ULTRASONIC TESTS ON SETTING PROPERTIES OF CEMENTITIOUS SYSTEMS

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ABSTRACT

Continuous monitoring of strength gain in fresh cementitious system is an important assessment for determining the long term strength gain properties in concrete. The present study is aimed to evaluate the setting characteristics of fresh cementitious systems containing ordinary Portland cement (OPC) and flyash blended cement. Also, the influence of accelerators on the rate of strength gain in flyash concrete was studied systematically. A fabricated plexiglass mould was used to monitor the setting properties of fresh cement paste using an ultrasonic pulse velocity at different time intervals for different cementitious systems. The test results indicated that the pozzolanic reaction of flyash in cement was enhanced with the addition of accelerator. Also, a drastic reduction in setting time was noticed for large replacement of flyash (40%) at increased dosage of accelerator (upto 3%). However, the rate of hardening was found to be consistently higher for an optimum flyash replacement (30%) in cement with 1% of accelerator dosage and exhibited a highest compressive strength of 42.21 MPa. It can also be concluded from the ultrasonic pulse velocity test results that the cementitious system containing 20% flyash in cement with the addition of 3% accelerator dosage recorded a good improvement in the early age strength.

Keywords: cementitious systems, setting, accelerator, ultrasonic pulse velocity, compressive strength, flyash.

1. INTRODUCTION

High performance concrete made using various mineral admixtures such as fly ash, slag and silica fume have demonstrated excellent performance, in terms of both mechanical and durability properties. The key factor responsible for the contribution of supplementary cementing property by these mineral admixtures is primarily due to its inherent pozzolanic reaction. However, it is well understood from many research studies that pozzolanic action is delayed in these admixtures and resulted in delayed setting time. The consumption of soluble calcium hydroxide by these pozzolanic admixtures resulted in additional hydration product.

It can be noted that the ultrasonic technique is one of the versatile nondestructive method to assess the integrity of concrete and this research paper primarily deals with the monitoring of setting properties of fresh cementitious systems using ultrasonic pulse velocity technique. According to the theory of the sound propagation in solids, the sound transmission velocity is a function of the density and an elastic modulus of the material. Accordingly, waves were analyzed to determine the ability of setting process of cementitious materials based on the different process of hydration and formation of structure and also the setting phenomena of the hydration process involved in the dormant period based on the degree of hydration and also to study the correlation between the ultrasonic wave transmission method (USWT) and ultrasonic wave reflection method (USWR) conduced on cementitious materials (Gregor Trtnik *et al*., 2009). A good number of studies were conducted to monitor the strength gained by cementitious systems by means of measuring the ultrasonic pulse velocity during the stiffening of process. It can be noted from several studies that the relationship can be derived between the compressive strength of concrete and ultrasonic pulse velocity based on the rate of hardening of the cementitious materials. Quality of the material can also be identified with the use of calibration curve obtained from the ultrasonic pulse velocity values (Romel Solıs *et al*., 2007). In this similar area of research, the microstructure formation at early ages involves the cement particle dissolution, agglomeration, precipitation diffusion and a variety of chemical reactions with lower water-to-binder ratios were systematically investigated and experimental results showed that the mechanical properties of cementitious materials changed rapidly (Zhiyong Liu *et al*., 2011). This method can be used to monitor the strength gain in cementitious materials at an early age. The chemical reactions and heat of hydration of the cementitious materials had affects the setting phenomena in the cementitious materials. The term setting has been used to describe the onset of rigidity in fresh cement pastes, mortar and concrete, a linear relationship was found between degree of hydration and the mechanical strength. The ultrasonic pulse velocity of the specimen for ordinary Portland cement with various water/cement ratio ranging from 0.4 to 0.6 (W/C ratio) was analyzed and showed the rate of setting being higher at lower water content in the cementitious systems (Kim H.C *et al*., 1988). In an another study the effect of adding mineral admixtures with ordinary Portland cement (OPC) on the compressive strength and ultrasonic pulse velocity (UPV) were determined for different curing period. It was noted that, with the increase in the curing period, both compressive strength and UPV of all the samples increased especially for samples containing fly ash. The relationship between UPV and compressive strength was found to be exponential for mineral admixtures (Ramazan *et al*., 2004). The non-destructive testing using ultrasonic testing technique was further extended to study the steel fiber reinforced cementitious materials to predict the influence of the fibre geometry and volume fraction on the mechanical properties of concrete (Acebes M *et al*., 2011). Further studies are needed to throw light on the strength development of cementitious systems at early stages of hardening and to identify the factors influencing the rate of strength development in flyash concrete.

