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Effect of PCE superplasticizers on rheological and strength properties of high strength self-consolidating concrete

S.D. Bauchkar^{1a} and H.S. Chore^{*2}

¹Admixture Systems, BASF India Ltd., C-405 and 407, MIDC, Thane Belpaur Road, Turbhe, New Mumbai, 400 613, India
²Department of Civil Engineering, Dr. B.R. Ambedkar National Institute of Technology Jalandhar, G.T. Bye Pass Road, Jalandhar, 144011, India

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Abstract. A variety of polycarboxylate ether (PCE)-based superplasticizers are commercially available. Their influence on the rheological retention and slump loss in respect of concrete differ considerably. Fluidity and slump loss are the cardinal features responsible for the quality of concrete. These are related to the dispersion of cement particles and the hydration process which are greatly influenced by type of polycarboxylate ether (PCE)-based superplasticizers. On the backdrop of relatively less studies in the context of rheological retention of high strength self-consolidating concrete (HS-SCC), the experimental investigations were carried out aiming at quantifying the effect of the six different PCE polymers (PCE 1-6) on the rheological retention of HS-SCC mixes containing two types of Ordinary Portland Cements (OPC) and unwashed crushed sand as the fine aggregate. The tests that were carried out included T_{500} , V-Funnel, yield stress and viscosity retention tests. The supplementary cementitious materials such as fly ash (FA) and micro-silica (MS) were also used in ternary blend keeping the mix paste volume and flow of concrete constant. Low water to binder ratio was used. The results reveal that not only the PCEs of different polymer groups behave differently, but even the PCEs of same polymer groups also behave differently. The study also indicates that the HS-SCC mixes containing PCE 6 and PCE 5 performed better as compared to the mixes containing PCE 1, PCE 2, PCE 3 and PCE 4 in respect of all the rheological tests. The PCE 6 is a new class of chemical admixtures known as Polyaryl Ether (PAE) developed by BASF to provide better rheological properties in even in HS-SCC mixes at low water to binder mix. In the present study, the PCE 6, is found to help not only in reduction in the plastic viscosity and yield stress, but also provide good rheological retention over the period of 180 minutes. Further, the early compressive strength properties (one day compressive strength) highly depend on the type of PCE polymer. The side chain length of PCE polymer and the fineness of the cement considerably affect the early strength gain.

Keywords: workability; rheology; rheological retention; polycarboxylate ether; polyaryl ether

1. Introduction

In the metropolitan cities, the concrete batching plants are forced to be located away from the

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^{*}Corresponding author, Associate Professor, E-mail: hschore@rediffmail.com, chorehs@gmail.com aManager, E-mail: sunil_bauchkar@rediffmail.com, sunil.bauckar@basf.com

site due to lack of space at construction site and environmental constraints. Further, owing to increase in traffic and changing weather conditions, the concrete producers not only find it difficult to maintain the workability of concrete for longer time but also to deliver the concrete that can satisfy the performance requirements. The average lead time for a commercial ready-mixed concrete to be delivered to any site is between 120-180 minutes. One of the solutions to counter this long retention workability drop is the selection of suitable concrete admixtures which can give good dispersion and desired retention.

Over the years, the change of admixture technology from lignosulphonates to napthalene sulphonates to poly-carboxylates stands testimony to this transformation. Traditional napthalene-based super-plasticizers can be inexpensive amongst available admixtures; but they are not capable of addressing to the issues of special high strength and high-performance concrete, where long workability is required without affecting the strength. The Polycarboxylate Ethers (PCE) based admixtures, called hyper-plasticisers, have paved the way for newer concrete technology and the demand for such admixtures has ever been increasing. The PCEs, in general, impart better control over the rheology of the concrete and that is one of the reasons as to why such admixtures are always used for producing self-compacting, smart dynamic concrete, etc.

High-range water-reducing superplasticizers use a new customized PCE polymer technology providing advanced rheological properties to the concrete. Also, the insight gained on the contribution of supplementary cementing materials such as fly ash, slag, silica fume, etc., have increased the scope of admixtures in the concrete today. Modernization and mechanization have also added new dimensions to the growth of this industry and helped in launching of various new placing technologies like pumping of concrete. The shortage of skilled labourers has made self-compacting and smart dynamic concrete the preferred choice for most of the engineers and contractors. The poly-carboxylates (PCE) is the 'new generation superplasticizer' that provides the workability enhancement at low water to cement ratios, resulting in the production of durable and flowable concrete, is the back-bone of these technologies. Polymers of this family can be produced with almost infinite variations in their chemical structure, which allows the fulfillment of specific properties.

The use of High-Strength, High-Performance Concrete, Self-Compacting Concrete and High-Strength Self-Compacting Concrete (HSC/HPC/SCC/HS-SCC) has proved to be a boon for the high-rise building construction. On the other hand, the concrete industry encounters the challenges with viscosity at the concrete batching plant and also at the point of placing at construction sites. Special high grade and high-performance concrete warrants long mixing time and high power consumption. Extra addition of water is required to overcome high viscosity. Sticky concrete is difficult to pump, finish and vibrate. Further, it leads to smooth surface deficiencies of hardened concrete and affects the durability. Proper mix design and selection of supplementary cementitious material combination in conjunction with tailor- made chemical admixtures for obtaining low viscous and stable concrete mix is the prime area of research nowadays.

2. Background

Workability is one of the most important properties of cementitious materials because it directly influences the construction process of fresh cementitious materials and even mechanical properties and durability of hardened ones. The workability is an assembly of several properties such as fluidity, plasticity, stability and cohesion (Rechtie 1962). Many researchers (Murata 1984,

Banfill 1994, Struble and Sun 1994, Hu and de Larrard 1996, Schwartzentruber and Catherine 2000, Flatt and Houst 2001) have studied workability by evaluating the rheological properties of fresh cementitious material paste. Few more researchers (Banfill 2003, Zhang *et al.* 2006, Li and Kong 2009) found that due to various chemical and mineral admixtures used in high strength concrete and high-performance concrete, the rheology of traditional cementitious materials may no longer be suitable for the modern cementitious materials.

