based superplasticizer (PCE). For example, when the optimum dose, corresponding to the highest strength, was slightly exceeded, there was a possibility of a sharp drop in strength, even if the segregation was not noticeable. At construction sites, however, the workability adjustment is required to control the slump loss. The question here is how sensitive are different fine cement mixtures that are differently blended with fine additions, like silica fume, to a small increment of this superplasticizer at different water content. In this study, this sensitivity was studied throughout four series of different fine mixtures. For each series, two water to cement ratios (w/c) were used, 0.35, and 0.45, while the superplasticizer dose, the superplasticizer to cement ratio, was varied from 0.011 to 0.0132 (g/g) for each (w/c) ratio. It was found that the small PCE increment caused strength improvement for some mixtures, while it caused strength reduction for others. When the content of both sand and silica fume were reduced, the small increment, along with increasing (w/c) ratio, could significantly decrease the strength by 7.5 MPa. Accordingly, it was concluded that the effect of the dose increment could be related to the actual water content rather than the (w/c) ratio. Hence, an indicator of the actual water content was proposed, which was useful to define a safe method for workability adjustment.

Key words: polycarboxylate ether-based superplasticizer, fine mixtures, silica fume, workability, compressive strength

INTRODUCTION

The most effective superplasticizer on the workability is the polycarboxylate ether-based (PCE), compared with the traditional sulphonate-based superplasticizers. While all kinds of superplasticizers can spread out the cement particles by the electrostatic mechanism, PCE can also apply another scheme for dispersion that is the steric mechanism introduced by its side chains which are not found in the other traditional ones [1,2]. Overlapping the side chains of PCE provides a very dense coating around cement particles [1]. Such a phenomenon greatly impacts the fresh properties of cement mixtures more than the dispersion mechanism of the traditional superplasticizers [1-5] and [6] (p. 271). The yield stress, agglomeration, of the cement paste, can sharply be eliminated from 40 to about 4 Pa by a dosage of 1.9% of PCE to the cement, g/g, while the traditional superplasticizers show almost a stable reduction of the yield stress from 40 to 30 Pa with even larger dosages up to 6% g/g [5]. These observations recall the necessity of having a good prediction about the influence of even a small incremental dose of this superplasticizer before the time of the mixture production. Halim et. al. [8], and Han [9] showed that the behavior of cement mortar mixed with PCE is related to both the binder type and the water to cement ratio. Furthermore, the strength can increase with the increase of the PCE dose until reaching the optimum dose beyond which a drop in the strength is encountered. This study, however, does not aim to track the optimum PCE values but to examine the possible change in the properties of the fresh and hardened mixture when a small increment of the PCE is introduced. Using silica fume is valuable in improving strength [10-17], and durability [18,19], however the effect of PCE on cement mixtures blended with silica fume has not fully understood. Adding fine supplementary cementitious material SCMs has also been known for enhancing the segregation resistance, however, more addition of fine materials may require a lower dose of the superplasticizer to keep stable plasticity [20]. The new generation of superplasticizers, like the one investigated in this paper, greatly enhance the air content [21,22]. Whilst Han et. al., [9], did not record any correlation between the compressive strength and the percentage of air content, which may be attributed to two reasons. Firstly, the ability of the fresh mixture to retain air voids can depend on the fluidity of the mixture; the hardened mixture may contain different air void content. This suggestion, however, has not been approved. Secondly, not only does the content of the air voids matter but also their distribution does matter [23,24]. There is a significant need to adjust the flowability because of the slump loss. The slump loss can result from the impediment mechanism presented by the clay minerals that may be found in aggregate [25-27], or when the concentration of the SP decreases due to the

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high absorption of the SP into the materials, if the mixing time is prolonged, more than four minutes or the composing materials have a good affinity to do so, like limestone [28]. In addition, the slump loss can be a result of water evaporation due to occasional hot weather. Some authors recommended checking the influence of the SP at different water contents but near to that proposed to be used at the time of concrete production [29,30]. Yet, the sensitivity of different fine cement mixes to a given SP increment at different water to cement ratios is still unclear. For this purpose, such sensitivity was studied for cement mortars admixed with PCE and containing varying proportions of silica fume through some tests, namely mixing time, slump, and flowability of fresh mortars, in addition to the compressive strength, and the dry weight of the hardened mixtures. Four series of different fine mixtures were made so that each series contained fixed sand to cement ratio and a fixed proportion of silica fume. For each series, two water to cement ratios (w/c) were used, 0.35, named as case 1, and 0.45, named as case 2, while the superplasticizer dose or the superplasticizer to cement ratio (SP/c) was varied from 0.011 to 0.0132 (g/g) for each case.