The present study is aimed to explore the setting characteristics of different types of cementitious system containing flyash and accelerator at different dosages. Also, the rate of hardening was monitored using a portable ultrasonic pulse velocity tester. The influence of accelerator and pozzolanic reaction of flyash on the setting characteristics is widely studied using ultrasonic test by monitoring the rate of setting characteristics in fresh cementitious paste and mortar.

2. MATERIALS AND TEST METHODS

An ordinary Portland cement of 53 grade (OPC) and Portland pozzolona cement (PPC) was used throughout the investigation. The physical and chemical properties of cement tested as per IS 4031-1988 and found to be conforming to various specifications as per IS 12269-1987. The properties of the cement are provided in Tables 1 and 2. The various proportions of cementitious mixes were arrived based on flyash dosage levels and accelerator dosage which is provided in Table-3. A class F Flyash was used as pozzolanic material at different replacement level of 10, 20, 30, 40 and 50% by weight of cement and a calcium nitrate based accelerator of specific gravity 1.12 was used throughout the study. Cement mortar mixtures of ratio 1:3 was prepared based on the IS 4031 - 1988 proportions (cement replaced with flyash and accelerator dosage level of 1, 2 and 3% by weight of binder material). The setting properties of fresh cementitious material were studied using PUNDIT (portable ultrasonic pulse velocity Tester) in a plexiglass mould which was fabricated for a standard size of 100 x 100 x 100 mm. The transducers were fixed to the sides of specimen and clamped using a fixed arrangement as shown in Figure-1. The pulse velocity was measured for different fresh cementitious mixtures at different time interval and monitored immediately after mixing with water and placed in the mould. Similarly, the ultrasonic tests were conducted for mortar specimens and the detailed mix constituents are given in Table-4.

Table-1. Physical properties of various materials used in the study.

Observation	**Fine aggregate**	**Flyash class - F**	**Cement** (OPC)	**Cement** (PPC)
Specific gravity	2.63	2.1	3.16	3.1
Average particle size (µm)	1.18	10	45	30
Bulk density (kg/m^3)	1604	492	1510	1550
Specific surface area (m^2/kg)	-	300	349	360
Fineness modulus (m^2/kg)	2.67	400	325	310
Normal consistency (%)	-	48	31	32
Grading	Confirming to IS 383 -1970 (Zone II)		53	-

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ARPN Journal of Engineering and Applied Sciences

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Table-2. Chemical characteristics of class - F flyash and cement used in the study.

Observation	**Fly ash class F** (%)	**Cement**
SiO_2	60.54	20.81
Al_2O_3	26.20	4.79
Fe_2O_3	5.87	3.2
CaO	1.91	63.9
MgO	0.38	2.61
SO_3	0.21	2.27
Na_2O	2.16	0.18
K_2O	0.01	0.75
Cl^-	0.52	0.002
Loss on ignition	2	0.98
Insoluble residue	-	0.12
Moisture content	0.73	-

Table-3. Details of various flyash levels and accelerator dosage used in the study.

Type of mix Constituent	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**
OPC + Accelerator	1, 2 and 3%				
OPC + Fly ash	10%	20%	30%	40%	50%
Accelerator dosage	1, 2 and 3%	1, 2 and 3%	1, 2 and 3%	1, 2 and 3%	1, 2 and 3%
PPC + Accelerator dosage	1, 2 and 3%				

Figure-1. Experimental test setup showing the ultrasonic pulse velocity measurements.

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Table-4. Ultrasonic pulse velocity (m/sec) test results for various fresh cementitious mixtures.

Mix Id	**Ultrasonic pulse velocity** (m/sec)						
	0 (min)	**30** (min)	**60** (min)	**90** (min)	**120** (min)	**150** (min)	**180** (min)
OPC	1480	1500	1250	1020	1200	2240	2260
OPC1	1510	1540	1560	1600	1620	1580	1610
OPC2	1500	1580	1530	1580	1620	1690	1780
OPC3	1610	1620	1640	1700	1740	1800	1850
PPC	1090	1040	1170	1360	1200	1320	1420
PPC1	1040	1050	1390	1390	1080	1230	1220
PPC2	1000	1060	1000	1020	1080	1120	1210
PPC3	1030	1030	1090	1130	1140	1180	1200
A0	1220	1200	1320	1410	1480	1640	1740
A1	1210	1190	1190	1230	1380	1510	1600
A2	1070	1130	1300	1320	1400	1600	1640
A3	1300	1010	1320	1360	1520	1700	1720
B0	1370	1790	1440	1720	1690	1800	1970
B1	1040	1090	1020	1020	1110	1180	1200
B2	1090	1180	1180	1200	1260	1250	1320
B3	1210	1370	1440	1510	1620	1780	2010
C0	1290	1100	1360	1130	1110	1170	1240
C1	1020	1000	1020	1080	1020	1120	1160
C2	1120	1040	1200	1120	1200	1250	1280
C3	1150	1170	1090	1240	1250	1370	1410
D0	1140	1150	1030	1130	1140	1220	1230
D1	1010	1030	1080	1120	1140	1100	1210
D2	1020	1040	1080	1070	1090	1190	1280
D3	1010	1160	1140	1230	1270	1240	1350
E0	1110	1120	1080	1030	1190	1150	1180
E1	1040	1030	1050	1200	1100	1100	1220
E2	1100	1050	1090	1160	1100	1140	1260
E3	1070	1070	1240	1240	1250	1280	1300