Banfill (1981) reported that the admixtures help to reduce both the yield value and plastic viscosity. The reduction in the yield stress of cement and concrete to very low values is attributed by Tattersall and Banfill (1983) to the dispersion of flocculated cement particles. Few more researchers (Wallvik 1990, Hu and de Larrard 1996, Ferraris *et al.* 2001, Banfil 2006) pointed out impact of admixture on developments in materials and processes, such as flowing and self-compacting concrete with the aid of the dispersing admixtures that have become common since the 1970s and which extended the range of concrete rheology and need to measure the same. It has clearly diverted focus to other test methods based upon rheological principles.

Superplasticizers are, nowadays, essential component of concrete. These admixtures reduce the amount of water needed in the preparation of concrete, enhancing its mechanical strength and durability. Ohta reported the use of the latest generation polycarboxylate ether (PCE) based superplasticizers, which induces the highest water reduction capability in concrete mix (up to 35-40%), have become imperative in high performance, high strength and self-compacting concretes (Ohta et al. 2000). Further, Puertas et al. (2005) explained that PCE admixtures are characterized by a 'comb' type structure in which the backbone is a linear hydro- carbonate chain and carboxylate and ether groups form the lateral chains. According to some of the researchers (Jolicoeur and Simard 1998, Kreppelt et al. 2002, Heikal et al. 2005) the adsorption of superplasticizers onto cement particles and subsequently, its dispersion due to the generation of electrosteric repulsion improves the rheological properties of cement paste and at the same time often visibly affects the cement hydration process, mostly by retarding cement hydration. Few researchers (Flatt and Houst 2001, Alonso et al. 2007, Winnefeld et al. 2007) confirmed that the flowability induced by PCE admixtures depends directly on the structure of these admixtures (length of the main and lateral chains, density of lateral ether groups, molecular weight and molecular weight distribution) while some of the researchers (Lewis et al. 2000, Kauppi et al. 2003) pointed out that the progress of the hydration reactions causes stiffening (slump loss) and this can be a serious practical problem. Felekoğlu et al. (2008), further, reported that molecular architectures of the PCE admixtures can be modified to adjust their effects on concrete and adapt incompatibility issues. Zhang et al. (2016) pointed out that the effects of different combinations of superplasticizer and retarder on the fluidity were quite different and the combinations of the retarders and PCE had better influence on the fluidity and flow loss than naphtha based superplasticizer.

Moreover, the effect of chemical admixtures on the rheology of self-compacting concrete was studied by some of the researchers (Ramchandran 1992, Neubauer *et al.* 1998, Jayashree and Gettu 2008, Aydin *et al.* 2009, Kwan and Ng 2009, Plank *et al.* 2009). They found that the superplasticizers could significantly improve the workability of concrete, reduce the water demand and enhance the strength of cementitious construction materials. Due to dispersion effect, the fluidity of the paste was found to increase thereby reducing the yield stress and plastic viscosity.

Till date, numerous studies have investigated the relation between molecular structure of PC and its dispersing properties in cement systems. These studies confirmed that the dispersive effect of superplasticizers and specifically that of polycarboxyalate-based admixtures depends on cement

properties such as C_3A content, type and contents of calcium sulphate used as a setting regulator, clinker alkali content, specific surface and particle size distribution; and type of mineral addition (Mollah *et al.* 2000, Yamada *et al.* 2000, Flatt and Houst 2001, Yoshioka *et al.* 2002, Chandra and Bjornstrom 2002, Puertas *et al.* 2005, Winnefeld *et al.* 2006, 2007, Feys *et al.* 2016). Of this, Yamada *et al.* (2000) reported that the comb-type copolymers with longer side chains show higher dispersing power while Winnefeld *et al.* (2006, 2007) reported that the type of polycarboxylate ether, molecular structure and the chemical composition have a significant influence on the rheology of concrete.

Nawa (1999) found that the comb-type copolymers with short side chains exhibit better effects than those with long side chains. The studies carried out by Ran *et al.* (2009) suggested that 'adsorption of comb-type copolymers controls dispersion of cement systems and the dispersion effect increases as the adsorption amount of comb-type copolymers increases'. Hence, the relationship between molecular structure of PCE and its dispersing property still has some contradictory conclusions and warrants further investigations in this context.

Recently, Bauchkar and Chore (2014) reported the rheological properties of self-consolidating concrete (SCC) with various mineral admixtures. Bauchkar and Chore (2017), Bauchkar *et al.* (2017) presented the experimental investigations on the evaluation of rheological properties of smart dynamic concrete (SDC) containing different supplementary cementitious Materials.

3. Relevance and significance of the present work

Many studies have explored the effect of chemical admixtures on the rheological behavior of concrete. These include studies with PCE type admixtures, too. However, the study in the context of rheology retention over time due to effect of different types of PCE with respect to High strength self-consolidating concrete (HS-SCC) has been seldom reported. An increase in the use of high strength concrete for high rise constructions poses various challenges to concrete experts working in the field. Some of the challenges comprise that of flow retention and rheological retention. Understanding the effect of different PCE on the rheological retention of HS- SCC would be useful to the engineers to select suitable PCE based admixture for high-rise structures. Pursuant to this, the study on the rheological retention properties of High strength self-consolidating concrete (HS-SCC) in respect of six different PCE polymers is presented here.

4. Materials and proportions of mixes

4.1 Supplementary cementitious materials

An Ordinary Portland Cement (like ASTM - Type-I, 2004) conforming to the requirements of OPC 53 of two different brands (OPC 1 and OPC 2) was used. Two types of supplementary cementitious materials, namely- fly ash (PFA) and micro silica (MS), were also used in ternary blends in HS-SCC mix. The physical and chemical properties of OPC 1, OPC 2, PFA, and MS as are summarized in Table 1. The supplementary cementitious materials are shown in Fig. 1.