EXPERIMENTAL WORK AND DETAILS

Materials

For all mixes, Portland Limestone Cement CEM II/A-L 42,5 R, with a fineness of (0.3776 m2/g), and conforming to EN 197-1 [31] was used. Commercial Silica Fume (SF) was added by (5% to 20%) of the cement weight. The silica fume was with a specific surface area of 22 m2/g based on the BET test, with 90.2% of silicon oxide, and the chemical properties conform to the specification (ASTM C-1240 [32]). The used water was potable. Natural river sand was used and conforming to the Specification (ASTM C 144 [33]). The superplasticizer was polycarboxylic ether based whose density ranges between (1.082~ 1.142) kg/litter, and the chlorine and the alkaline contents were in accordance with the standards EN 480-10 [34] and EN 480-12 [35], respectively.

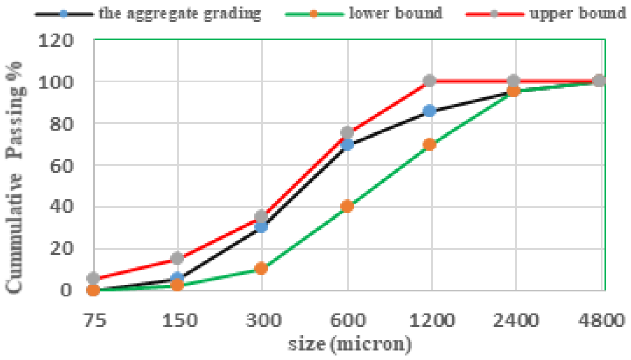

Figure 1: The Aggregate Grading

Mixture Details

Four series of different fine mixtures were made so that each series contained fixed sand to cement ratio, and a fixed content of silica fume, see Table 1. Series one and three were aimed to be reference series for series two and four, respectively. The sand to cement ratio (s/c) was 2.75 for series one and two, and 2.5 for the others.

Table 1: Fresh Mortars: Proportions and Properties

Series	Case	Mixture	Water Content (w/c)	Superplasticizer (SP/c) (g/g)	Mixing Time (min)	Flow Diameter (mm)
Series One	Case 1	SC1SF5	0.35	0.011	3:40	105
		SC2SF5	0.35	0.0132	3:40	106
	Case 2	SC3SF5	0.45	0.011	3:30	156
		SC4SF5	0.45	0.0132	3:30	160
Series Two	Case 1	SC1SF20	0.35	0.011	3	--
		SC2SF20	0.35	0.0132	3	--
	Case 2	SC3SF20	0.45	0.011	3	--
		SC4SF20	0.45	0.0132	3	--
Series Three	Case 1	CS1SF5	0.35	0.011	3:20	111
		CS2SF5	0.35	0.0132	3:20	126
	Case 2	CS3SF5	0.45	0.011	3	179
		CS4SF5	0.45	0.0132	2:40	211
Series Four	Case 1	CS1SF10	0.35	0.011	3	--
		CS2SF10	0.35	0.0132	3	--
	Case 2	CS3SF10	0.45	0.011	3	164
		CS4SF10	0.45	0.0132	3	170

For each series, two water to cement ratios (w/c) were used, 0.35, named as case 1, and 0.45, named as case 2, while the superplasticizer dose or the superplasticizer to cement ratio (SP/c) was varied from 0.011 to 0.0132 (g/g) for each w/c. The caution was taken to ensure precise measure of the materials' proportions.

Mixing, Casting, Curing and Testing

Firstly, sand and silica fume, and cement were mixed until the color uniformity. Then the mix was piled and a pit was made on the top into which the solution of water and superplasticizer , according to the manufactures recommendations, was added. Soon after, the mix was quickly turned over many times with compress by a shovel till the homogeneity was satisfied. Next, mixing time was measured from the time of water added to the beginning of the casting, moreover the flow test was performed according to ASTM C 1437 [36]. As soon as the fresh mortar tests finished, the mortar was molded in to three specimens. Specimens were cured in their moulds for 24 hours at room temperature 29±2°C and 60±10 % relative humidity and then were cured in water until 28 day-age. The casting and the compressive test of specimens were made according to ASTM C109 [37]. For each mixture, three specimens were also molded to determine the oven-dry weight and the apparent density to be utilized as an indicator of the air voids of the hardened specimens. The apparent density was determined as the ratio of the oven-dry mass to the total apparent density of the hardened specimens.

