Optimization of superplasticizer in portland pozzolana cement mortar and concrete

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Abstract: Chemical Admixtures are added to concrete at the time of mixing of its constituents to impart workability. The requirement of right workability is the essence of good concrete. It has been found that the use of optimum use of admixtures is very important since low dosage may result in loss of fluidity and over dosage could lead to segregation, bleeding, excessive air entrainment etc in concrete. Hence it is essential to find optimum dosage of superplasticizer for getting good strength and workability. But large number of trial tests are required in the field to find the saturation dosage of superplasticizer in concrete which requires more materials and consume more time. The paper deals with developing a co-relation between the quantity requirements of superplasticiser in mortar to that of cement concrete to get good workability. In this work for preparing mortar and concrete 4 brands of locally available Portland pozzolana cement (PPC) and superplasticizer (SP) belonging to 4 different families namely Polycarboxylate Ether (PCE), Lignosulphate (LS), Sulfonated Naphthalene Formaldehyde (SNF) and Sulfonated Melamine Formaldehyde (SMF) are used. Two different brands of SP’s are taken from each family. Workability study on the superplasticized mortar with cement to sand ratio 1:1.5 and water cement ratio of 0.4 was performed using marsh cone and flow table test and workability study on the concrete with same cement/sand ratio and water cement ratio was done using slump cone and flow table test. Saturation dosage of superplasticizer in mortar and concrete determined experimentally was compared to study the correlation between two. Compressive strength study on concrete cubes were done on concrete mixes with a superplasticizer dosage corresponding to the saturation dosage and a comparative study were done to analyse the improvement in the compressive strength with addition of superplasticizer from different family.

1. Introduction

Today superplasticizer (SP) is one of the most important constituents of any concrete mix. These micromolecular organic agents are grouped in four families based on their chemical contents as sulphonate melamine formaldehyde, sulphonate naphthalene formaldehyde, modified lignosulphonates, and copolymers containing sulphonic and carboxyl groups [1, 2, 3, 4, 5]. Hydroxyl, sulphonate or carboxylate groups attached to the main organic unit of the superplasticizer which is

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usually anionic, gives the water solubility to these admixtures [6, 7]. The workability and compressive strength of concrete increases with the use of superplasticizer [2, 8, 9]. Workability improvement by the superplasticizer in the concrete is mainly by increase in the surface potential force, the solid-liquid affinity, and the steric hindrance mainly in PCE based superplasticizer.

Large amount of CO$_2$ is emitted to the atmosphere from the cement plants by the decomposition of CaCO$_3$ during the production of cement. The environmental pollution can be reduced by reducing the cement consumption by replacing the cement with the pozzolanic materials like flyash. They are the byproduct from the coal fired thermal power plant. Because of its pozzolanic activity, the calcium hydroxide produced during the hydration of cement will be converted to Calcium Silicate Hydrate [3,5].

This way strength and durability of the flyash blended hardened cement matrix will be improved. Because of the slow reaction of the pozzolanic material, the problem due to thermal cracking can also be avoided [10]. It is usually reported that the addition of mineral admixtures reduces the workability due to the higher surface area and consequently increase the water and superplasticizer demand [6]. But before using the superplasticizer in concrete, the study on the cement-SP interaction has to be performed. Unexpected and undesirable tendencies like bleeding, increased setting time and low cost-effectiveness were experienced by the usage incompatible cement and superplasticizer. Microstructure of the heavily and moderately superplasticized concrete were studied by Sidney Diamond (2006) to determine the modification in patched microstructure of the base OPC concrete with superplasticizer (PCE based) addition. From the study it is observed that patched structure is present in both moderately and heavily superplasticized concrete also but the porous patches are appeared to be relatively fewer in number and generally smaller in size in heavily superplasticized concrete.

In this work to prepare cement mortar four brands of PPC namely C1, C2, C3, C4 and four families of SP’s namely Polycarboxylate Ether (PCE), Lignosulphate (LS), Sulfonated Naphthalene Formaldehyde (SNF) and Sulfonated Melamine Formaldehyde (SMF) are used. Two different brands of SP’s are taken from each family. Marsh cone test and flow table tests were performed to determine the flow behavior of PPC mortar and to find the saturation dosage of superplasticizer in cement mortar of cement to sand proportion 1:1.5.