Similarly, the details of the particle size distribution analysis of two different OPC, PFA and MS are summarized in Table 2 and is shown in Fig. 2. The systematic laboratory investigations were carried out at the Research and Development centre of Counto Microfine Products Pvt Ltd., Goa (India) to arrive upon these properties.

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	Material	Unit	PFA	GGBS	MS	UFS	OPC 1	OPC 2
	Blaine fineness	(m²/kg)	345	390	-	-	335	325
	BET Surface Area	(m²/kg)	-	-	22000	4968	-	-
	Compressive strength as % of cement	(%)	84.2	92	156	-	100	100
	Lime reactivity	MPa.	5.6	-	8.5	-	-	-
	Autoclave expansion	(%)	0.06	-	NA	13.5	0.059	0.09
	Sp. gravity	(%)	2.3	2.86	2.2	NA	3.14	3.15
	Loss on ignition (LOI)	(%)	1.2	0.37	2.6	2.9	2.81	1.18
	Silica (SiO ₂)	(%)	60.72	33.72	92.3	1.9	20.68	20.30
	Iron oxide (Fe_2O_3)	(%)	5.32	0.64	0.06	26.8	4.76	5.31
	Alumina (Al ₂ O ₃)	(%)	27.5	18.22	0.62	2	5.54	4.18
	$SiO_2 + Al_2O_3 + Fe_2O_3$	(%)	93.54	52.58	93.88	21.9	30.98	29.81
	Calcium oxide (CaO)	(%)	1.42	34.51	0.3	50.7	61.39	63.22
	Magnesium oxide (MgO)	(%)	0.48	11.22	0.3	31.5	1.07	1.22
	Total sulphur (SO ₃)	(%)	0.21	0.22	0.05	1	2.5	2.64
	Alkalis $(Na_2O + K_2O)$	(%)	1.71	0.53	0.6	-	0.38	0.062
	Chloride	(%)	0.36	0.001	0.001	-	0.055	0.022
	Retained on 45 microns	(%)	15	1.55	0.2	-	10.66	17

Table 1 Physical and chemical compositions of the cementitious materials used in the study



(a) OPC cement

(b) Fly ash Fig. 1 Supplementary cementitious materials used in the study

Table 2 Particle size distribution (PSD) of the cementitious materials used in the	study
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	PSD in μ m								
Details	D10	D50	D90	D95	D100				
OPC 1	4.07	21.38	61.96	79.06	181.97				
OPC 2	3.97	22.38	63.96	81.06	191.90				
Fly ash	6.78	32.65	83.42	103.15	181.97				
Micro silica	3.54	24.90	50.46	58.07	91.20				

4.2 Aggregates

The crushed basalt aggregates of size 4.7 mm, 10 mm and 20 mm were used as the coarse aggregates (CA) and fine aggregates. The unwashed crushed sand and the aggregates of 10 mm



Fig. 2 Particle size distribution (PSD) curve of the cementitious materials used in the study



Fig. 3 Unwashed crushed sand, 20 mm and 10 mm aggregates

Table 3 Physical properties of coarse and fine aggregates used in the study

	IS Sieve Size (mm)	20	10	4.75	2.36	1.18	0.6	0.3	0.15	Silt content (%)	Fineness Modulus	Specific Gravity	Water Absorption
Crushed Sand	%	100	100	93.9	65.2	43	28.9	17.6	10.4	12.50%	3.41	2.72	3%
20 mm	Passing	97.4	2.9	1.1	1.1	1.1	1.1	1.1	0	0.50%	6.94	2.82	1.50%
10 mm		100	82.6	3.4	2.8	2.8	2.8	2.8	0	0.50%	5.6	2.8	1.80%

and 20 mm size used in the present investigation is shown in Fig. 3. The physical properties (dry sieve analysis) of the aggregates obtained through systematic laboratory investigations carried out at the Research and Development Centre of BASF India Ltd., Navi Mumbai (India) are given in Table 3.

4.3 Admixtures

A commercially available PolyCarboxilate Ether based superplasticisers PCE1, PCE 2, PCE 3, PCE 4, PCE5 and PCE 6 were used in this study. The physical properties of PCE1 to PCE 6 as

Product	PCE 1	PCE 2	PCE 3	PCE4	PCE5	PCE 6
Relative Density @ 25° C	1.01	1.02	1.01	1.01	1.01	1.01
Dry Material content (%)	25	25	25	25	25	25
pH	> 6	> 6	> 6	> 6	> 6	> 6
Chloride-ion content	< 0.2%	< 0.2%	< 0.2%	< 0.2%	< 0.2%	< 0.2%
					<u></u>	
PCE 1 (HRWR)		PCE3 (HRW	/R - SR)	PCE 5	6 (MRWR -S	R-VR)
					eterical reputsion	
PCE 2 (HRWR)		PCE 4 (HRV	WR-SR)	PCE 6	6 (MRWR -S	R-VR)
Fig. 4 Poly	carboxylat	e ether (PCE)	-based superp	lasticizers us	ed	

Table 4 Physical properties of polycarboxylate ether (PCE)-based superplasticizers

presented in Table 4 were evaluated using state- of- the- art instrumentation available at the Research and Development centre of BASF India Ltd., Navi Mumbai (India). The PCE structures of different polymers used in study are shown in Fig. 4.

PCE 6, a new class of chemical admixtures, known as PolyAryl Ether (PAE) has been developed by BASF to provide improved rheological properties in concrete even in demanding mix designs. Although it exhibits similar chemical side chains as that of other PCEs, the presence of new chemical backbone units in combination with a very high density of negative charges is believed to increase the affinity to the cement surface whereas the presence of side chains imparts a steric effect to the electro-static component of the inter-particle repulsion forces.

