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## **Evaluation of Superplasticizer Performance in Concrete**

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#### ABSTRACT

Superplasticizers are an essential component in modern concretes, providing workability enhancement at low water to cement ratios, and resulting in the production of durable and sustainable concrete. Although the mechanisms leading to superplasticizer performance are well understood, issues of cement – superplasticizer incompatibility are regularly seen in field applications. In this paper, laboratory investigations on cement concrete from a recently concluded research project are reported. The experimental program was divided into two phases: (i) evaluation of the influence of ambient conditions, and (ii) study of the influence of mix parameters - size of mix and speed of mixing - on superplasticizer effectiveness. In both studies, concrete designed for strength of 40 MPa at 28 days (representing typical ready mix composition) with ordinary Portland cement (OPC) and Portland pozzolan cement (PPC) was evaluated for its early age properties, using SNF and PCE based superplasticizers. The results of the investigations reveal the complex nature of interactions between cement and superplasticizers in concrete.

Keywords.Superplasticizer; SNF; PCE; PPC; compatibility; slump retention

### INTRODUCTION

Common problems that arise as a result of incompatibility between cement and water reducers are: rapid loss of workability, excessive quickening / retardation of setting, and low rates of strength gain. Very often, there even exists incompatibility between a particular chemical and a certain batch of the same otherwise compatible cement, indicating that the nature of the problem is complex, and needs further understanding. Moreover, high performance concretes, which are in wide use today, almost always incorporate a mineral admixture or filler such as silica fume, fly ash and limestone powder. This further complicates the physico-chemical behaviour of the cement-based system since the mineral admixtures play an important role in the evolution of the hydration reactions and the availability of free water during the early ages of concrete. The objective of this study was to understand the effect of delayed addition of the superplasticizer,

mix size, mixing speed, and ambient temperature on the early age properties of concrete prepared with SNF and PCE based admixtures.

**Delayed addition of superplasticizer.** Delayed addition is proposed as a means of retaining fluidity in the case of Sulphonated Melamine Formaldehyde (SMF) and Sulphonated Naphthalene Formaldehyde (SNF) based admixtures (Uchikawa et al., 1992, Aiad et al., 2002). The amount of admixture adsorbed reduces when cement hydrates; in other words, adsorption is greater on unhydrated compounds compared to the hydrated phases (Flatt et al., 1998). As a result, when a delayed addition is done, there is more admixture available in the solution to maintain the fluidity. According to Aiad (2003), the optimum delaying time of the admixture is 10 - 15 min, and this time does not depend on the cement and superplasticizer (SP) type. Delayed addition has been shown to result in lesser participation of the polymer in the formation of the organo-mineral phase (Flatt and Houst, 2001). Uchikawa et al. (1995) linked the improvement in fluidity of concretes with later addition of SPs to the increased availability of the admixture in solution. They also found that SNF based chemicals were more sensitive to delayed addition compared to polycarboxylic ether (PCE) and lignosulphonates.

**Concrete at high temperatures.** The placing of concrete at high ambient temperatures adds a new dimension to the problem of incompatibility. Low temperature has been reported to decrease fluidity. This decrease in workability at low temperature cannot be compensated with SP (Gettu et al., 1997). On the other hand, high temperatures increase SP adsorption which increases fluidity (Greisser, 2002). Conversely, temperature increase causes increase in reactivity of C<sub>3</sub>A which causes higher ettringite contents with fine morphology in the presence of SP (Greisser, 2002, Spiratos et al., 2003), thus causing a higher rate of slump loss. The influence of temperature on cement – SP interaction is closely associated with the cement composition, primarily the C<sub>3</sub>A, SO<sub>3</sub>, and alkali contents, along with the cement fineness. Concrete placement temperatures exceeding 40 °C are routinely encountered in practice, which exacerbates the sensitivity of the cement – superplasticizer combination.

#### **EXPERIMENTAL DETAILS**

Aggregates from the Chennai region – river sand as fine aggregate and crushed granite as coarse aggregate – were used for all the studies on mortar and concrete. The particle size distributions of the aggregates conformed to IS 383 (1970). The specific gravity for the river sand varied between 2.5 and 2.7, while the specific gravity for the two sizes of coarse aggregates used (12 mm and 20 mm maximum nominal size) were between 2.7 and 2.9.

Fresh concrete properties were evaluated using the slump test as per ASTM C143 (2010). The SP dosage was varied to obtain an initial slump of 170 mm for all the mixes. The slump retention was measured at 60 min after the initial test. Before the 60 min test, the concrete was mixed for 1 min. At the end of the slump retention test, 10 or 15 cm cubes were cast using the concrete, and compaction was done using a table vibrator. At specific ages (e.g. 7, 14, 28, and 56 days), the compressive strength was determined as per IS 516 (2008).

