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Development of rapid-set high-strength cement using statistical experimental design

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Abstract

Many applications like aircraft runway, busy roads, highway or motorways, water tank repair, etc. demand a cement that sets fast and gains the required strength in a few hours. Though there are few cements available to meet the requirements given above, most of them are very costly, like magnesium phosphate cement, jet cement, geopolymeric cement, etc. So, an attempt has been made to make cost-effective rapid-set high-strength cement having initial setting time of ~15 min, final setting time of ~30 min, 4 h cold compressive strength (CCS) of ~12 MPa (minimum), 8 h CCS of ~24 MPa and 1 day CCS of ~40 MPa for the neat cement. The experiments were designed using orthogonal array technique in L_9 array with three factors, namely OPC/high-alumina cement/anhydrous calcium sulphate, fineness of the cement, and type of additives, at three levels each. The responses studied are initial setting time, final setting time, and CCS after 4, 8, and 24 h curing. The response data were analysed using analysis of variance (ANOVA) technique with a software package, ANOVA by Taguchi Method (ATM). In the case of setting time, fineness of the cement and OPC/HAC/anhydrous calcium sulphate ratio plays a significant role. Additive type and the OPC/HAC/anhydrous calcium sulphate are significant factors affecting the CCS at different ages. The confirmatory trial results clearly indicate that the setting time and CCS at different ages targeted were achieved using design of experiments.

Keywords: Compressive strength; Admixture; Cement paste; Mortar; Experimental design

1. Introduction

Rapid setting and high early strength in cement can be obtained by any one or a combination of the following methods:

1. by modification of the mineral phase composition
2. by addition of admixtures
3. by multicomponent mixtures
4. by increasing the fineness of the cement.

Few rapid-set high-strength binders available in the literature are calcium sulphoaluminate-based cements (C_4A_3S), calcium fluoroaluminate ($C_{11}A_7 \cdot CaF_2$) with calcium sulphate ($CaSO_4$) or OPC, magnesium phosphate cements, geopolymeric cements, OPC with calcium aluminate additives, glass cements, etc. Most of these cements are costly. In all these cements, except magnesium phosphate and glass cements, the early setting and strength is due to the rapid formation of ettringite [1–10]. Glass cements of $CaO-Al_2O_3-SiO_2$ system when ground to fineness in the range of 5000 Blaines leads to rapid set and hardening due to the formation of hydrogarnet and Stratlingite phases [11].

Rapid-set high-strength cement is a special type of cement which can be used in emergency repair works such as a highway repair during traffic, in dams, tanks, airport runways, sanitary and plumbing systems, and busy places like pedestrian walk ways.

2. Experimentation approach

In the conventional approach of experimentation, at a time, one factor is kept varying, and all the other factors are kept constant. The optimum conditions arrived in the conventional approach may not be a true optimum if the interactions between the factors are present. To study the

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Table 1
Factors and levels of the experiment

Factors	Levels		
	1	2	3
(A) OPC/high-alumina cement/anhydrous calcium sulphate	70:20:10	75:15:10	80:15:5
(B) Fineness of the cement (cm^2/g)	5000	5500	6000
(C) Additive type	alkali carbonate (AC)	alkali sulphate (AS)	mixture of alkali carbonates (MAC)

factors and its interactions, factorial experiments and response surface designs are available. In the case of a full factorial design, the number of experiments is numerous, and it is practically not possible to carry out the experiments in most of the cases. So the fractional factorial experiments using orthogonal array was investigated by Taguchi [12,13], which can substantially decrease the number of experiments and feasible to study the effect of factors and its interactions. The linear graph developed by Taguchi [12] is useful to scientists and engineers to design and analyse the experimental data without having basic knowledge of factorial design by Galois field.