4.4 Details of the High strength self-compacting concrete mix

In the present work, twelve different mixes of high strengths self-compacting concrete (HS-SCC) were designed. The concrete mix proportions were calculated in accordance with the norms laid in IS-10262 (2009) and these mix proportions are shown in Table 5. It was also ensured that all the HS-SCCs mixes satisfied the regulations given by *EFNARC* (2005) guidelines.

5. Experimental procedure

The HS-SCC mixtures were mixed in the batches of 40 litters using a Pan mixer. Mixing efficiency, type of mixer, sequence of mixing, ambient temperature, etc. are some of the factors affecting the rheology of HS-SCC during its production. Therefore, these factors were not changed throughout the study. The mixing materials were kept at temperature of 30 ± 2 °C (approximately) before mixing. At the end of mixing, the mixtures had approximately constant temperatures of

for meology rea	study									
Mix Details	Cement (OPC 1) (Kg)	Fly Ash (Kg.)	MS (Kg.)	w/b	Free water (Kg.)	20 mm (Kg.)	10 mm (Kg.)	Crushed sand (Kg.)	Paste vol. (%)	PCE Type
HSSCCOP1A1	450	130	50	0.25	158	350	520	845	41	HRWR
HSSCCOP1A2	450	130	50	0.25	158	350	520	845	41	HRWR
HSSCCOP1A3	450	130	50	0.25	158	350	520	845	41	HRWR - SR
HSSCCOP1A4	450	130	50	0.25	158	350	520	845	41	HRWR - SR
HSSCCOP1A5	450	130	50	0.25	158	350	520	845	41	MRWR - SR- VR
HSSCCOP1A6	450	130	50	0.25	158	350	520	845	41	MRWR - SR- VR
Min Dataila	Compart (ODC 2)	Else A sh	MC	/l-	Free	20	10	Crushed	Paste	PCE

w/b

0.25

0.25

0.25

0.25

0.25

0.25

water

158

158

158

158

158

158

20 mm 10 mm

520

520

520

520

520

520

350

350

350

350

350

350

sand

845

845

845

845

845

845

vol.

41

41

41

41

41

41

Type

HRWR

HRWR HRWR -

SR HRWR -

SR MRWR -

SR- VR MRWR -

SR- VR

MS

50

50

50

50

50

50

130

130

130

130

130

130

Table 5 Mix Proportions (kg/m³) of concrete type HS-SCC with unwashed crushed sand as fine aggregate for rheology retention study

 33 ± 2 °C. The mixing procedure for concrete mixtures consisted of homogenizing the fine and coarse aggregate for 1 minute and introducing 25% of the mixing water. Following a rest period of 1 minute to allow the saturation of the aggregates, binder and 50% of water were added. After 2 minutes of mixing, the HRWR diluted with the remaining water was introduced gradually over 2 minutes while the mixer was turned on. Following the rest period of 2 minutes further, the concrete was mixed for 3 additional minutes.

The concrete was discharged directly from the pan mixer into the container of ICAR Rheometer. Simultaneously, the sample was also withdrawn for other engineering fresh properties such as flow, V-funnel etc. The testing for Rheology and the workability using the traditional method was repeated for every 60 minutes till retention period of 180 minutes. Two types of tests were performed using ICAR rheometer. The first one was a 'stress growth test' in which the vane was rotated at a constant slow speed of 0.025 rev/sec. The initial increase of torque was measured as a function of time. The maximum torque measured during the test was used to calculate the static yield stress. The other type of test was a 'flow- curve test' to determine the dynamic yield stress and the plastic viscosity. The set-up of the ICAR rheometer, flow table, V-funnel used for the testing, is shown in Fig. 5 and Fig. 6.

The slump cone was lifted and three measurements were taken- one corresponding to the time for the concrete to spread over a horizontal diameter of 500 mm (T_{500}) measured in seconds;

Mix Details

HSSCCOP2A1

HSSCCOP2A2

HSSCCOP2A3

HSSCCOP2A4

HSSCCOP2A5

HSSCCOP2A6

Cement (OPC 2) Fly Ash

450

450

450

450

450



Fig. 5 ICAR Rheometer set up and testing





(a) V-Funnel(b) Slump flo(c) Fig. 6 V-funnel, flow table, ICAR Rheometer used for the testing

second one, the final horizontal spread diameter which is nothing but flow measured in mm; and third one, the visual stability index (VSI). The VSI ratings, which were determined based on the definition as given by Wallevik (2017) were made on a scale of 0 to 3, with 0 exhibiting excellent stability and 3, poor stability. Other than slump- flow test, all the mixes satisfied VSI 1 requirements. *V*-funnel test (Fig. 6(a)) was performed as per *EFNARC* (2005) standards.

The compressive strength of the concrete was determined in accordance with Indian Standards. In order to determine the compressive strength, 180 cubes of size $150 \times 150 \times 150$ mm for twelve mixes were cast. The cube specimens were tested for compressive strength at the age of 1 day, 3 days, 7 days along with 28 days and 56 days.

6. Results and discussion

The experimental investigations were carried out on High Strength- Self Compacting concrete (HS-SCC) for studying its rheological performance in respect of high-range water reducer (HRWR), high range water reducer- cum- slump retainer (HRWR-SR); and Mid range water reducer - cum- slump retainer and viscosity reducer (MRWR-SR-VR) as an admixture.

For the purpose of evaluation, the conventional (traditional) methods as used in the context of SCC and ICAR Rheometer were resorted to. This experimental study was carried out using two

different brand of Ordinary Portland Cements (OPC), two types of supplementary cementitious materials such as Fly Ash and Micro Silica and six different types of PCE polymers (as shown in Fig. 4) at constant mix paste volume and the flow parameters of concrete. The objective of the study was to study the effect of PCE type on rheology retention of HS-SCC. To understand the effect of different types of PCE polymers on the rheological retention of the HS-SCC, the comparative analyses of the obtained values of the yield stresses and viscosity retention were carried at the intervals of 60 minutes over a total duration of 180 minutes. The results are reported in Tables 6 and 7.