For the measurement of initial and final setting time, the mortar fraction was obtained from the fresh concrete by sieving through a 4.75 mm sieve. The penetrometer test as per ASTM C403 (2008) was conducted on the mortar to determine initial and final setting time.

One commercially available 53 grade OPC (equivalent to ASTM Type I) and one PPC (Portland Pozzolana Cement) were used to study the interactions with SNF and PCE based superplasticizers in different simulated environments. The slump retention, setting time, and compressive strength development were studied in the environments described in Table 1. M40 concrete was designed for this set of studies, with a cement content of 400 kg/m<sup>3</sup> and a w/c of 0.43. Conditioning of materials and equipment, as well as preparation and storage of concrete specimens, were performed inside the environmental chamber. Specimens were removed after 1 day from the chamber and tested for compressive strength.

No.	Temperature	Relative Humidity	Climatic Type
	(°C)	(%)	
1	6	80	Cold
2	15	95	Cool and humid
3	27	65	Normal
4	35	90	Warm and humid
5	45	40	Hot and dry

Table 1. Different environments simulated in the environmental chamber

**Influence of mixing characteristics.** This study was performed to evaluate the performance of PCE and SNF class of superplasticizers based on mix parameters such as time of addition, size of the mix and speed of mixer. Keeping the size of mix constant, slump measurements for three different times of addition of SP, namely, 2 min, 20 min, and 40 min were performed along with the 60 min retention in each case. Keeping the time of addition (2 min) constant, slump was measured for different size of concrete mixes - 40 kg, 70 kg, 175 kg and 250 kg - along with the 60 min retention in each case. Finally, keeping the size of mix (250 kg) and time of addition (2 min) constant, slump was measured for different speeds of mixer - 15 rpm, 20 rpm and 25 rpm - along with the 60 min retention in each case. For all the cases the 3, 7 and 28 day strengths were measured.

#### RESULTS

**Influence of Temperature and Humidity on Fresh and Hardened Properties of Concrete.**The influence of ambient conditions on the SP dosage required to produce an initial slump of 170 mm is presented in Figure 1. The results clearly indicate that irrespective of the type of cement or SP, the dosage required for a given workability (w/c being constant) is increased at higher temperatures. In other words, the SP demand is increased in hot conditions. It can also be seen from the results that the concretes with PPC require higher SP dosages for the same workability as compared to the concretes with OPC. This is possibly because of higher





Figure 1. Variation in SP dosage at different temperature/humidity conditions

The slump retained at 60 min is shown in Figure 2. In general, the increase in temperature decreased the potential for slump retention. However, in the case of the PPC-SNF combination, the trend was not as clear as in the other cases. Probably, this combination is not as significantly affected as the other combinations with regard to loss in slump at different temperatures. However, the slump retention was uniformly poor at all temperatures for this combination.



Figure 2. Slump retention at 1 hour of concretes in different ambient conditions

At all temperatures (35 °C being an exception), the concretes with PCE performed better than the concretes with SNF, clearly indicating the superior nature of these chemicals. In terms of the cement, PPC performed favourably compared to OPC, which is expected because of the slower rate of hydration of PPC. PPC concretes showed a slump at 1 hour of 80 mm or better at

temperatures up to 35 °C, whereas OPC concretes had slump values of 80 mm or better only up to 15 °C.

The influence of temperature on concrete setting time is presented in Figure 3. The increase in temperature lowered the setting time in general, but the difference between final and initial setting time was not affected significantly by temperature. The results for OPC and PPC, or SNF and PCE, were not much different (as seen from Figure 4), with the exception of the OPC – SNF combination, where there was a clear reduction in the time between initial and final setting. In this case, it appears that the temperature causes a speeding up of both setting and hardening.



Figure 3. Influence of temperature on setting time of concrete

The 1 day compressive strength of the four types of concrete at different temperatures is plotted in Figure 5. As expected, the early age strength of concretes with OPC is better than with PPC. Furthermore, the 1 day strengths of concretes with PCE are better than for those with SNF. However, the differences between the four types of concrete are not large – the largest difference (between OPC-PCE and PPC-SNF) is only of the order of 20%.

The results in this section indicate that temperature is crucial for the attainment of a specific slump as well as the retention of slump in concrete. PCE based SPs show smaller sensitivity to temperature compared to SNF, while PPC performs marginally better than OPC over a wider temperature range.

Effect of Time of Addition of SP on Fresh and Hardened Concrete Properties.Delayed addition of SP is known to present advantages in regard to improved retention of slump, possibly because the adverse competition from SP molecules for adsorption sites on cementitious phases is avoided, and the sulphates have the first opportunity to interact with the cement (this is true for SNF based admixtures). Furthermore, delayed addition also implies that the SP molecules can even disperse the early formed hydrates, thus leading to improved slump retention.



