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Studies on the compatibility of Ordinary Portland Cement with Polycarboxylate Superplasticisers

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ABSTRACT

The addition of superplasticisers along with mineral admixtures like fly ash and slag in concrete improves the strength and workability of concrete at lesser water cement ratios. To get this benefit, the compatibility between cement and admixtures must be studied. SP's are normally adsorbed on the cement particles and sometimes the adsorption will not be even and slightly unreliable. This is due to the clinker composition of cement and the type of SP's used. Various combinations of materials including mineral admixtures affect the behaviour of the cement–based system and become incompatible like slump loss, delayed setting of concrete etc. In the present work, four brands of Ordinary Portland Cements are selected and is checked for its compatibility with 4 types of PCE superplasticisers available in the market using Marsh cone test. The results of these tests conducted on the cement paste is analyzed to find out the optimum dosage of superplasticiser for all the four brands. Chemical analysis and XRD analysis is done for the 4 types of cements to study the behaviour. The work concludes that the presence of MgO in ordinary portland cement affects the cement superplasticiser compatibility to greater extent.

Keywords: *Marsh Cone test, Marsh Cone Time, saturation Point, optimum Dosage of Superplasticisers, Poly Carboxylate Ether, Workability.*

1. INTRODUCTION

Concrete has changed over the course of time to cope with the day to day challenges and the use of Mineral and chemical admixtures have been a magic tool for the same. The ideal concrete is the one having high workability at low water cement ratio. To achieve the desired properties in a good quality concrete, these chemical and mineral admixtures are added to the cement. Especially the superplasticers are the high range water reducers used for the proper dispersion of the cement particles in a concrete suspension (Ramachandran, 1995). The addition of superplasticiser will reduce the water cement ratio of the concrete to the range of 0.20 to 0.30, without affecting its strength and workability. These admixtures increase the durability of concrete by making the concrete more and more dense.

These days, due to the availability of different types of admixtures and cement in the market, there is a lot of flexibility in choosing the right composition of the concrete according to the desired parameters, keeping in mind the overall economy and environmental safety. Admixtures, especially new superplasticizers are being developed regularly, which dramatically change the properties of the concrete. But if there is any incompatibility issue arises between the cement and the admixture, it may cause rapid loss of workability, excessive retardation or setting issues and low rates of strength gain, in addition to the economic loss. Optimum dosage of PCE superplasticiser for each brand of cement with four different sets of admixtures are found. The physical and chemical analysis is carried out on the cement samples to study the behaviour.

2. SUPERPLASTICIZERS

Types of Superplasticizers
Superplasticizers can be classified into four groups:

a) Sulfonated melamine–formaldehyde condensates (SMF)
b) Sulfonated naphthalene-formaldehyde condensates (SNF)
c) Modified lignosulfonates (MLS)
d) Polycarboxylate Derivatives (PCE based)

2.1 Property of superplasticizers

Cement particles have a strong tendency to flocculate when they come in contact with water. Even atmospheric moisture is sufficient to result in flocculation of cement. Superplasticizers are the dispersants, having a polar hydrophilic group attached to a non-polar hydrophobic organic chain with some polar groups, which prevent the flocculation of fine particles of cement. The polar groups in the chain get adsorbed on the surface of the cement grains, and the hydrophobic end with the polar hydrophilic groups at the tip point outwards from the cement grain. The hydrophilic tip is able to reduce the surface tension of water, and the adsorbed polymer keeps the cement particles apart due to electrostatic repulsions. Thus the fluidic property of the concrete increases and the concrete is workable even at lower water–cement ratios. [The Concrete Portal (2013)]

2.2 Properties of the PCE superplasticizers used in this study

Table-1 shows the performance test data for all the 4 PCE superplasticizers used in this study.

Table 1: Properties of the PCE superplasticisers

S no. and coding	Aspect	Relative density	pH	Chloride ion content
S1	Reddish brown liquid	1.08+/-0.02 at 25 deg C	>=6	Nil
S2	Light yellow coloured liquid	1.05+/-0.02 at 25 deg C	>=6	Nil
S3	Light yellow coloured liquid	1.08+/-0.02 at 25 deg C	>=6	Nil
S4	Light yellow coloured liquid	1.05+/-0.02 at 25 deg C	>=6	Nil

3. MARSH CONE TEST

Fig. 1: Marsh cone test setup.

Marsh cone test is reliable and simple method to study the rheological properties of cements and mortars. Flow time of cement/mortar through marsh cone is indicator of viscosity, which depends upon cement superplasticizer compatibility. It is widely used to study cement superplasticizer compatibility and to determine optimum superplasticizer dosage of a specific cement-superplasticizer combination. The Marsh cone test setup is shown in figure 1.