Table 6 Laboratory trial flow, T_{500} and V-funnel retention properties of HS-SCC with different types of PCE polymers

Description	Flow (mm)					$T_{500} (sec)$				V funnel (Sec)			
Time in minutes	(%)	05	60	120	180	05	60	120	180	05	60	120	180
HSSCCOP1A1	0.67%	650	580	500	180	8.8	11.3	15.6		17.5	28.1	58.2	
HSSCCOP1A2	0.72%	650	580	510	200	6.9	9.1	12.7		11.9	18.9	36.5	
HSSCCOP1A3	0.80%	680	610	560	350	6.7	8.7	12.2		12.3	19.2	28.1	
HSSCCOP1A4	1.00%	660	640	560	510	5.4	7.6	9.9	14.1	11.7	16.7	24.3	43.2
HSSCCOP1A5	1.40%	650	610	590	520	5	6.7	7.1	10.3	10.9	16.2	23.1	39.8
HSSCCOP1A6	1.80%	650	630	590	530	4.3	4.9	5.9	8.8	8.1	12.8	18.9	30.2
HSSCCOP2A1	0.56%	660	620	580	500	6	8.8	11.3	15.6	13	17.5	28.1	58.2
HSSCCOP2A2	0.58%	650	630	580	530	5.5	6.9	9.1	12.7	8	11.9	18.9	36.5
HSSCCOP2A3	0.66%	700	700	680	660	4.1	5.7	6.7	8.9	10.9	13.3	17.4	23.2
HSSCCOP2A4	0.83%	690	670	650	630	3.3	4.9	6.6	8.5	8.3	12.2	15.3	20.3
HSSCCOP2A5	1.19%	700	690	680	670	3.1	4.4	6.4	7.9	8.2	10.8	13.7	17.1
HSSCCOP2A6	1.57%	690	680	680	670	2.2	3.2	3.5	3.9	7.7	9.3	11.1	12.6

Table 7 Laboratory trial rheology retention properties of HS-SCC with different types of PCE polymers

Description	γ	ield Str	ess, τ, (I	Pa.)	Viscosity, μ , (Pa. s)				Air Content (%)			
Time in minutes	5	60	120	180	5	60	120	180	5	60	120	180
HSSCCOP1A1	172.7	185.1	329.1	658.2	107.4	109.1	198.2	396.4	1.2	0.9	0.8	-
HSSCCOP1A2	90.2	133.3	233.8	420.84	61.6	77.4	147.1	264.78	1.4	1.1	0.9	-
HSSCCOP1A3	80.6	126.8	189.7	303.52	61.6	82.1	128.3	205.28	1.5	1.1	1	-
HSSCCOP1A4	82.1	111.2	176.7	247.38	49.3	66.7	106.1	148.54	1.6	1.3	0.9	0.7
HSSCCOP1A5	72.7	100.5	165.8	182.38	43.6	60.3	99.5	109.45	1.5	1.3	1	0.8
HSSCCOP1A6	69.1	87.6	119.3	125.265	41.5	52.5	71.6	75.18	1.4	1.2	1	0.9
HSSCCOP2A1	169	172.7	175.1	203.1	98.4	107.4	109.1	150	1.4	12	1	0.8
HSSCCOP2A2	85.2	90.2	113.3	145	51.6	61.6	77.4	89	1.6	1.3	1.1	0.9
HSSCCOP2A3	62.1	72.5	92.3	98.3	37.3	45.7	60.1	71.9	1.4	1.2	1.1	1
HSSCCOP2A4	60.1	66.3	80.8	91.2	36.1	41.8	52.5	62.4	1.5	1.3	1	0.8
HSSCCOP2A5	58.8	62.8	74.9	78.9	35.2	39.6	48.7	49.7	1.2	0.9	0.8	0.7
HSSCCOP2A6	57.9	59.8	68.1	70.2	34.7	37.7	44.3	44.8	1.6	1.3	0.9	0.7

	71	<u> </u>
Mix ID	Admixture (%)	Initial flow (mm)
HSSCCOP1A1	0.67%	650
HSSCCOP1A2	0.72%	650
HSSCCOP1A3	0.80%	680
HSSCCOP1A4	1.00%	660
HSSCCOP1A5	1.40%	650
HSSCCOP1A6	1.80%	650
HSSCCOP2A1	0.56%	660
HSSCCOP2A2	0.58%	650
HSSCCOP2A3	0.66%	700
HSSCCOP2A4	0.83%	690
HSSCCOP2A5	1.19%	700
HSSCCOP2A6	1.57%	690
2.5%		
2.5%	OPC 1	
Q ^{2.0%}	v = 0.0023x + 0.002	8 🛹
m 1.5% -	$R^2 = 0.8876$	
ల్లి 1.0% -	0.000	
80 0 5% -	y = 0.002	0 8748

Table 8 Effect of PCE and OPC types on admixture dosages

Fig. 7 Effect of PCE and OPC types on admixture dosages

HSSCCOP2A3

HSSCCOP2A4

HSSCCOP2A6

HSSCCOP2A5

The effect of the types of PCEs and OPCs on various parameters of workability, rheological properties and rheology retention is discussed in the sub-sequent subsections.

6.1 Effect on admixture dosages for similar workability

[SSCCOP2A1

HSSCCOP2A2

Admixture do

0.0%

The results of slump flow test performed on twelve mixes are reported in Table 8 and indicated graphically in Fig. 7. Slump flow test helps to assess the flow of concrete in the absence of obstruction. It is one of the most commonly used test method which gives a good assessment of the filling ability and mix segregation visual indication. The significance of this test is that higher the flow value, greater will be the ability to fill the formwork under its own weight. A flow value of at least 650 mm is required for SCC and reported results are well above the required limit for SCC.