3. EXPERIMENTAL TEST RESULTS AND DISCUSSIONS

3.1. Setting properties of cementitious systems

The test results on the pozzolanic reactivity of flyash in cement are given in Table-5. It can be noted from the result that the pozzolanic action of flyash is effective at an accelerator dosage of 2%. However, with the increased dosage of flyash the effect of accelerator on the pozzolanic action is not realized. This can be evidently seen from the setting time of different cementitious mixes given in Table-6. It can be noted from Figures 2 and 3 that the setting time of PPC mixes were found to be higher compared to OPC. With the addition of accelerator in PPC the addition of higher dosage of accelerator showed good reduction in setting time. However, with the addition of 40% flyash in OPC an accelerator dosage at 3% there was a phenomenal decrease in the setting time. However, with the increasing flyash dosage, the initial setting time alone was reduced at higher dosage of accelerator. This can be substantiated that the optimum addition of flyash for good pozzolanic reaction has direct effect on the accelerator dosage.

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Table-5. Pozzolanic activity index of class-F flyash (determined as per IS-1727-1967).

Fly ash (%)	**Cement** (%)	**Accelerator** (%)	**PAI index at 7 days**	**PAI index at 28 days**
25	75	0	57.08	69.35
50	50	0	39.98	51.23
25	75	1	59.71	72.04
25	75	2	61.34	73.25
25	75	3	60.87	72.06
50	50	1	41.42	55.22
50	50	2	46.78	59.41
50	50	3	46.01	57.02

Table-6. Initial and final setting time of cement for various percentages of admixtures.

Mix ID	**Constituents**			**Setting time** (minutes)		**Time lag** (minutes)
	Cement (%)	**Fly ash** (%)	**Acl** (%)	**Initial**	**Final**	
OP	100	0	0	160	265	105
OP1	100	0	1	95	176	81
OP2	100	0	2	45	152	107
OP3	100	0	3	35	73	38
PPC	100	0	0	170	275	105
PPC1	100	0	1	80	133	53
PPC2	100	0	2	65	124	59
PPC3	100	0	3	53	117	64
A0	90	10	0	130	240	110
A1	90	10	1	87	136	49
A2	90	10	2	65	185	120
A3	90	10	3	47	140	93
B0	80	20	0	79	177	98
B1	80	20	1	60	155	95
B2	80	20	2	45	87	42
B3	80	20	3	36	70	34
C0	70	30	0	80	120	40
C1	70	30	1	45	115	70
C2	70	30	2	34	94	60
C3	70	30	3	20	60	40
D0	60	40	0	90	150	60
D1	60	40	1	80	170	90
D2	60	40	2	65	155	90
D3	60	40	3	20	58	38
E0	50	50	0	88	200	112
E1	50	50	1	60	155	95
E2	50	50	2	52	142	90
E3	50	50	3	22	137	115