Table 2
Orthogonal array for $L_9(3^4)$

Experiment number	Columns			
	A	B	C	e
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Table 3
Physical layout for the experiments

Experiment number	OPC/HAC/anhydrous $CaSO_4$	Fineness of cement	Additive type
1	70:20:10	5000	AC
2	70:20:10	5500	AS
3	70:20:10	6000	MAC
4	75:15:10	5000	AS
5	75:15:10	5500	MAC
6	75:15:10	6000	AC
7	80:15:5	5000	MAC
8	80:15:5	5500	AC
9	80:15:5	6000	AS

Table 4
Chemical analysis of raw materials used in the present work

Material	Weight (%)								
	LOI	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	TiO_2	Free lime
OPC-43 grade cement	3.50	60.40	20.60	5.90	5.10	0.90	2.41	–	0.90
High-alumina cement	0.35	24.50	0.95	73.30	0.20	0.45	–	Trace	–

Thus, in the present work, an attempt has been made to develop chloride-free rapid-set high-strength cement using three factors at three levels for each as shown in Table 1.

3. Design of experiments

The experiments were designed based on orthogonal array technique. Factors and its levels affecting setting time and cold compressive strength (CCS) at different ages were

Table 5
Properties of OPC used in the present work

Material	Fineness of cement (Blaines)	Setting time (min)		CCS of neat cement (MPa)		
		Initial	Final	4 h	8 h	24 h
OPC	3100	180	235	0	2.94	10.79

Table 6
Properties of high-alumina cement used in the present work

Material	Fineness of cement (Blaines)	Setting time (min)		CCS (MPa) (1:3 mortar)		Refractoriness Orton cone
		Initial	Final	1 day	1 day + 1 day at 110 °C	
High-alumina cement	4300	40	140	36.3	53.96	32

Table 7
Response data collected from the experiments

Experiment number	Setting time (min)				Compressive strength (MPa)		
	Initial	af.cor.*	Final	af.cor.*	4 h	8 h	24 h
1	14	− 1	30	0	1.96	11.77	23.54
2	17	+ 2	32	+ 2	6.87	9.81	33.35
3	14	− 1	27	− 3	5.89	13.73	44.15
4	25	+ 10	60	+ 30	5.89	9.81	29.43
5	22	+ 7	55	+ 25	4.91	9.81	25.51
6	14	− 1	30	0	3.92	9.81	21.09
7	25	+ 10	60	+ 30	5.89	17.66	35.32
8	23	+ 8	55	+ 25	3.92	17.66	43.16
9	15	0	34	+ 4	9.81	19.62	49.05

* Setting time after deduction of targeted setting time (15 min for initial and 30 min for final setting time).

Table 8

ANOVA table for initial setting time—after pooling

Source	Pool	Df	S	V	F	S′	rho%
A	[N]	2	64.8889	32.4444	3.4970	46.3333	24.19
B	[N]	2	89.5556	44.7778	4.8263	71.0000	37.06
C	[Y]	2	16.8889	8.4444			
Error	[Y]	2	20.2222	10.1111			
e1	[Y]	0	0.0000				
e2	[Y]	0	0.0000				
(e)		4	37.1111	9.2778		74.2223	38.75
Total (raw)	[−]	8	191.5556	23.9444			

ANOVA level average table for initial setting time—raw data

A	1	0.0000	B	1	6.3333	C	1	2.0000
	2	5.3333		2	5.6667		2	4.0000
	3	6.0000		3	−0.6667		3	5.3333

Table 10

ANOVA table for CCS at 4 h curing—after pooling

Source	Pool	Df	S	V	F	S′	rho%
A	[N]	2	5.3356	2.6678	25.0028	5.1222	13.22
B	[N]	2	5.9758	2.9879	28.0028	5.7624	14.87
C	[N]	2	27.2355	13.6177	127.6261	27.0221	69.72
Error	[Y]	2	0.2134	0.1067			
e1	[Y]	0	0.0000				
e2	[Y]	0	0.0000				
(e)		2	0.2134	0.1067		0.8536	2.20
Total (raw)	[−]	8	38.7603	4.8450			

ANOVA level average table for CCS at 4 h curing—raw data

A	1	4.9067	B	1	4.5800	C	1	3.2667
	2	4.9067		2	5.2333		2	7.5233
	3	6.5400		3	6.5400		3	5.5633

decided based on the brainstorming session on the subject. Three factors and three levels of each factors have been taken for experimentation. In the orthogonal array technique, the minimum required experiments for three factors at three levels are 9, so it is designed in $L_9(3^4)$.The factors and levels are shown in Table 1.