In all the twelve HS-SCC mixes, the effect of segregation was not observed. The coarse aggregate, mortar and the paste were observed to be uniformly distributed till the periphery of the concrete flow. Fig. 7 indicates that both OPC 1 and OPC 2 have linear variation of admixture dosage which is primarily dependent on side chain length of PCE polymers. It is also observed that



Fig. 8 Amount of the superplasticizer dosage to attain 675+/-25 mm initial slumpflow

the slope of OPC 1 mixes is on higher side as compared to that of in OPC 2 mixes. This indicates that the mix with OPC 1 is more sensitive to PCE polymer performance than that in mix with OPC 2. The regression analysis, further, confirmed the highest dosage variability and consequently, the highest dependency upon physical and chemical properties of cement. In Presented experimental study, the origin of the performance variability observed with two different cements. In this context, Plank *et al.* (2010) explained that the adsorption of the superplasticizers onto the cement grains is driven by both, enthalpic and entropic contributions. Under such circumstances, it may happen that minor variations occur in cement chemistry or sulfate solubility, thereby, resulting in the modifications of the solution ionic concentration, which in turn, affects the performance of PCE superplasticizers. The slump flow value corresponding to various admixture dosage in respect of different mixes is indicated in Fig. 8.

From Fig. 8, it is seen that the reduction in side chain length decrease the water reduction capacity of PCE and increase dosages for similar flow. The percentage of admixture used for PCE 5 and 6 superplasticizer is higher than that for PCE 1 and 2, thereby, confirming that PCE 5 and 6 superplasticizers have significantly lower efficiency than PCE 1 and 2 in terms of water reduction. The PCE 3 and 4 admixtures shows an intermediate dosage between the six different PCE's used. This means that the admixtures PCE 1 and 2 have a higher water reduction capability over PCE 3 and 4 while PCE 5 and 6 have the lowest water reduction capability amongst all the 6 PCEs tested. Further, PCE 1 and 2 are more sensitive in terms of the dosage or aggregate moisture variation. This indicates that even a slight variation in dosages or aggregate moisture will affect the fresh concrete properties significantly in case of PCE 1 and 2. The dosage variation could also be due to dispenser malfunction or change of OPC used. At the same time, it is observed that the PCE 5 and 6 are immune to any slight variation in dosage and do not affect the fresh concrete properties. This behaviour is also most commonly known as the robustness of the PCEs.

Hence, it can be interpreted that the PCE 5 and PCE 6 are more robust; and suitable to achieve designed concrete properties during the practical applications at site.

6.2 Effect on workability retention

Workability of concrete is also one of the critical criteria to ensure the ease of placement of designed mix at site. Hence, the mix is designed keeping in view, the retention time required considering the distance between the batching plant and site and also, the lead required for





Fig. 9 Effect of PCE Types on OPC 1 cement mix workability retention

Fig. 10 Effect of PCE Types on OPC 2 cement mix workability retention

pumping. When concrete is to be designed for a high strength with self-compacting properties, it is imperative to consider the flowability required at a low water binder ratio for the duration of the defined retention period. Traditional admixtures exhibit limitations in terms of the water reduction capability for higher cementitious contents and lower water binder ratio mixes. This makes PCE based admixtures, a preferred choice, to achieve designed properties for high strength self-compacting concrete mixes.

For HS-SCC mixes, the main types of admixtures that must be used are water reducing agents and superplasticizer. When water reducing agents are used, the total quantity of free water used will be kept under control as any increase in free water affects the strength of the concrete. In practical applications, the industry preferred retention period is usually 180 minutes. Hence, PCE polymers with slump retention properties are preferred to be used.

The workability retention over 180 minutes of different types of PCE mixed HS-SCC is indicated in Figs. 9-10. From the various parameters obtained in view of the flow retention of fresh HS-SCC concrete, the initial starting slump flow is found to be between 650 and 700 mm. This indicates the good deformability of the fresh concrete meeting SF 1 criterion for SCC. Though the paste volume of HS-SCC mixes is constant, the physical and chemical properties of cement influence the workability and the workability retention of concrete. The OPC 1 has faster workability drop over OPC 2 over period of 180 minutes. It is attributed to the higher fineness of OPC 1 cement which causes rapid hydration reaction and subsequently, faster workability loss.

The slump flow reduction over a period of retention time is observed to be significantly higher for PCE 1 and PCE 2 whereas for PCE 5 and 6, the decrease in flow decrease is observed to be nominal. Further, the variation in slump flow for PCE 3 and PCE 4 is not that drastic as compared to that in PCE 1 and PCE 2. The flow retention behaviour of HS-SCC mix is found to change with different types of PCEs. The concrete that loses its workability faster over the retention period is arranged as HRWR (PCE 1, PCE 2) followed by HRWR-SR (PCE 3 and PCE 4), which is then followed by MR-WR-SR-VR (PCE 5 and PCE 6). The retention behaviour of polymer is highly related to the delivered amount of PCE in mix. The PCE 5- 6 is found to require higher dosage as compared to PCE 1-4. From the test results, it is also observed that the flow retention of PCE 6 is superior amongst all the tested PCEs. This indicates that the PCE 6 delivers enough polymer for secondary reaction to support workability retention in case of MR-WR-SR-VR.



Fig. 11 Variation in slump flow at T_{500} mm for different type of PCE with OPC 1 cement





Fig. 12 Variation in slump flow at T_{500} mm for different type of PCE with OPC 2 cement



Fig. 13 Variation in *V*-funnel test value for different type of PCE with OPC 1 cement

Fig. 14 Variation in *V*-funnel test value for different type of PCE with OPC 2 cement

6.3 Effect on T₅₀₀ retention

The flow table test at T_{500} mm indicates the rate of flow within a defined flow distance of 500 mm. This test shows the filling ability of the SCC. The time taken to spread over a defined flow distance of 500 mm for the HS-SCC mixes with six types of PCE superplasticizers over period of 180 minutes is indicated graphically in Figs. 11-12.