Figure 4. Difference between initial and final setting times for all concretes



Figure 5.1 day compressive strength for all concretes

In this study, the time of addition of the SP was either 2 min (which is the normal schedule), 20 min or 40 min – after the cement and mixing water come into contact inside the concrete mixer. Figure 6 shows the slump retention performance of these concretes. From the results, it is apparent that while PCE is not sensitive to the time of addition of the SP (slump retention performance is similar irrespective of when the admixture is added), the concretes with SNF benefit marginally when the SP is added in a delayed manner. Probably, a change in the mixing and batching schedule in the ready mixed concrete plant can help improve the performance of the SNF based concretes. The compressive strength development of concretes with different times of SP addition is shown in Figure 7 for PCE and Figure 8 for SNF. For concretes with PCE, the early age strength is affected adversely for delayed addition of SP, while there is no significant difference in the strength development for SNF, i.e. the time of addition of the SP does not affect the early age strength for concretes with SNF. It is possible that the delayed

addition of PCE caused a slower rate of hydration in the early stages, leading to smaller strengths at 3 days. The difference is only marginal at 7 days, and completely eliminated at 28 days.



Figure 6. Slump retention for concretes with different times of SP addition (Note: blue color is for 2 min, red for 20 min, and green for 40 min)



Figure 7. Compressive strength development of concretes with different times of addition of PCE



Figure 8. Compressive strength development of concretes with different times of addition of SNF

**Influence of Mixer Speed and Mix Size.** The effect of varying the size of the mix from 40 kg to 250 kg on the slump retention performance of concretes with PCE and SNF is shown in Figure 9. The initial slump increases marginally for concretes with both PCE and SNF, and the corresponding 1 hour slump also increases for PCE based concretes when the mix size is increased. However, in the case of SNF based concretes, the slump retention performance was seen to be adversely affected when mix size was increased (barring the 70 kg mix). It must also be noted that the larger concretes (175 and 250 kg) with SNF showed bleeding initially, and the 70 kg mix with SNF showed bleeding even after 1 hour. Since the SP dosage was decided based on smaller sized mixes – and resulted in bleeding in the larger mixes – this means that some readjustment of the laboratory based designs is necessary before the mix can be adapted for a particular job.

Results for compressive strength of the concrete from mixes of sizes ranging from 40 to 250 kg are presented in Table 2. No clear trends are evident. It appears that compressive strength is not really affected by the size of the mix. The effect of changing speed of a 250 kg mix from 15 to 25 rpm on the slump retention performance of concrete is shown in Figure 10. While the initial slump of the concretes with both SNF and PCE admixtures increased with an increase in the mix speed, the slump value at 1 hour was 70 mm in all the six cases. In other words, the final slump value attained was similar irrespective of the speed of the mix. The study would have to be conducted on different types of mixers to ascertain whether this trend is universal. Compressive strength results presented in Table 3 indicate that mixer speed does not cause any significant change in the 3 and 7 day strengths.

	PCE Compressive strength (MPa)			SNF Compressive strength (MPa)		
Mix size (kg)	3 days	7 days	28 days	3 days	7 days	28 days
40	22.8	30.0	39.8	22.4	28.6	39.0
70	22.5	31.0	40.0	25.7	31.5	42.0
175	20.0	25.9	40.0	21.5	26.7	41.0
250	22.6	31.3	41.0	23.1	32.0	41.6

Table 2. Compressive strengths for concretes from different mix sizes



# Figure 9. Slump retention of concrete from different mix sizes (Note: blue color is for 40 kg, red for 70 kg, green for 17 kg, and violet for 250 kg)

Table 3. Compressive strengths for concretes with different mixer speeds

	PCE Compressi	ve strength (MPa)	SNF Compressive strength (MPa)		
Mix speed (rpm)	3 days	7 days	3 days	7 days	
15	23.1	35.3	22.2	32.0	
20	21.9	33.3	24.1	35.0	
25	20.9	32.5	25.0	36.1	



Figure 10. Influence of mix speed on slump retention with PCE and SNF

#### CONCLUSIONS

- 1. Ambient conditions, particularly temperature, had major influence on the superplasticizer demand, slump retention, setting time, and early strength of the concrete. The SP demand for a given slump as well as the 1 day compressive strength increased with a rise in the temperature, while the slump retention and initial setting time decreased. Temperature was not seen to influence the setting characteristics beyond the initial set.
- 2. From the temperature studies on concrete, the performance of PPC was seen to be superior compared to OPC.
- 3. The time of addition of SNF based SP was seen to have a significant influence on the slump retention of concrete, while PCE did not show sensitivity to the time of addition. However, in terms of the 1 day strength performance, the delayed addition of SNF did not show any effect, while the delayed addition of PCE decreased the early age strengths (28 day strengths were not affected).
- 4. While the increase in mix size resulted in a uniform increase in the initial slump for both PCE and SNF based concretes, the slump retention for PCE was not affected, but the slump retention of SNF concretes was affected adversely. The increase in mixing speed was seen to uniformly increase the initial slump, while not affecting the final slump after 1 hour.

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