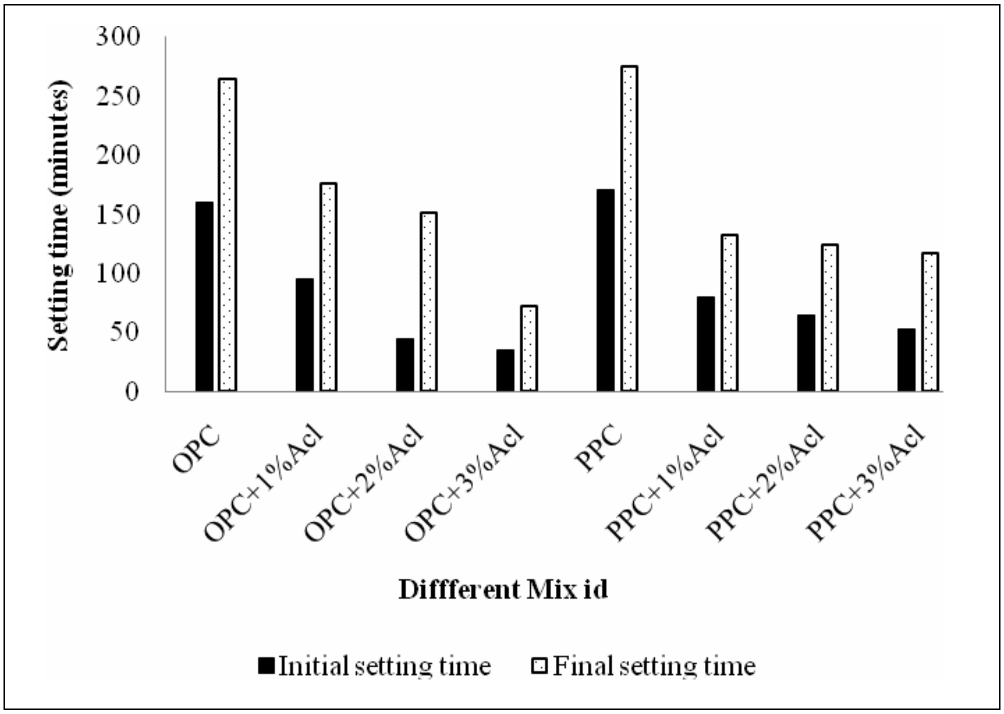

Figure-2. Initial and final setting time results for OPC and PPC with different percentage of accelerator.

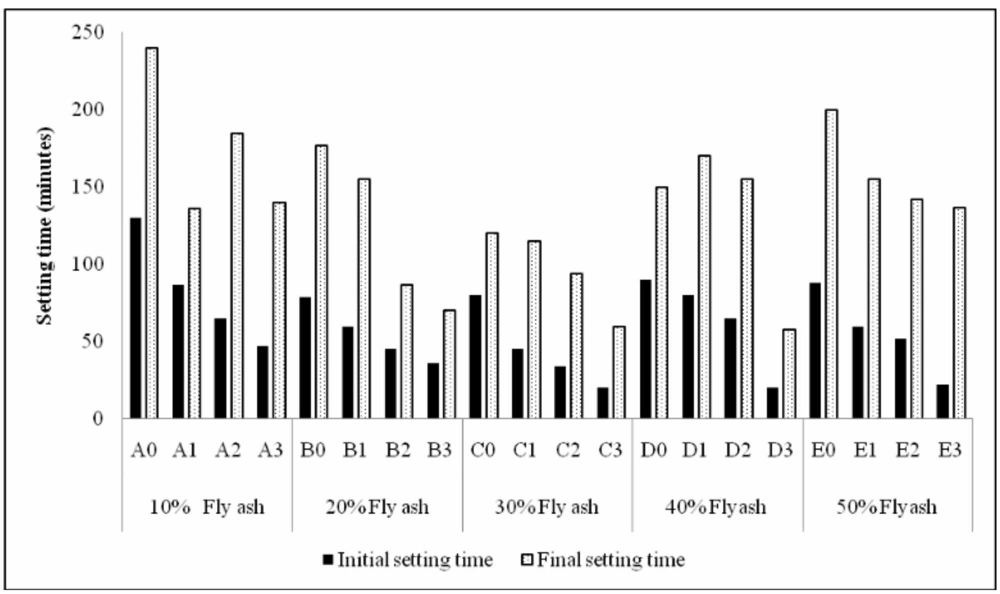

Figure-3. Initial and final setting time for different replacement level of flyash and accelerator.

3.2. Monitoring early age strength gain properties

The experimental trends showing the rate of setting for different cementitious systems are represented in Figures 4 to 11. It can be noted from Figure-4 that, with the increased dosage of accelerator the setting properties of the cementitious system are enhanced. When OPC substituted with flyash at 20%, the ultrasonic pulse values was found to be higher (as seen in Figure-5). It is clearly noted from Figures 6 and 7 that, at higher replacement of flyash higher dosage of accelerator (3%) are required for obtaining higher pulse velocity. However, with an optimum fly ash addition upto 30% and 3% accelerator the ultrasonic pulse value was found to be higher (as seen in Figures 8 and 9). Also, the higher dosage of flyash at 50% the effect of accelerator is marginally improved the pulse velocity. This indicates that the degree of hydration at the same duration is reduced in case of high volume flyash based mixture due to prolonged dormant period and delay in the acceleration period of hydration. The addition of 30% flyash based mixes showed gradual increase in the ultrasonic pulse velocity with increased pozzolanic activity due to accelerating admixture which also influences the setting phenomena at different time interval probably after the end of dormant period. The ultrasonic pulse velocity test conducted on various cement mortar specimens showed that the pulse velocity values (given in Table-7) were found to be consistently satisfying the IS 13311 specifications (above 3500 m/sec). However, for all the flyash based mixes marginal improvement in pulse velocity was observed due to early pozzolanic reaction as a result of activation energy provided by the accelerator. The effect of accelerator is observed only at higher dosage which increases the rate of hardening in fly ash mixed OPC. However, it can be seen that the pulse velocity was found to lesser in high volume flyash (50%) mortar specimens due to delayed pozzolanic reaction and the effect of accelerator is not phenomenal even at higher dosage. In general it can be justified that, for an optimum fly ash addition up to 30% with an accelerator dosage of 2% the ultrasonic pulse velocity was found to be higher.