The less time concrete mix takes to reach 500 mm on the base plate, more is the filling ability of the high strength self - compacting concrete (HS-SCC) and vice-versa. It is observed from Fig. 11-12, that the PCE 6 admixture gives good filling ability as compared to that in PCE 5, 4 and 3 admixtures. The admixtures PCE 1 and 2 are found to give less filling ability as compared to PCE 5, 4 and 3 admixtures over the period of 180 minutes. Further, the PCE 1 and 2 admixtures are found to have poor T_{500} retention limited up to 120 minutes as against the original target of 180 minutes. At the same time, the PCE 5 and 6 admixtures are found to exhibit good T_{500} retention up to 180 minutes.

6.4 Effect of V-funnel retention

V-funnel test is a test carried out to find out the filling ability of concrete. The lesser time the

concrete takes to empty the V-funnel as soon as the flip door is opened, better is the filling ability of concrete. The variation of V-funnel values as observed for different admixtures, i.e., PCEs, in respect of two different OPCs considered in the present investigation is indicated in Figs. 13-14.

It is seen from Figs. 13-14 that the PCE 6 significantly reduces V-funnel time values despite the influence of cementitious materials. When the behaviour of all six PCE superplasticizers in the same HS-SCC mix is examined, the admixtures PCE 1 and 2 are found to take more time than the rest of the PCEs, i.e., 3-6. It indicates that the long side chain polymer PCE drastically increases V-funnel retention whereas the PCE 5 and 6, having shorter side chains, gives lower V-funnel value over a period of 180 minutes.

The side chain length of the PCE polymer is found to have a significant influence on the *V*-funnel retention improvement. The shorter side chain length polymer is found to lower or reduce the *V*-funnel time and improve its retention. From Figs. 13-14, it is also found that the mixes prepared with OPC 1 show a higher increase in *V*-funnel values than that in mixes with OPC 2. This clearly indicates that the OPC 1 is more sensitive to the performance of PCE polymers than OPC 2.

6.5 Effect on air content over time

Certain types of the new generation PCE superplasticizers (SP) causes the rise of excessive air content in high strength self-compacting concrete (HS-SCC). The influence of different type of PCEs on air-contents in fresh HS-SCC is indicated in Fig. 15 and Fig. 16.

All the tested PCE polymers show the air entrapping tendency as evident from Figs. 15-16; the values being below 1.6% for all the PCEs. The PCE 6 polymer is found to show higher air content than that in other PCE polymers. It is also observed that the air content reduces over the period of 180 minutes.

The density measurements is conducted on fresh HS-SCC mixes and presented in Fig. 17 which confirms the entrapped air data.

The density values are found to be substantially similar for all the admixtures. The PCE 6 shows the lowest density value on fresh concrete as compared to that of other PCE products. The shorter side chain polymers generally show higher air content and lower density in fresh concrete with respect to long side chain PCE polymers.





Fig. 15 Entrapped air content value for different type of PCE with OPC 1 cement

Fig. 16 Entrapped air content value for different type of PCE with OPC 2 cement



Fig. 18 Effect of PCE Types on yield stress over retention period of 180 minutes.

6.6 Effect on rheology and rheological retention of HS-SCC

The effect of the types of PCEs used in the present investigation in conjunction with two different types of OPCs on yield stress as well as viscosity is also studied over a retention period of 180 minutes (Figs. 18-19). In these experimental investigations, all the HS-SCC mixes contain the same paste volume, SCM combinations and the w/b ratio. The mix was carefully designed to comply with EFNARC (2005) guidelines and avoid influence of other parameters on fresh and hardened properties. Hence, in this case, the HS-SCC rheology is highly dependent on the type of PCEs used. From Figs. 18-19, it is observed that the yield stress and plastic viscosity increases with the retention time.

The physical and chemical properties of the OPCs play an important role in rheological retention of the HS-SCC. But, in general, the site engineers expect that the same grade/ type of cement must also behave similarly. On the contrary, when cement is changed to OPC 2, the yield stress and viscosity is observed to reduce as compared to that in case of OPC 1. In simple words, the HS-SCC mixes are found to get less sticky with OPC 2 cement.

This is attributed to the fact that the fineness of different OPCs affects the static yield stress and thixotropy. The static yield stress greatly depends on the admixture dosages delivered in the mixes. This behavior can be because of the superplasticizer adsorption and surface coverage by the



Fig. 19 Effect of PCE Types on viscosity over retention period of 180 minutes

adsorbed polymer on the cement particles. The surface coverage affects the inter-particle forces as well as nucleation probability at the surface. An increase in the dosages of superplasticizer leads to higher surface coverage by the polymers. At higher surface coverage, the effective layer thickness increases and causes a reduction in the maximum attraction between the particles. Moreover, the number of available nucleation sites decreases and the bridging distance between the particles increases. Less force is needed to disperse the particles and hence, the static yield stress becomes lower.

Further, from Fig. 18 and Fig. 19, it is also observed that the type of PCE and side chain length along with the type of cement also influence the retention of plastic viscosity of HS-SCC. The variation in the viscosity of the HS-SCC mixes with PCE 1 and 2 type superplasticizers is on a higher side than that of the mixes with PCE 5 and 6. The results also indicate that the addition of PCE 6 results in the reduction in yield stress and viscosity retention of the mixes. With the addition of PCE 6 (as compared to that with PCE 1 results), the yield stress after 180 minutes is found to reduce from 658.2 Pa in respect of the mix with OPC 1 to 125.27 Pa, signifying an 80% yield stress reduction between PCE 1 and PCE 6 for the same retention period. Also, the viscosity is found to reduce from 396.4 Pa-s to 75.18 Pa-s, thereby, signifying a 19% viscosity reduction between PCE 1 and PCE 6 for the same retention, the yield stress is found to reduce from 201 Pa. to 70.2 Pa, indicating 65% reduction in yield stress. Similarly, the value of the viscosity is found to reduce from 150 Pa. s to 44.8 Pa. s, thereby, indicating 30% viscosity reduction.