Apparatus

Apparatus consist of a conical brass vessel held on a wooden stand with an orifice of 8mm or 10mm at the bottom. A stop watch is required to measure the time taken by 1 liter of cement paste to pass through the vessel. A mortar mixer is also needed to prepare the cement paste with desired w/c ratio. [Pamnani et. al. (2013)]

Materials Used

OPC 53 cements (C1, C2, C3 and C4), PCE Superplasticisers S1, S2,S3 and S4

Methodology

In the present study, 1L of cement paste is prepared in mortar mixer using 2 kg of cement and water at w/c ratio of 0.35. Water is added in two steps- 70% of water is added in beginning of mixing and rest 30% of water is mixed along with PCE superplasticizer (SP), and added afterwards. Cement slurry is prepared with the w/c ratio of 0.35 and admixture dosage of 0.4 %. 1 liter of cement slurry is made to flow through marsh cone after 5 minutes of mixing and time in seconds is noted using a stopwatch. The procedure is repeated gradually increasing the PCE S.P. dosage in steps of 0.2%. Similar tests are conducted for rest of the SP and graphs are plotted with time as Y axis and PCE S.P. dosage as X-axis.

Material Properties

Cement

Cement is the binding material in concrete mix and is costly material among all other materials in concrete. The properties of cement have bearing on the behaviour of concrete. OPC 53G cement is used in the present experimental work. The physical and chemical properties of the cements used in the experimental work is given in table 2 and 3.

Table 2: Physical and Chemical properties of the OPC53 used

Characteristics	C1	C2	C3	C4	Requirements of IS:269: 2015
Consistency (%)	28.25	24.50	27.75	25.50	-
Initial setting time, (minutes)	170	240	305	165	Min 30
Final setting time, (minutes)	220	285	365	210	Max 600
Fineness(m^2/ Kg)	310	295	308	314	Min 225
Compressive strength (MPa.)					
1 day	26.7	20.7	22.5	20.1	-
3 days	42.9	39.6	43.9	40.2	Min 27

7 days	49.8	48.6	51.7	53.5	Min 37
28 days	57.9	63.7	58.8	68.6	Min 53

Table 3: Chemical properties of the cement C1, C2, C3 and C4

Characteristics	C1	C2	C3	C4	Requirements of IS:269: 2015
SiO_2 (%)	19.41	20.25	19.9	21.71	-
Al_2O_3 (%)	4.68	5.52	5.13	4.66	-
Fe_2O_3 (%)	4.27	5.35	5.29	4.85	-
CaO (%)	62.00	61.34	62.45	59.79	-
MgO (%)	4.12	1.03	1.64	1.03	Max 6
LOI (%)	3.22	2.84	1.93	4.44	Max 4
SO_3 (%)	2.46	2.65	2.65	2.71	Max 3.5
IR (%)	0.58	1.75	0.92	4.32	Max 5

The cement samples are analysed in the XRD to find out its phase compositions. The analysis results are presented in table 4.

Table 4: Phase quantification in XRD for the OPC 53 samples

OPC Sample	Alite_M3 (%)	Periclase (%)	Free Lime (%)	Portlandite (%)	Alum_cubic (%)	Alumortho(%)
C1	24.0	4.12	0.35	0.34	2.64	0.3
C2	15.58	0.14	0.32	0.09	0.46	0.19
C3	17.7	0.16	0.38	0.16	0.4	0.3
C4	11.9	0.44	0.21	0.07	0.71	1.36

Water

Water Portable water with PH value of 7.0 confirming to IS 456-2000 was used for making mortar cubes and curing the specimen as well.

Fine aggregates (FA)

Locally available M sand confirming to grading Zone II of IS: 383-1970 was used in this experiential work. The specific gravity, water absorption and fineness modulus of this fine aggregate is 2.65, 0.8% and 2.9 respectively.

Course Aggregate (CA)

Coarse aggregate Locally available crushed stones confirming to graded aggregate of nominal size 20mm as per IS: 383-1970 was used in this experimental work. Its physical properties, specific gravity and water absorption are 2.76 and 2.1% respectively.

4. ANALYSIS OF THE RESULTS

Results of the marsh cone test. The results of the Marsh Cone Test for the cement slurry containing different cements C1, C2, C3 and C4 with PCE SP's S1, S2 S3 and S4 are found out and tabulated below in Table 3. The same results are shown graphically to compare the test results for different PCE superplasticizers and cements in figure 2.