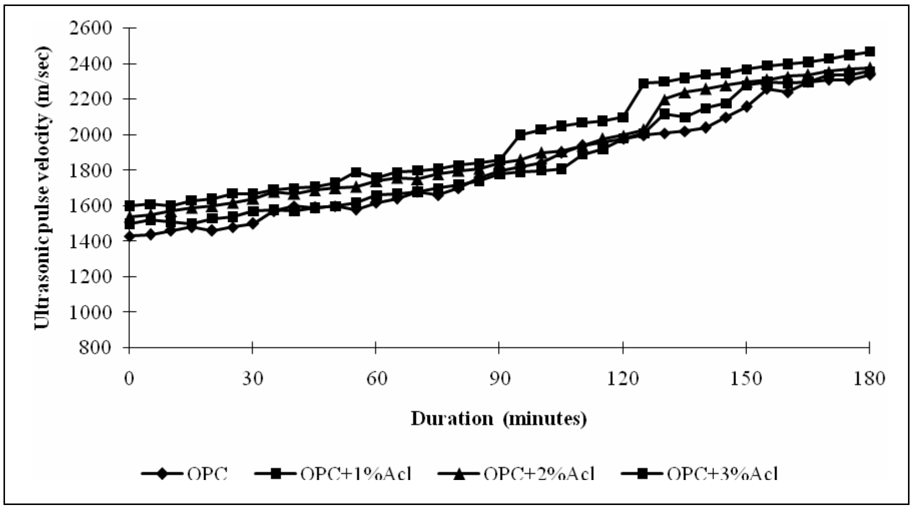

Figure-4. Ultrasonic pulse velocity of fresh cement paste for different percentage of accelerator.

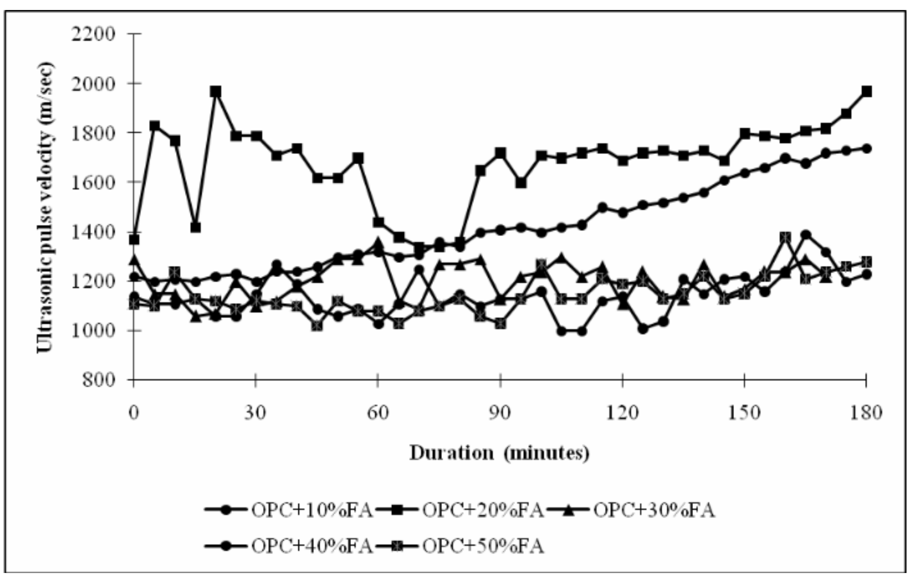

Figure-5. Ultrasonic pulse velocity of fresh cement paste for different percentage of flyash.