It is also observed that the mixes with OPC 1 exhibits higher yield stress and viscosity over the same retention period as compared to that in case of the mixes with OPC 2. These results indicate that the PCE 6 polymers, which are a new class of admixtures, significantly reduce- both, the plastic viscosity and yield stress. On the contrary, the other PCE's are only able to reduce the yield stress. The PCE 6 represents a strong improvement in the rheological retention over other PCEs, i.e., 1-5.

These studies suggest that the HS-SCC mixes with longer side chain PCE polymers exhibit a higher yield stress and plastic viscosity retention when compared to the PCE polymers with shorter side chain. Hence, the type of PCE in HS-SCC should be decided with the utmost care, especially, when the cement type is fixed. The results also suggest that the PCE 6 (PAE), in combination with



Fig. 20 Relation between percentage change in flow and yield stress for retention period of 180 minutes



Fig. 21 Relation between percentage change in V-funnel time and viscosity for retention period of 180 minutes

OPC 2 (Low fines cement) would be a more suitable solution to reduce the effect of stickiness on the rheological retention of HS-SCC.

6.7 Relationship between flow, V-funnel, Yield stress and viscosity changes

An effort is made to find out the significance of the relationship between the changes in the flow value, *V*-funnel value, yield stress and the viscosity for the retention period of 180 minutes in HS-SCC. For this purpose, the statistical modeling is also carried out using multiple linear regression. The relationship between flow value and the yield stress is indicated in Fig. 20 and that between *V*-funnel value and the viscosity, in Fig. 21.

In absence of the ICAR Rheometer at the site the charts in Fig. 20 and Fig. 21 can be used to



Fig. 22 Effect of different PCE polymers on compressive strength

establish the impact of change of V-Funnel time and the slump flow with the change in viscosity and yield stress, respectively for the designed HS-SCC mix. Moreover, these charts show the rate of change of V-funnel value with respect to the type of OPC used.

Further, the results indicate that the percentage change in the spread of slump flow is the unique function of the percentage change in yield stress and similarly, the percentage change in *V*-funnel time is a unique function of the percentage change in viscosity. It implies that a linear correlation exists between the percentage changes. The slump flow drops over time and the yield stress correspondingly increases over the time. A similar conclusion can also be drawn for *V*-funnel time and the viscosity.

6.8 Effect on compressive strength

The compressive strength of the HS-SCC mixes obtained in respect of six different types of PCE for 1 day, 3 days, 7 days, 28 days and 56 days curing period corresponding to two different brands of OPC is shown in Fig. 22.

It is observed from Fig. 22 that the longer side chain polymers (PCE 1 and PCE 2) show better one day compressive strength as compared to shorter side chain polymers (PCE 3, PCE 4, PCE5 and PCE 6). Further, the mixes with OPC 2 show lower one day compressive strength as compared to the mixes with OPC 1 in HS-SCC mixes. However, the compressive strength of HS -SCC mixes containing OPC 2 is marginally higher than that in mixes with OPC 1 in respect of highest curing period, i.e., 56 days. It may be noted that the side chain length of PCE polymer and the fineness of the cement considerably affect the early strength gain.

7. Conclusions

The extensive experimental investigations were carried out to study the effect of different types of PCEs in conjunction with two types of OPCs on rheological retention properties of high strength self-compacting concrete (HS-SCC). The rheological properties retention control is an important parameter that has not been studied or evaluated thus far. Some of the broad conclusions arrived upon from the present investigations are given below:

i. The rheological properties and the demand of super plasticizer (admixture) in respect of HS-SCC are strongly dependent on the physical and chemical properties of the cement and the PCE structure.

ii. The PCE with longer side chain has higher water reduction capacity than the short side chain PCE. Further, the efficiency of super plasticizers [PCE 1 (HRWR), PCE 2 (HRWR)] with longer backbone and side chains is distinctly higher when compared with those built from shorter back- bone and side chains [PCE 5 (MRWR -SR- VR, PCE 6 (MRWR -SR- VR)].

iii. The PCE 6 is a new class of chemical admixtures which can reduce both, plastic viscosity and yield stress. The conventional water reducing PCE super plasticizers can reduce the yield stress.

iv. The rheological retention property is increasingly becoming important since placement of concrete occurs between 1-3 hours which has different rheology as compared to that in 5-10 min measurement. This property in respect of high strength self-consolidating concrete highly depends upon the selection of polymer type. The structure and chemistry of the PCE governs the ability to hold rheology constant over time.

v. The low fineness OPC 2 in conjunction with PCE 6 can be an effective solution for designing an optimum performance based HS-SCC for high rise construction owing to its rheological retention performance.

vi. The longer side chain PCE (PCE 1 and PCE 2) contributes lesser entrapped air as compared to shorter side chain polymer (PCE 5 and PCE 6). However, entrapped air reduces below 1% during retention period of 180 minutes in all PCE.

vii. The early compressive strength properties (one day compressive strength) highly depend on the type of PCE polymer. The longer side chain polymer helps to achieve early strength better as compared to the shorter side chain polymer. The PCE polymers exhibiting the highest slump flow loss also shows highest compressive strength at one day. However, the gain in the compressive strength does not get influenced by the types of PCE polymers.

viii. It is possible to co-relate the fundamental relationship between slump flow to yield stress changes and V-funnel time to viscosity changes with the help of statistical modeling

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