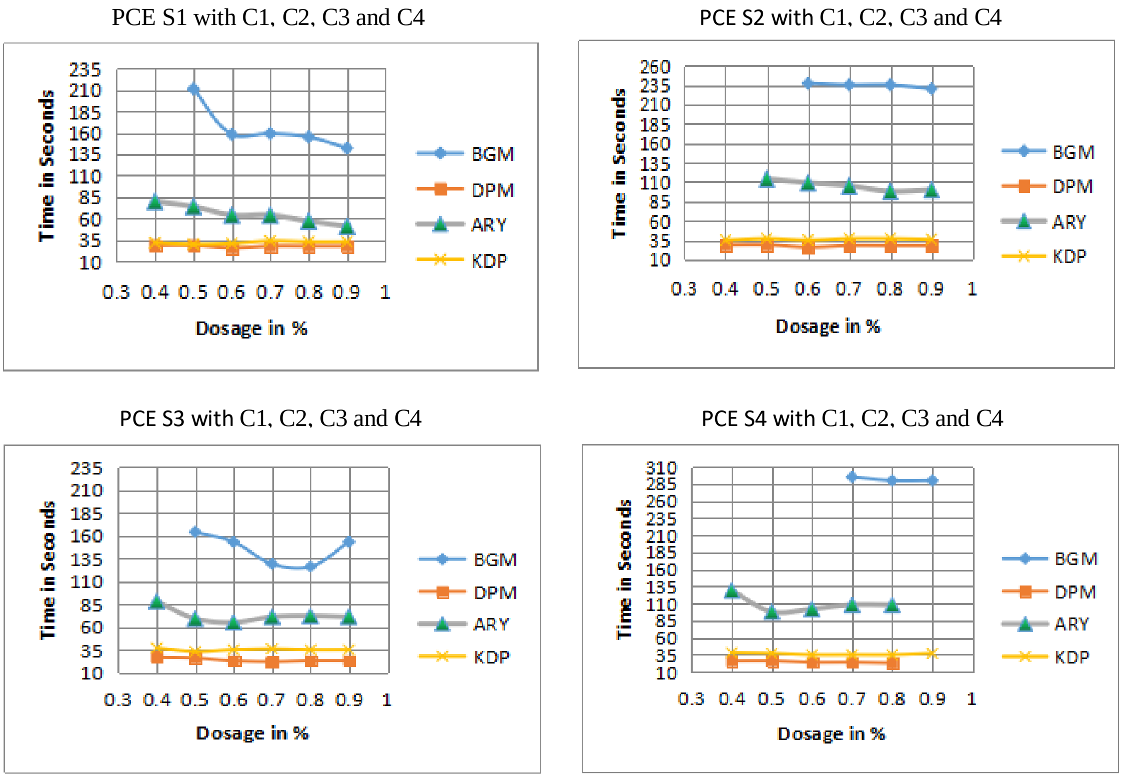

Fig. 2: Optimum dosage of PCE SP's S1,S2, S3 & S4 for cements C1, C2, C3 and C4.

The optimum dosage results for the PCE SP's are presented in table 4. The optimum dosage of PCE SP S1 is 0.6, 0.6, 0.4 and 04 with cement brands C1, C2, C3 and C4 respectively. The optimum dosage of PCE SP S2 is 0.6, 0.5, 0.4 and 0.4 with cement brands C1, C2, C3 and C4 respectively. The optimum dosage of PCE SP S3 is 0.7, 0.5, 0.4 and 0.4 respectively. The optimum dosage of PCE SP S4 is 0.7, 0.5, 0.4 and 0.4 respectively.

Table 4: Marsh cone test results

Cement type	Optimum dosage, S1%	Optimum dosage, S2%	Optimum dosage, S3%	Optimum dosage, S4%
C1	0.6	0.6	0.7	0.7
C2	0.6	0.5	0.5	0.5
C3	0.4	0.4	0.4	0.4
C4	0.4	0.4	0.4	0.4

The optimum dosages of the PCE SP's S1, S2, S3 and S4 is in the range of 0.4% to 0.7% for all the 4 cement brands. It is observed that the marsh cone time for all these dosages are in the same range except for the C1 cement. The marsh cone time for all the samples are given in table 5.