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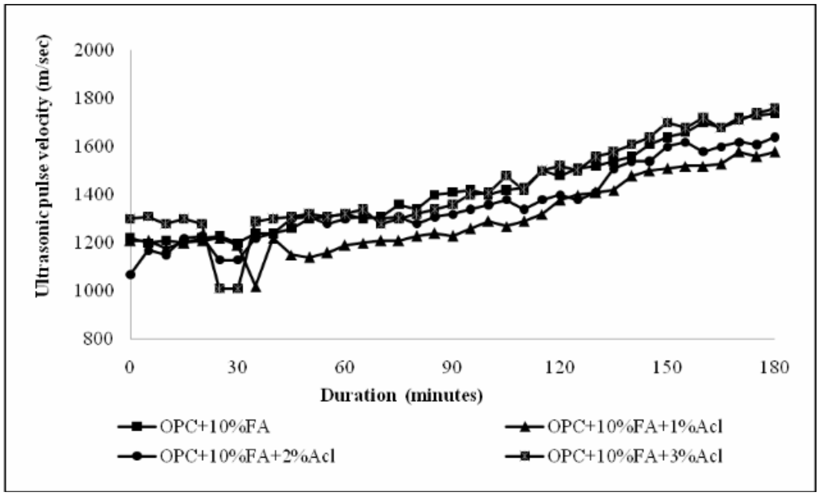

Figure-6. Ultrasonic pulse velocity of fresh cement paste for 10% flyash and different dosage of accelerator.

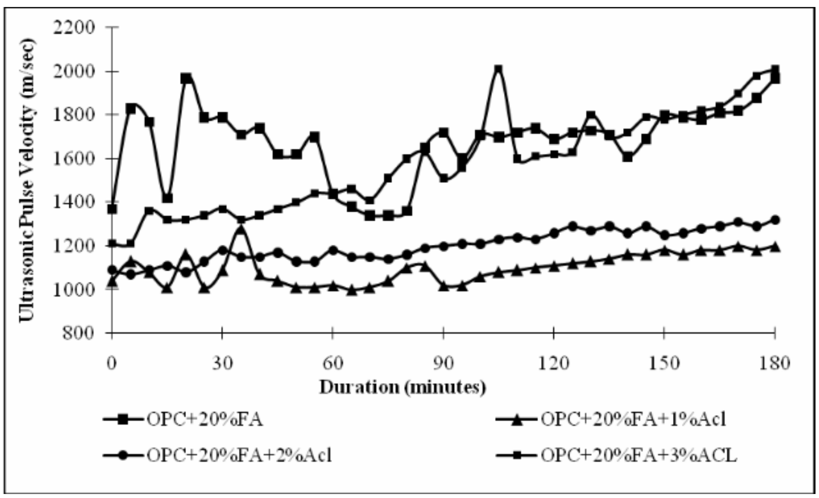

Figure-7. Ultrasonic pulse velocity of fresh cement paste for 20% flyash and different dosage of accelerator.

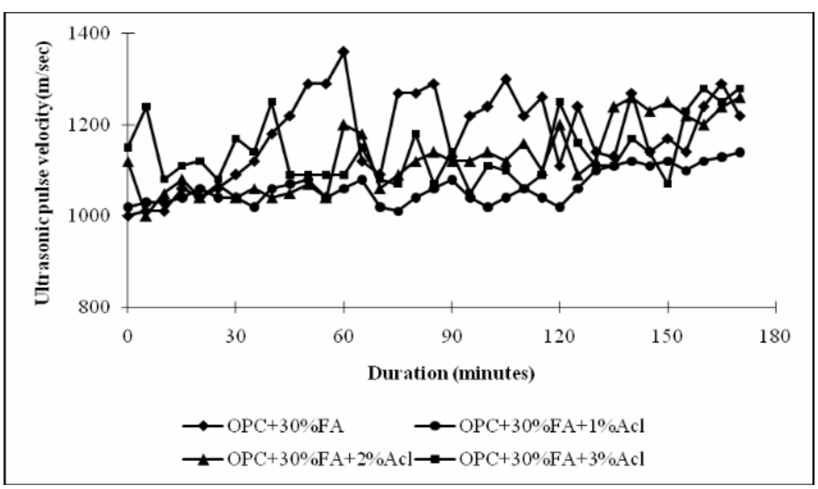

Figure-8. Ultrasonic pulse velocity of fresh cement paste for 30% flyash at various dosage of accelerator.

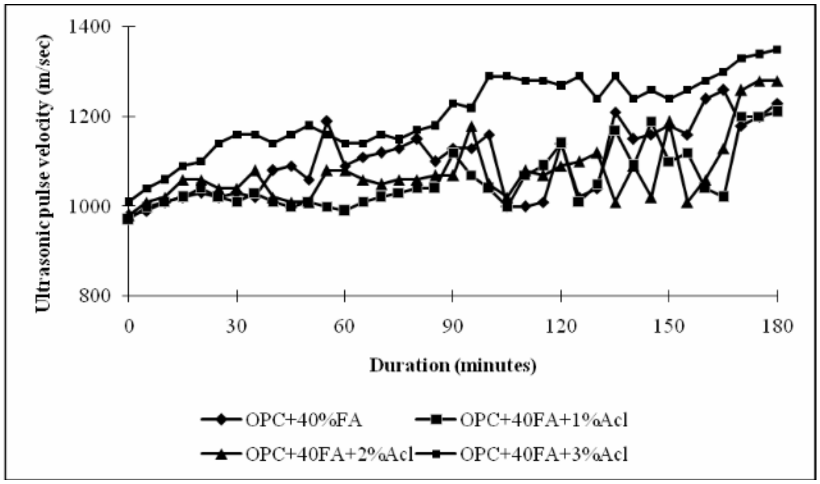

Figure-9. Ultrasonic pulse velocity of fresh cement paste for 40% flyash at various dosage of accelerator.