The experimental layout as per orthogonal array for $L_9(3^4)$ is shown in Table 2 and the physical layout is shown in Table 3. In Table 2, A, B and C represent the factors shown in Table 1 and ‘e’ represents the error column. Since the test of significance requires error variance, error column is essential in the layout.

4. Materials and methodology adopted

The chemical analysis and properties of raw material used in the present work is shown in Tables 4–6. The OPC-43-grade cement, high-alumina cement (70% alumina grade) and calcium sulphate(anhydrous, 95% purity) were mixed and ground to the required fineness as shown in Table 3 in a laboratory-type ball mill. The specific surface area of the ground cement was determined by Blaines Air Permeability apparatus. The additives were dissolved in stipulated amount of water and added while preparing the test specimens.

Setting time and CCS moulds for different ages of neat cement were cast with fixed water/cement ratio of 0.27 in 50-mm cube moulds. Water/cement ratio of 0.27 was fixed based on the preliminary work to achieve good workability with minimum water/cement ratio. The setting time was determined using Vicat apparatus as per IS-4031 [14]. The CCS cubes were cured at 27 °C and 90% relative humidity for 24 h. Subsequently, the demoulded cubes were cured in water; until that time, the cubes are tested as per schedule for later ages.

5. Data analysis by ANOVA technique and discussion

The response data shown in Table 7 was analysed using analysis of variance (ANOVA) technique using a software called “ANOVA by Taguchi Method” (ATM) and data analysis are shown in Tables 8–12.

Table 9

ANOVA table for final setting time—after pooling

Source	Pool	Df	S	V	F	S′	rho%
A	[N]	2	750.2222	375.1111	7.1983	646.0000	38.91
B	[N]	2	682.8889	341.4444	6.5522	578.6667	34.85
C	[N]	2	122.8889	61.4444	1.1791	18.6667	1.12
Error	[Y]	2	104.2222	52.1111			
e1	[Y]	0	0.0000				
e2	[Y]	0	0.0000				
(e)		2	104.2222	52.1111		416.8888	25.11
Total (raw)	[−]	8	1660.2222	207.5278			

ANOVA level average table for final setting time—raw data

A	1	−0.3333	B	1	20.0000	C	1	8.3333
	2	18.3333		2	17.3333		2	12.0000
	3	19.6667		3	0.3333		3	17.3333

Table 11

ANOVA table for CCS at 8 h curing—after pooling

Source	Pool	Df	S	V	F	S′	rho%
A	[N]	2	118.9635	59.4817	55.7414	116.8293	90.42
B	[N]	2	5.9758	2.9879	2.8000	3.8416	2.97
C	[Y]	2	0.8537	0.4268			
Error	[Y]	2	3.4148	1.7074			
e1	[Y]	0	0.0000				
e2	[Y]	0	0.0000				
(e)		4	4.2685	1.0671		8.5369	6.61
Total (raw)	[–]	8	129.2078	16.1510			

ANOVA level average table for CCS at 8 h curing—raw data

A	1	11.7700	B	1	13.0800	C	1	13.0800
	2	9.8100		2	12.4267		2	13.0800
	3	18.3133		3	14.3867		3	13.7333

Table 12

ANOVA table for CCS at 24 h curing—after pooling

Source	Pool	Df	S	V	F	S′	rho%
A	[N]	2	442.1633	221.0816	3.8757	328.0769	41.82
B	[Y]	2	112.7851	56.3925			
C	[Y]	2	102.2600	51.1300			
Error	[Y]	2	127.2140	63.6070			
e1	[Y]	0	0.0000				
e2	[Y]	0	0.0000				
(e)	[–]	6	342.2591	57.0432		456.3455	58.10
Total (raw)		8	784.4224	98.0528			