Main parameters which affect the concrete flow behavior are paste rheology, aggregate volume fraction and aggregate particle size distribution. So Influence of aggregates and the aggregate-paste interfaces need to be studied to correlate the paste results with concrete [11, 10]. Hence for the study on the flow behavior, concrete mixture of same cement to sand proportion as that of mortar was prepared using four brands of cement and four families of superplasticizer and flow table test and slump cone tests were performed. Saturation dosage of superplasticizer in concrete was determined from the test result and an attempt is made to correlate the flow behaviour of cement mortar with that of concrete. All the tests were carried out at a water-cement ratio of 0.40.

2. Material Details

2.1 Cement

Physical properties of the cement tested according to the codal specifications [12, 13, 14] are listed in table1.

| Tests                  | C1     | C2     | C3     | C4     |
|------------------------|--------|--------|--------|--------|
| Fineness (%)           | 1.06   | 1.83   | 0.93   | 2.26   |
| Specific gravity       | 2.83   | 2.89   | 2.75   | 2.77   |
| Consistency            | 36     | 35     | 35     | 38     |
| Initial setting time   | 175    | 120    | 150    | 170    |
| Final setting time     | 210    | 200    | 190    | 275    |
2.2 Aggregate
Properties of the fine aggregate and coarse aggregate tested according to the codal provision [15, 16] is tabulated in table 2 and table 3 respectively. The results were compared with the specification given in the IS code [17] and found suitable for use.

Table 2. Properties of fine aggregates.

| Tests                        | Results  |
|------------------------------|----------|
| Maximum size of the aggregate(mm) | 4.75     |
| Fineness Modulus (%)          | 2.864    |
| Grading Zone                  | II       |
| Specific Gravity              | 2.517    |
| Bulk Density(kg/m³)           | 1709     |
| Percentage Voids (%)          | 56.169   |
| Maximum Percentage of Bulking (%) | 18.3    |
| Corresponding Moisture Content (%) | 2       |
| Water Absorption (%)          | 2.14     |

Table 3. Properties of coarse aggregate

| Tests                        | Results  |
|------------------------------|----------|
| Maximum size of the aggregate | 20 mm    |
| Fineness Modulus (%)          | 7.14     |
| Specific Gravity              | 2.724    |
| Bulk Density(kg/m³)           | 1530     |
| Percentage Voids (%)          | 84.12    |
| Water Absorption (%)          | 0.05     |

2.3 Superplasticisers
Solid content of SPs was determined according to IS 9103:2004 - Annex E [18]. The density of SPs obtained from the data sheet provided by the respective manufacturer is also tabulated along with the solid content in table 4.

Table 4. Properties of the superplasticisers

| Designation | Density (kg/litre) | Solid Content (%) |
|-------------|--------------------|-------------------|
| PCE-1       | 1.09               | 38.39             |
| PCE-2       | 1.09               | 34.88             |
| SNF-1       | 1.2                | 38.56             |
| SNF-2       | 1.24               | 33.32             |
| SMF-1       | 1.15               | 35.21             |
| SMF-2       | 1.2                | 22.06             |
| LS-1        | 1.18               | 31.58             |
| LS-2        | 1.19               | 33.33             |

2.4 Mortar and Concrete Mix Details
Mortar of cement to sand proportion 1:1.5 and water to cement ratio 0.4 is used for this study. Concrete mixes were prepared for a characteristic compressive strength of 35MPa using the codal provision IS 10262 [19] and adjustment were done in the ingredient quantity for making the cement/sand proportion as 1:1.5. The concrete mixture details used for this study are given in the table 5. Water correction for the aggregate moisture was done by considering water absorption of aggregate.
### Table 5. Mix proportion of concrete.