RESULTS AND DISCUSSION

Many trials were made to capture the smallest superplasticizer dose that can trigger mortar plasticity of series 1. For Series 1, mixture SC1 was poorly workable, namely, with only a 105 mm flow diameter, see Table 1. For mixture SC2, adding a small increment of PCE could barely increase the flow diameter by 1 mm. The weak adjustment of the PCE increment for the plasticity of SC2 could be attributed to a deficiency in the water required to cope with the sand roughness. As long as the water content was only increased to 0.45, the fresh mortar properties were improved so that the flowability raised from an average value of 105.5 mm to an average value of 158 mm. The adjustment of the PCE increment for the plasticity was slightly more useful in SC4 than SC2. In other words, increasing PCE dose from 0.011 to 0.0132 enhanced the flow at case 2 more than those at case 1; the required mixing time was 3:40 min, and 3:30 min, for case 1 and case 2, respectively. These observations can be attributed to the fact that the need for water can be mitigated to a certain degree by a longer mixing time [38,39]. On the other side, the small PCE increment caused two different trends to the strength development when w/c is moderately changed. The strength increased from (18 to 25.72) MPa for mixture SC1 versus mixture SC3, while it decreased from (20.4 to 24.8) MPa for mixture SC2 versus mixture SC4, see Fig. 2 and Fig.3. The first positive behavior can be explained by the fact that well dispersion of cement particles supports hydration by better water distribution, which in turn supports the strength development [8, 40]. The second behavior of the strength degradation can be explained by the excess repulsion of cement causing thin mortar layers around sand particles [28], and even some weak zones in the microstructure because of the excess in the air-entraining [21], and the entropic action [41]. The density values of all the mixtures were proportional to the strength values, which confirm these two effects of the PCE increment, see Fig. 4. For series 2, the dryness was so severe to affect the plasticity and the strength, thus increasing w/c was essential to wet the highly fine mixtures of this series [42]. Compared to series 1, the reference series, a different scenario was exhibited by the small PCE increment that is negative for the first case, but positive for the other. The positive effect, for case 2, was in parallel with case 1 of series 1 for the same reason, that is the great role of the PCE in deagglomeration the highly fine mixtures, while the other negative one can be attributed to the hypothesis provided by many researchers [43,44], that the hydration of the cement compounds and the successive reactions can be impeded by the adsorption of the superplasticizer. In this paper, the authors believe in this hypothesis and found it was crucial when the mixtures were super dry. However, particular studies in this regard are required to assess such behavior. Density values were also advocating the PCE effect on the two cases. For Series 3, compared with series1, the reason that stands for somewhat improving plasticity is reducing the roughness of mortars by lowering the s/c ratio to (2.5). Thus, the plasticity of the mortars was slightly higher than that of series 1 so that the flow diameters were larger than all the corresponding ones of series one. In addition, the workability was significantly triggered by the small PCE increment. In fact, the flowability of CS2 was enhanced by 14 mm more than the enhancement of SC2 due to increasing the PCE dose just from 0.011 to 0.0132. A reason for this flowability can be attributed to the literature findings that mortars containing more fines can gain higher unit weight if PCE dose is increased [45]. Thus, in addition to the friction minimizing by fines, the higher flowability of series two is also due to the larger weight yielding to gravity while dropping the flow table. The flow of SC4 increased by 4 mm compared with SC3 while the flow of CS4 increased by 32 mm compared with CS3. Thus, the flow sensitivity to PCE increment was even more severe when more water was added so that the improvement difference was interestingly doubled to 28 mm, the difference between (32 and 4) mm, when w/c raised to 0.45 and s/c raised to 2.5. Stunningly, the mortar CS4 was easy to blend to homogeneity by only 2:40 min while segregation was avoided. Similar to the results of series one, the small PCE increment caused two different effects on the strength development when w/c is changed. In case 1, the strength was slightly improved from (42.24 to 43.52) MPa, due to the PCE increment. While, in case 2, the small PCE increment caused a remarked drop in the strength from (39.12 to 31.6) MPa, for mixture CS3 versus CS4. The positive effect can be explained by the efficient dispersion. Whilst, the other can be attributed to the violated air-entraining because the PCE, like air-entraining agents, can reduce the water surface tension which enables air to enter the mortar [46], and this effect can be significant when increasing the water content, like case 2. The strength decline at case 2 / series 3 is significantly more noticeable than that at case 2/ series 1, see Fig. 3. The water content or w/c is typically named based on the cement mass only. Thus, the lower the s/c is, the higher the actual water content around the composing particles. Because increasing the actual water content can double the effect of the PCE, the mixtures with smaller sand addition are more sensitive to the conjugate increment of w/c and the PCE dosage. On the contrary, the positive fixed effect of the PCE on the strength aspect is well manifest from the results of series 4, and more interestingly, a reduction in the density was associated with higher strength for both case 1, and case 2, which is of great interest in producing high-performance mixtures, lightweight mixtures but with good strength. The optimizing of the microstructure is reachable when the solid density of the cement paste around the sand particles condenses as high as possible which does not necessarily contradict with reducing the apparent density of the whole hardened specimen. The results confirm that of the literature, [28] that no correlation between the proportion of air voids and the compressive strength. Reducing the apparent density, or increasing the air voids, can also refer to the pozzolanic reaction of silica fume that causes the hydrogen bubbles [47], which can well redistribute the air voids for better homogeneity [48]. The increased strength may also refer to the ability of air bubbles to push the micro-silica to the interfacial transition zones (ITZs) [49], resulting in a higher dense matrix [10]. Since this pozzolanic reaction is a subsequent reaction, after initial reactions of cement components with water, [50] (p. 285), this fact may assess that those mixtures of series 2, with a higher proportion of silica fume, may either not greatly be utilized from, or even not experienced, this reaction due to the restricted primary reactions by the PCE dose.