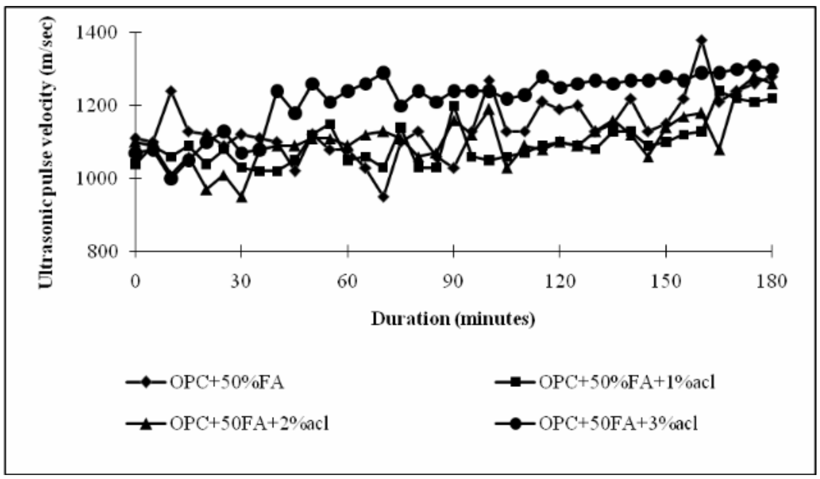

Figure-10. Ultrasonic pulse velocity of fresh cement paste for 50% flyash at various dosage of accelerator.

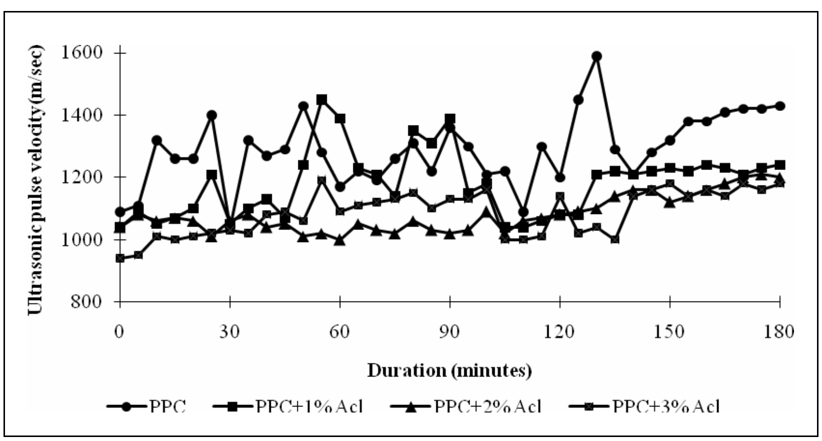

Figure-11. Ultrasonic pulse velocity of fresh cement paste containing PPC at various dosage of accelerator.

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ARPN Journal of Engineering and Applied Sciences

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Table-7. Ultrasonic pulse velocity (m/sec) test results for various mixture proportions of cement mortar cubes.

Ultrasonic pulse velocity (m/sec)			
Mix Id	**Curing days**		
	7	**14**	**28**
OPC	3740	3780	3820
OPC1	3680	3700	3720
OPC2	3640	3660	3700
OPC3	3530	3600	3620
PPC	3720	3830	3900
PPC1	3650	3700	3720
PPC2	3710	3740	3800
PPC3	3680	3700	3720
A0	3780	3790	3860
A1	3640	3650	3820
A2	3680	3720	3790
A3	3590	3620	3740
B0	3780	3970	4010
B1	3750	3880	4020
B2	3590	3440	3670
B3	3740	3480	3730
C0	3780	3880	3920
C1	3580	3550	3800
C2	3650	3740	4000
C3	3660	3910	3810
D0	3620	3780	3830
D1	3640	3720	3810
D2	3310	3380	3510
D3	3340	3485	3520
E0	3740	3830	3900
E1	3580	3300	3640
E2	3580	3605	3700
E3	3680	3700	3880