| Ingredients of concrete | Quantity (kg/m³) |
|------------------------|------------------|
| Cement                 | 450              |
| Fine aggregate         | 675              |
| Coarse aggregate       | 1095             |
| Water                  | 180              |

3. Test Details

3.1 Fresh stage properties of mortar and Concrete

3.1.1 Mortar

Flow behavior of cement mortar was studied by using Marsh cone test and flow table tests. The mixing sequence suggested in literature [20] was adopted for preparing the mortar.

a) Flow Table Test

Flow Table test was used to find the fluidity of the mortar (by measuring the spread diameter) and the saturation dosage of SP in the cement mortar. In this study, a flow table of diameter 350 mm with a conical mould of 70 mm internal diameter on the top end, 100 mm internal diameter on lower end and 50 mm height was used. The oiled mould was placed on the table and filled with mortar. After vertically withdrawing the mould, the flow table with mortar was jolted 25 times, and the spread diameter is measured in four directions and average is calculated. The test gives the fluidity of mortar in terms of the spread diameter. From the spread diameter Vs SP/C % graph, saturation dosage of SP was calculated. The saturation point is the dosage of superplasticizer beyond which further addition of SP does not increase spread diameter but result in segregation.

b) Marsh Cone Test

The Marsh Cone test is also used to evaluate the fluidity and the saturation SP dosage in the cement mortar. In this study, a metal cone (as per European standards EN 445 ASTM C 939) with a bottom nozzle of diameter 13 mm was employed. An initial volume of 1000 ml of mortar was poured into the cone and the time required for 500 ml of mortar to flow down through the nozzle (collected in a beaker) was observed. In all cases, there is increase in fluidity with increase in the dosage of SP up to certain dosage.

The test gives the fluidity of mortar in terms of the flow time; higher the flow time, lower is the fluidity of the mortar. In this method, superplasticizer dosage corresponding to an internal angle of 140° ±10° is taken as the saturation dosage. This criterion has been proposed by Gomes [21] on the basis of about 200 tests on superplasticised cement pastes.

3.1.2 Concrete

In this work, the flow behavior of cement Concrete was studied using flow table test and slump cone test. For preparing the concrete mix, the material were dry mixed followed by the mixing sequence adopted in an earlier study [20]. The total mixing time was 5 minutes.

a) Flow Table Test

Workability test on concrete was performed by conducting flow table test [22] using flow table and mould of dimensions specified in the IS code [23]. In that test the concrete was filled in two layers and the mould was lifted vertically upwards and jolted 15 times in 15 seconds. The spread diameter was measured in six directions and the average was noted. The test gives the fluidity of concrete in terms of the spread diameter; higher the spread diameter, higher is the fluidity of concrete. The saturation point is the dosage beyond which further addition of SP does not increase spread diameter but can produce segregation; the saturation dosage can taken as the optimum dosage for given concrete mixture.
b) Slump Test
Workability was also measured by conducting slump test on concrete mixtures at the saturation dosages (as obtained in flow table test) according to the procedure given in the IS code [22] using a slump cone of dimension specified in IS code [24]. The concrete was placed in slump cone in four layers and sufficient tamping was given to each layer. The slump value was measured as the subsidence of concrete on removal of mould.

4. Results and Discussion

4.1 Fresh stage properties of mortar and concrete

a) Mortar
The saturation dosage of superplasticizer in mortar was calculated from both marsh cone and mini slump test. In few mixes small difference was observed in the saturation dosages obtained from both the tests. In such cases the saturation dosage from the marsh cone is considered for comparative evaluation. The graphical representation of the marsh cone flow time for different dosages of SP for representative samples are shown in the figure 1. The saturation dosage of superplasticizer (SP/C %) for different cement superplasticizer combination are given in the table 6.