Table 5: Marsh cone time in seconds

Marsh cone test for C1 cement with PCE SP's				
Dosage in %	**S1, time in seconds**	**S2, time in seconds**	**S3, time in seconds**	**S4, time in seconds**
0.5	211		164	
0.6	158	237	153	
0.7	159	235	129	295
0.8	155	235	126	290
0.9	150	230	153	290
Marsh cone test for C2 cement with PCE SP's				
0.4	29	29	27	26
0.5	29	25	26	26
0.6	26	25	23	24
0.7	28	23	22	24
0.8	28	24	23	23
0.9	28	24	23	23
Marsh cone test for C3 cement with PCE SP's				
0.4	80		88	129
0.5	74	114	69	98
0.6	64	109	65	102
0.7	64	105	71	108
0.8	57	98	72	108
0.9	51	100	71	108
Marsh cone test for C4 cement with PCE SP's				
0.4	32	35	37	38
0.5	30	37	33	37
0.6	31	35	35	35
0.7	34	37	36	35
0.8	33	37	35	35

All the PCE SP's optimum dosage with C1 cement is 0.6 to 0.7. But the time taken for the paste to come out from the marsh cone (Marsh cone time) is in the range of 153 to 295 seconds. The same for the other three cement brands with the 4 PCE SP's is less than 100 seconds. This shows that the cement brand C1 is not compatible with any of the 4 PCE SP's considered in the study. This results in poor workability in concrete and will be uneconomical if C1 cement is used with any of these PCE SP's. On physical testing and chemical analysis of the cement brands, it is observed that all the 4 brands confirm to the IS specifications. In chemical analysis, MgO of all the cements were observed and found that C1 cement has high percentage in the range of 4.12%. the same is 1.03, 1.03 and 1.64 for C2, C4 and C3 cement respectively. High MgO leads to nucleation of C_3S rather than growth. Due to this, the fine crystals will react with water instantly when water is added to cement.

The XRD analysis results from the table 3 shows that the periclase percentage is high in the case of C1 cement. This periclase readily reacts with water and forms $Mg(OH)_2$ this reduces the free water present in paste. This results in increased marsh cone time and reduced workability making the cement incompatible with the PCE SP.

Concrete properties

Concrete trials are carried out to understand the behaviour of these admixtures with cement in terms of strength and workability. For this, M50 concrete mix is designed as per IS 10262. Admixtures are used at their optimum dosages in these trials. The mix used for the concrete trials is, Cement - 430kg/m^3, Water - 151kg/m^3, M sand - kg/m^3, CA - 1130kg/m^3 and optimum dosage of admixtures.

The concrete workability and strength results are tabulated in table 6. From the test results, it may be observed that there is not considerable difference in strength though the admixtures are changed. But there is significant difference in workability in terms of initial slump and the slump at 30 and 60 minutes.

Table 6: Concrete workability and strength

Cement Name	Admixture Name	% Of Admixture	Slump in mm.			CS, MPa	
			0 Min	30 Min	60 Min	7D	28D
C1	S1	0.6	110	60	0	61.7	71.0
	S2	0.6	55	0	0	60.3	69.8
	S3	0.7	Collapse	130	20	59.0	71.0
	S4	0.7	Collapse	120	0	58.0	70.0
C2	S1	0.6	Collapse	Collapse	Collapse	63.0	72.0
	S2	0.5	Collapse	Collapse	120	58.0	69.0
	S3	0.5	Collapse	Collapse	Collapse	62.0	72.0
	S4	0.5	Collapse	Collapse	100	61.0	70.0
C3	S1	0.4	Collapse	Collapse	Collapse	63.4	75.2
	S2	0.4	Collapse	Collapse	145	60.4	72.7
	S3	0.4	Collapse	Collapse	Collapse	62.8	75.4
	S4	0.4	Collapse	Collapse	Collapse	61.0	73.0
C4	S1	0.4	Collapse	Collapse	Collapse	55.1	69.9
	S2	0.4	Collapse	0	0	51.1	72.7
	S3	0.4	Collapse	Collapse	Collapse	60.9	73.0
	S4	0.4	Collapse	Collapse	100	60.0	73.0

6. CONCLUSION

PCE SP's compatibility with four brands of cement is studied and saturation point is obtained using Marsh cone test. For the considered water cement ratio of 0.35, minimum admixture dosage was observed for superplasticiser S1 i.e. 0.4% for cement brands C3 and C4. The minimum admixture dosage for S2 PCE SP is 0.4% for cement brand C3 and C4. The same for S3 and S4 is 0.4% for cement brands C3 and C4.

It is observed that the optimum dosage of all PCE SP's for the cement brand C1 is very high. It is 0.6% for S1, S2 and 0.7% for S3,S4 respectively. This makes the brand C1 incompatible with all the four PCE SP's considered in the study. The same trend is observed in the concrete trials also.

This concludes that the brand C2 and C4 is compatible with all the 4 PCE SP's. Though the performance of the brand C3 is slightly poor compared to the C2 and C4 cements.

Concrete may be produced economically, if the PCE admixture is compatible with the cement at a lesser dosages. The MgO content in the cement also plays a major role in making the PCE SP's effective. As discussed earlier, high MgO leads to nucleation of C_3S rather than growth. Due to this, the fine crystals will react with water instantly when water is added to cement and make the PCE SP less effective.

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