3.3. Compressive strength of cement mortar cubes

The compressive test results on cement mortar for various mortar mixture proportions are presented in Table-8. It can be observed from the results that addition of accelerator in ordinary Portland cement had shown an increase in strength (43.3 MPa) with lower dosage of accelerator and the rate of strength gain was found to be higher than OPC without accelerator. In the case of Portland pozzolana cement the effect of accelerators at higher dosages is not realized and exhibited a highest strength of 31.3 MPa only at 1% of accelerator. Also compared to all other flyash based mixes, the mix containing OPC with flyash at 30% with 1% accelerator recorded a maximum compressive strength of 42.21 MPa. The higher replacement level of fly ash beyond 30% resulted in harsh mix and becomes still poorer with higher dosage levels of accelerator due to faster setting and resulted in poor mixing. The effect of accelerators on the strength gain was realized in the mix containing 30% flyash with 1% accelerator. This can be concluded from the test result that the optimum addition of accelerator (1%) has direct effect on the setting and pozzolanic action of flyash. Also, the increased dosage levels of accelerator may not result in uniform mixing due to loss in consistency and poor workability. This may result in non-homogeneous mortar with air pockets and result in loss in strength.

VOL. 7, NO. 11, NOVEMBER 2012 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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Table-8. Compressive and rate of strength gain for cement mortar.

Mix ID	Constituents			Comp strength (MPa)			Strength gain (%) w.r. t 28 days	
	Cement (%)	Fly ash (%)	Acl (%)	7th day	14th day	28th day	7th day	14th day
OP	100	0	0	21.32	29	42	51	69
OP1	100	0	1	28.19	34.1	43.4	65	79
OP2	100	0	2	27.62	32.4	39.2	70	83
OP3	100	0	3	33.1	34	41.2	80	83
PPC	100	0	0	18.32	23.21	33.1	55	70
PPC1	100	0	1	23.41	24.51	31.3	75	78
PPC2	100	0	2	21.52	23.7	29.32	73	81
PPC3	100	0	3	20.31	21.42	25.62	79	84
A0	90	10	0	24.2	31.3	37	65	85
A1	90	10	1	26.7	33.7	40.26	66	84
A2	90	10	2	25.1	26.1	28.75	87	91
A3	90	10	3	20.31	22.7	26.4	77	86
B0	80	20	0	24.01	29.99	38.9	62	77
B1	80	20	1	27.78	37.3	41.2	67	91
B2	80	20	2	24.45	29.2	39.92	61	73
B3	80	20	3	21.2	24.51	34.83	61	70
C0	70	30	0	29.68	33.83	40.89	73	83
C1	70	30	1	34.17	38.1	42.21	81	90
C2	70	30	2	29.7	32.17	38.72	77	83
C3	70	30	3	26.31	27.8	32.2	82	86
D0	60	40	0	17.34	23.67	24.28	71	97
D1	60	40	1	16.0	21.0	22.59	71	93
D2	60	40	2	15.0	17.8	25.28	59	70
D3	60	40	3	14.8	16.4	17.39	85	94
E0	50	50	0	17.45	13.85	22.27	78	62
E1	50	50	1	15.8	13.5	19.46	81	69
E2	50	50	2	12.35	16.75	16.85	73	99
E3	50	50	3	13.0	17.5	17.86	73	98

4. CONCLUSIONS

The important conclusions arrived based on the experimental investigations are summarized below:

i) The rate of pozzolanic reactivity was found to higher for flyash based mixes at an optimum level of flyash at 25% and 2% accelerator.

ii) Setting property was found to be decreased for 40% flyash and 3% accelerator and the time lag was found to be around 38 minutes. This showcases the early strength gain properties of cementitious system and can be ideal choice for fast track concreting applications.

iii) Ultrasonic pulse velocity measurements were found to be an ideal tool for monitoring the early age strength gain properties of cementitious systems. The choice of accelerator and flyash replacement levels can be readily judged from the pulse velocity measurements. From the test results it can be concluded that a good agreement of pulse velocity was observed for cementitious mixes containing 20% flyash and 3% accelerator.

ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

iv) With the increased dosage of accelerator the improvement in setting is noticed in all the mixes, however, the rate of hardening at higher dosages was affected due to loss in consistency and immediate setting. In the case of optimum flyash addition up to 30% and accelerator dosage between 1 to 2% the rate of setting was better than PPC and on par with OPC.

v) In the case of mortar studies conducted the flyash replacement at 30% and 1% accelerator had shown a highest compressive strength of 42.2 MPa which was found to higher than PPC. This evidently denotes that the optimum addition of flyash at 30% in cement resulted in better strength than blended cement (PPC) without strict quality control on the amount of flyash addition.

vi) The ultrasonic pulse velocity conducted on mortar specimens dictated that high quality mixes was obtained for flyash at 20% flyash and 1% accelerator which was satisfying the IS 13311 codal provisions.

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