![Figure 1. Marsh cone flow time curve for C2](image)

**Table 6.** Saturation dosage of superplasticizer in mortar.

| SP      | C1  | C2  | C3  | C4  |
|---------|-----|-----|-----|-----|
| PCE1    | 0.3 | 0.5 | 0.2 | 0.1 |
| PCE2    | 0.3 | 0.4 | 0.5 | 0.5 |
| LS1     | 0.6 | 0.5 | 1.3 | 0.4 |
| LS2     | 0.5 | 0.4 | 0.4 | 0.5 |
| SNF1    | 1   | 0.8 | 0.9 | 1   |
| SNF2    | 1   | 0.7 | 0.8 | 0.6 |
| SMF1    | 0.2 | 0.3 | 0.3 | 0.4 |
| SMF2    | 0.8 | 1   | 0.7 | 1   |

b) Concrete
Flow table spread of concrete for different dosages of SP (only representative samples) are shown in the figure 2. Results of flow table test corresponding to the saturation dosage of superplasticizers are tabulated in table 7. Slump test results at the above saturation dosages are also presented. However many of the mixtures did not show a slump subsidence.
Table 7. Spread diameter and Slump Value at saturation dosage of SP in concrete.

| SP    | Cement | Saturation dosage of SP in concrete (%) | Average Spread diameter (cm) | Slump Value (cm) |
|-------|--------|----------------------------------------|-----------------------------|------------------|
| PCE1  | C1     | 0.3                                    | 50.33                       | 19.25            |
|       | C2     | 0.5                                    | 53.83                       | 0                |
|       | C3     | 0.2                                    | 50.66                       | 0                |
|       | C4     | 0.1                                    | 41.06                       | 0                |
|       | C1     | 0.4                                    | 65                          | 0                |
| PCE2  | C2     | 0.5                                    | 56.60                       | 0                |
|       | C3     | 0.8                                    | 61.20                       | 0                |
|       | C4     | 0.6                                    | 59                          | 0                |
|       | C1     | 0.9                                    | 57.50                       | 0                |
| LS1   | C2     | 0.8                                    | 55.50                       | 0                |
|       | C3     | 1.3                                    | 57.92                       | 0                |
|       | C4     | 0.6                                    | 55.67                       | 19.25            |
|       | C1     | 0.7                                    | 55                          | 18.75            |
| LS2   | C2     | 0.5                                    | 42                          | 0                |
|       | C3     | 0.5                                    | 47.33                       | 19               |
|       | C4     | 0.6                                    | 49.33                       | 16.25            |
|       | C1     | 0.4                                    | 40.58                       | 16.50            |
| SMF1  | C2     | 0.7                                    | 41.25                       | 14.25            |
|       | C3     | 0.7                                    | 52.75                       | 16.75            |
|       | C4     | 0.5                                    | 58.33                       | 0                |
|       | C1     | 1.0                                    | 48.37                       | 13               |
| SMF2  | C2     | 1.3                                    | 39.40                       | 0                |
|       | C3     | 0.9                                    | 46.25                       | 12.5             |
|       | C4     | 1.1                                    | 50.25                       | 14               |
|       | C1     | 1.1                                    | 60.25                       | 0                |
| SNF1  | C2     | 0.9                                    | 61.50                       | 0                |
|       | C3     | 1.0                                    | 60                          | 0                |
|       | C4     | 1.1                                    | 59.42                       | 0                |
|       | C1     | 1.1                                    | 56.42                       | 0                |
|       | C2     | 0.8                                    | 61                          | 0                |
| SNF2  | C3     | 0.9                                    | 60                          | 0                |
|       | C4     | 0.7                                    | 57.33                       | 16               |
c) Saturation dosage of Mortar and Concrete

Comparison of saturation dosage of superplasticizer in mortar and concrete is shown in the figure 3(a) and 3(b). Most of the cases slump test didn’t give a realistic figure so the flow table spread was used for comparing the workability of concrete at saturation dosage of super plasticizer.