ANOVA level average table for CCS at 24 h curing—raw data

A	1	33.6800	B	1	29.4300	C	1	29.2633
	2	25.3433		2	34.0067		2	37.2767
	3	42.5100		3	38.0967		3	34.9933

ANOVA is an important statistical analysis and diagnostic tool which helps us to reduce the error variance and quantifies the dominance of a control factor. The column under the rho% gives an idea about the degree of contribution of the factors to the measured response. If the rho% is high, the contribution of the factors to that particular response is more. Likewise, if the rho% is low, the contribution of the factors to that particular response is less. The rho% accounted by the different factors cumulatively should be > 60% to show that the factors and levels taken for the experiment is reasonably correct. The compounded error% due to error column in the experiment and duplication of experiment should be low. The ANOVA tables (Tables 8–12) for setting time and CCS at different ages clearly indicate that the factor selection and the experimentation is correct.

In the case of setting time, the aimed initial setting time is around 15 min, and final setting time is around 30 min. The aimed setting times were reduced from the initial and final setting time obtained from nine experiments as shown in Table 7 to achieve the targeted setting time. The ANOVA

Table 13
Summary of analysis of variance (ANOVA) tables

Response	Factors pooled	Significant factors and levels
IST	C	A1, B3
FST		A1, B3, C1
CCS (neat cement)		
4 h		A3, B3, C2
8 h	C	A3, B3
24 h	B, C	A3

Table 14
Response data for four combinations

Experiment number	Setting time (min)		CCS (MPa)		
	Initial	Final	4 h	8 h	24 h
1	14	26	5.89	9.81	39.73
2	15	24	7.85	9.81	23.54
3	11	30	12.75	22.56	41.20
4	17	28	12.75	28.45	41.20

Table 15
Test results for neat cement and cement mortar

Properties	Rapid set high-strength cement		OPC with 6000 Blaines
	(A3B3C1)	(A3B3C2)	
Initial setting time (min)	12	17	60
Final setting time (min)	28	29	115
Compressive strength (MPa)			
For neat cement paste			
4 h	13.73	17.66	0
8 h	22.56	31.88	2.45
24 h	39.24	51.99	6.87
3 days	43.16	53.46	36.79
7 days	47.09	55.92	69.16
28 days	51.01	56.90	91.23
90 days	75.54	80.93	93.20
MOR (MPa)			
After 28 days	5.69	4.91	–
For Mortar (MPa)			
(1 part cement:3 parts sand)			
4 h	–	12.26	0
8 h	–	14.72	1.96
1 day	16.19	30.41	16.19
3 days	26.98	36.30	37.28

level average of ANOVA table for both setting time to be close to zero to get into the target setting time. The level of each factors which have shown the value close to zero has been taken as a significant level. In the case of CCS, the factor level which has shown the highest ANOVA level average has been taken as a significant level. Table 13 shows the summary of the ANOVA table for setting time and CCS.

5.1. Summary of the ANOVA table

Based on Table 13, four more trials were carried out to obtain the optimum conditions. The four experiments are (1) A1B3C1, (2) A1B3C2, (3) A3B3C1, and (4) A3B3C2 where A—OPC/(HAC + $CaSO_4$), B—fineness of cement (6000 Blaines), and C—Additive type.

The response table for the above four combinations are given in Table 14.

5.2. Response table for four combinations

Since the Experiment numbers 3 and 4 in Table 14 have shown comparatively higher CCS at all ages and setting time close to the target, further tests on neat cement and cement mortar were carried out with experimental combination of numbers 3 and 4 and compared with OPC-43-grade cement ground to 6000 Blaines The test results are shown in Table 15. Table 15 clearly indicates that additive C2 is more effective in achieving the target setting time and higher CCS at all ages for neat cement and mortar. Since the CCS for neat cement and mortar at 4,8, and 24 h curing is very important for rapid repair, it was compared with OPC-43-grade cement ground to 6000 Blaines. The results clearly indicate that rapid-set high-strength cement is much better than OPC for early repair work.

6. Conclusion

1. The aim of developing chloride-free rapid-set high-strength cement is achieved using orthogonal array technique.
2. The powers of achieving the objective with the least number of experiments have been demonstrated in the present work by fractional factorial design using orthogonal array. To fulfill the objective, only 13 experiments were needed in comparison to 27 experiments in full factorial design.

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