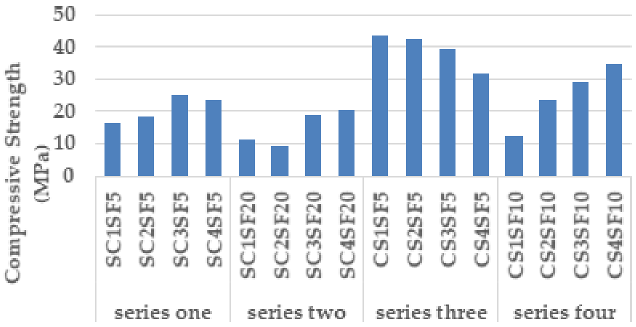

Figure 2: The Compressive Strength Average of Each Mixture

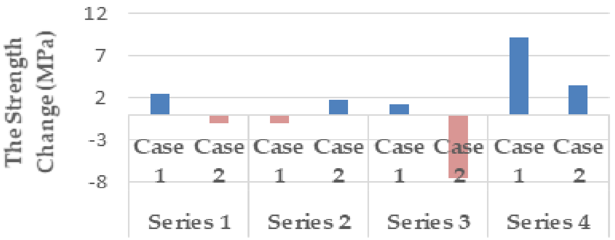

Figure 3: The Compressive Strength Change at each Case of the Series

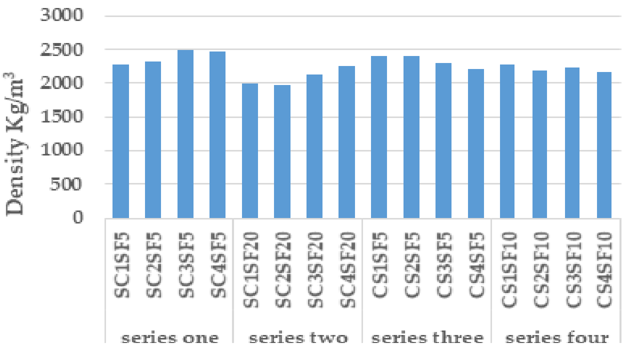

Figure 4: Apparent Density of Each Hardened Mixture

SENSITIVITY PREDICTION BASED ON THE STRENGTH CHANGE

The water volume and the cement volume are important parameters in estimating the strength by the model of Feret [51] (p. 142); using a constant water content as a ratio of the cement mass for different fine mixtures means a difference in the actual water volume due to the difference in the so-called the water to cement distance [42]. Thus, it is worthwhile to analyze strength sensitivity to the PCE increment at different water content based on the fineness of the cement and the SCMs, the size of the aggregate grains, and the water volume. Moreover, given that the surface area of cementitious materials specifies the water demand and thus the strength level [42; 52], and given that the presence of the aggregate less intensely increases the water demand to line up with its usual prescription as saturated surface dry [14] (p. 117); a proposed factor of the water envelope covering the composing particles is expressed as follows:

$$E = \frac{W_V}{S * [ac * \sum(P_{\emptyset} * \emptyset)]} \quad (1)$$

Where:

E: the water envelope expressed as a percentage, unitless

W_V: the water volume

S: the surface area of the cement and the supplementary cementitious materials

ac: the aggregate to cement ratio

$P_{\emptyset}$: the passing percentage of aggregate from sieve size $\emptyset$

From the values of the water envelope, see Table (2), two limits can be inferred that is the moisturizing limit (m) and the saturation limit (s). The moisturizing limit is the lowest water envelop required to initiate cement hydration with the presence of the superplasticizer, which was about 1.4 e-8 in this study, see Fig. 5, while the saturation limit is the maximum water envelope for adsorption with a marginal free water excess above which strength drop is expected, which was about 5.2 e-8 in this study, see Fig. 5. Based on the obtained results of the different series, the strength sensitivity of the mixtures was always one of three situations based on the water envelope and based on the two aforementioned limits. The First situation happens when the water envelope is less than the moisturizing limit, then the superplasticizer can dominantly prevent the hydration. Secondly, when the envelope is greater than the moisturizing limit but lower than the saturation limit, then the dispersion action of the superplasticizer is under control due to the low effect of decreasing the surface tension of the low free water, the not adsorbed water [22], positive sensitivity. Lastly, when the envelope is greater than the saturation limit, the excess air-entraining along with the entropic action of the PCE can reduce the strength, negative sensitivity. In short, the range of the safe water envelop is between the moisturizing limit and the saturation limit. For a given mixture composition, if the sensitivity of the strength to a specified SP dose and one increment of it is tested experimentally at different water to cement ratios, the safe range of the water envelope, and consequently the water to cement ratio can be addressed. The range, however, has to be narrowed to ensure that the strength level is within the design requirements. Exceptionally, the strength sensitivity may not be predicted when the cement is blended with clay minerals due to the so-called poor clay tolerance of this kind of superplasticizers [53]. However, if this problem is avoided in the future by the promising efforts to develop a new improved synthesis of PCE [54,55], the sensitivity prediction may be possible.

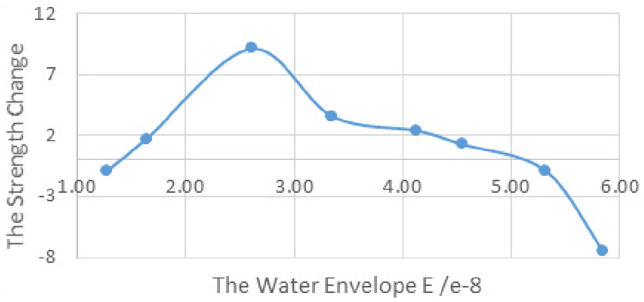

Figure 5. The Relation between The Strength Change and The Water Envelope (E).

CONCLUSIONS

In this study, the effect of increasing the PCE dose from 0.011 to 0.0132 was examined on different mixtures divided into four series. For each series, mixtures contained fixed sand to cement ratio, and a fixed content of silica fume but made with two w/c, namely 0.35 or 0.45. It was shown that increasing the water content can double the effect of PCE increment on the strength drop, but it was difficult to detect that effect by the w/c only. However, there was a relation between the small PCE increment and the water content that was measured according to a proposed parameter called here the water envelope. For safe workability adjustment by a specified PCE increment, two limits for the water envelope should be addressed these are the moisturizing limit (m) and the saturation limit (s). Between these two limits, the water content could be considered safe to introduce the studied PCE increment or any smaller increment. Some other remarks can be summarized as follows:

- A very small increment of the polycarboxylic ether-based superplasticizer from 0.011 to 0.0132 (g/g) could enhance the strength by 9.2 MPa for the stiffest mixture, on the other side it could decrease the strength by 7.5 MPa accompanied with 32 mm increment in the flow diameter.

Table 2: Sensitivity Prediction

Series	Case	The Water Envelope	Situation of The Water Envelope	Strength Change	Sensitivity Type
one	1	4.13 e-8	m<E<s	2.4	positive
	2	5.31 e-8	E>s	-0.88	negative
two	1	1.28 e-8	m>E	-0.92	negative
	2	1.64 e-8	m<E<s	1.72	positive
three	1	4.54 e-8	m<E<s	1.28	positive
	2	5.84 e-8	E>s	-7.52	negative
four	1	2.60 e-8	m<E<s	9.16	positive
	2	3.35 e-8	m<E<s	3.6	positive

- Reducing mixing time to less than 3 min can be an indicator of a possible undesirable loss in compressive strength.
- The intense sensitivity to a small PCE increment was observed when increasing the water to cement ratio combined with the reduction in the sand to cement ratio, or when dealing with relatively stiff fine mixtures.

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