It is found that the performance of the PCE based superplasticizer are better than other families of SPs in concrete. This is mainly due to the steric hindrance between the cement particles in addition to electrostatic repulsive force [5]. But it is observed that not only SPs of different basic groups behave differently, but even the SP within the same basic group also behaves differently. Similar trends were observed in an earlier study also [25]. This change in behavior is mainly due to difference in their synthesis process [1, 26]. The cement concrete saturation dosages of SP are observed to be at a higher range than that of cement mortar. Similar observations were mentioned in another study on OPC-SP combination properties [20]. This has been attributed to the adsorption of superplasticizer on the fines in the crushed coarse aggregate and the consequent reduction in the amount of superplasticiser availability for the cement paste.
4.2 Hardened state properties of concrete
Cubes of 100 mm size for testing compressive strength were cast for the concrete mixtures made with superplasticizer corresponding to the saturation dosage. Water curing was given for concrete cubes and tested for 7th and 28th day compressive strength, which are tabulated in the table 8.

| Cement | Superplasticizer | 7th day compressive strength (MPa) | 28th day compressive strength (MPa) |
|--------|------------------|-----------------------------------|------------------------------------|
| C1     | PCE1             | 28                                | 42                                 |
|        | PCE2             | 28                                | 43                                 |
|        | SMF1             | 32                                | 41                                 |
|        | SMF2             | 29                                | 40.5                               |
|        | SNF1             | 26                                | 41                                 |
|        | SNF2             | 22.5                              | 40                                 |
|        | LS1              | 18.2                              | 41                                 |
|        | LS2              | 19                                | 40                                 |
|        | PCE1             | 29                                | 40                                 |
|        | PCE2             | 30                                | 42                                 |
|        | SMF1             | 33                                | 42                                 |
|        | SMF2             | 30                                | 45                                 |
|        | SNF1             | 29                                | 40                                 |
|        | SNF2             | 27.5                              | 42                                 |
|        | LS1              | 23                                | 40                                 |
|        | LS2              | 31.25                             | 42                                 |
|        | PCE1             | 31                                | 41                                 |
|        | PCE2             | 31.5                              | 40                                 |
|        | SMF1             | 28                                | 39.5                               |
|        | SMF2             | 33.2                              | 41.25                              |
|        | SNF1             | 28                                | 38                                 |
|        | SNF2             | 25.5                              | 39                                 |
|        | LS1              | 28                                | 41                                 |
|        | LS2              | 25                                | 41.75                              |
|        | PCE1             | 35                                | 42                                 |
|        | PCE2             | 35                                | 43                                 |
|        | SMF1             | 29.5                              | 41                                 |
|        | SMF2             | 28.5                              | 40.75                              |
|        | SNF1             | 31.5                              | 41.5                               |
|        | SNF2             | 29                                | 39.5                               |
|        | LS1              | 22                                | 39.5                               |
|        | LS2              | 29.5                              | 40.5                               |

Superplasticised PPC concrete mixtures had higher compressive strength than those of control mixtures. This observation is consistent with the observation of other researchers [11, 20, 27, 3, 4, 28-31, 32]. Improvement in the compressive strength of the superplasticized concrete mixtures is mainly due the improved compaction obtained in the concrete by the addition of superplasticizer and also due to the improvement in the pore structure[33]

5. Conclusions
Following conclusions are made on the basis of tests conducted on superplasticized mortar and concrete.

1. Saturation dosages of superplasticizer in cement concrete are observed to be at a higher range than that of cement mortar. This is due to the adsorption of superplasticizer on the fines in the
crushed coarse aggregate and consequently, the superplasticiser amount available in the cement paste decreases and more amount of superplasticizer is required to maintain the workability.

2. The difference in the superplasticizer quantity for optimum dosage in mortar and concrete is less in the case of PCE based superplasticisers due to its better dispersing action contributed by steric hindrance and electrostatic repulsive force.

3. After the saturation point, the increase of SP dosage doesn’t show any improvement of the workability of mortar/concrete rather it decreased the spread and increased the flow time. Addition of SP beyond saturation dosage causes segregation of the mortar/concrete.

4. It is observed that with PCE based superplasticiser, even a slight increase beyond its saturation dosage results in bleeding and segregation. So good quality control is required in concrete construction if PCE based superplasticisers are used.

5. Due to enhancement in workability, superplasticized concrete yielded higher compressive strength than the PPC control mixtures. Compressive strength variation is only marginal for concretes made using different families of superplasticisers, at their optimum dosages.

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