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Experimental Evaluation of Sulphate Limits of Mixing Water in Concrete Mortar Mix

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ABSTRACT

A uniform mix of cement with sand and water is referred as Cement Mortar. But this combination of cement, sand and water has gone obsolete now-a-days. As, today is high pace time for infrastructure, rather high strength-durable infrastructure, so, many more materials are tested to be added with cement mortar to improve its engineering properties and to replace the costliest material viz. cement. Today is the scenario of sky scrapping and complex infrastructures, which results in increasing demand of basic civil engineering material i.e. cement. Engineers are looking for alternative of expensive construction since long. Cement, binder in mortar, is an expensive and exorbitant civil engineering material and it increases the Constructional budget. Not only this, but also cement marks the highest consumption throughout the world after water. Pozzolanic materials also possess binding properties and are inert in nature thus reducing heat of hydration. Pozzolanic materials generally used in mortar production are as follows:

- Slag
- Fly ash,
- Silica fume
- Metakaolin, etc,

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1. INTRODUCTION

Materials are amalgamated with PC (Portland cement) to trigger the pivotal aspect of mortar i.e strength and adhesiveness. The least discussed ingredient of mortar is Water. Water is least cared but most influencing material in case of cement mortar. Good quality water results in good mortar mix whereas poor quality water adversely affects mortar quality and strength. Standards of water used in mixing should be studied in detail.

The standards of water should be within tolerable limits, otherwise, its impurities adversely affects the major properties of cement viz. soundness, strength, setting time and durability. Another predominant effect i.e efflorescence in caused explicitly by the movement of salt from cement mortar to the surface. This salt is present in water used for mixing mortar. Potable water or water having pH between 6 and 7.5 is generally considered satisfactory for preparation of mortar.

2. Literature review

Omar Saeed Baghabra A.L.Amoudi reviewed in length in his paper how sulphate rich environment impacts the durability of reinforced cement concrete. He accentuates the complete theory behind sulphate attack in his paper. 15 different samples were tested with a major focus on sulphate attack. Corrosion of steel was accelerated due to presence of excess sulphate ions. He created an artificial/research environment by combination of sodium and magnesium sulphate and closely studied the behavioral change in the test specimens. He used 1:1:0.6 proportion of OPC, Mineral admixture and water. From results he inferred that PCC behaves better in sulphate rich environment than compared to reinforced cement concrete.

Hasan Biricik convened their study on Magnesium and sodium sulphate. They discovered the potential of wheat straw ash (WSA) as a modifier of conventional mortar. Wheat straw was collected and burned under controlled conditions in electrical furnace to produce WSA. This WSA cooled, cleaned and sieved for required gradation and used as a modifier in mortar. It showed pozzolanic properties and imparted strength to cement. The experimentations carried out in their investigation were as follows:

- Compressive strength test
- Flexural strength test
- Test for Density/Change in mass

From these experimentations they inferred that if the replacement level of cement by WSA is kept Up to 24% all the tests showed positive results. In case if the limit exceeds it weakens the cement mortar.

Kotaiah and Kumara Swamy conducted a joint study and revealed from their study the tolerable limits of various impurities in water. They presented a detailed literature detailing all the impurities along with respective tolerable limits

Bing Tian in their work investigated and explored the potential effect of sulphate attack on the production of gypsum a constitute of cement. Sulphate attack causes expansion to gypsum formation. Other ill effects of presence of sulphate are also studied in length. After detailed exploration he preented his paper in two sections: Literature and Laboratory exploration. In literature part theory of sulphate attack is explained whereas in laboratory explorations detailed experimentations were carried out and there results were analysed and discussed to reach a conclusive note, that sulphate attack hinders almost all the properties of cement.

Raphael Tixier used blended PPC to show how precisely/well these pozzolanic admixtures performs when blended with cement, otherwise poses problem of disposal or environmental pollution. These pozzolana are benefic in terms of strength, improving life of serviceability, better microstructure of concrete, durability and many more. To quantify these benefits the research was carried under two heads:

A. Exploration of micro structural properties of cement.
B. Exploration of macro structural properties of cement.

3. Material Used

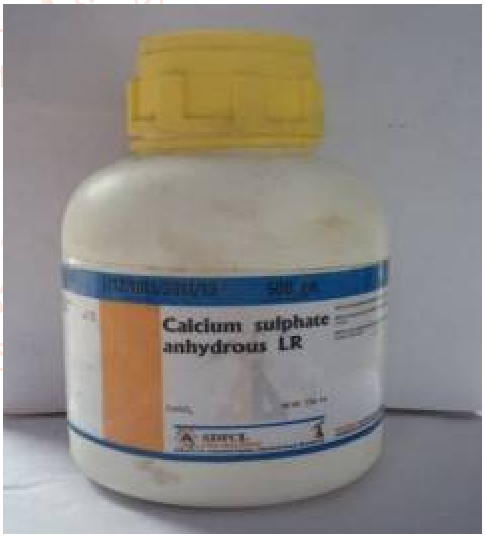
The following materials are implemented to reach the desired objectives.

1. Flyash modified cement
2. Fines or fine-aggregates
3. Different sulphates of Ca, Mg, Na, (NH4) and Fe

Fly ash blended Cement: Inorganic binding material in concrete mix is known as cement the most commonly used cement is called Portland cement. When it is mixed with water, it hardens; hence it is called hydraulic cement. Materials mainly present in cement are oxides of calcium, silicon oxide, aluminum oxide, iron oxide and alkalis, etc. the raw materials are limestone and clay or minerals, which are rich in lime, and rich in silica and alumina, these material are burnt together at a temperature of 1400 degree Celsius or so to produce clinker, which is nothing but a solid solution of these minerals forming cement compounds. Clinker is grinded further and mixed with a little bit of gypsum, of the order around 5% gypsum to control the setting properties. Production of PPC is somewhat similar to OPC the major difference is that in production of PPC 68% Clinker, 4% Gypsum and 23% Fly ash are grinded together.

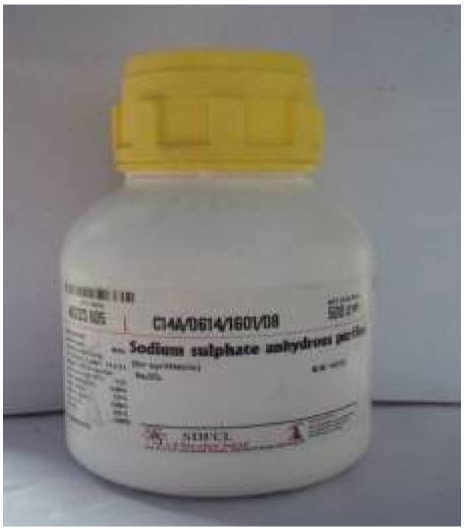
Fine Aggregates:
The small size filler materials in mortar are termed as fine aggregates. These are the particles which passes through 4.75mm sieve whereas unable to pass through 0.075mm sieve. Eg Sand, stone screenings, surkhi, cinders, burnt clay, etc. River sand, crushed stone sand, crushed gravel sand are the major sources of fine aggregates. The surface area of fine aggregate is higher than that of coarse aggregates.

Sulphates:
Sulphates used in present investigation are shown in figure below.

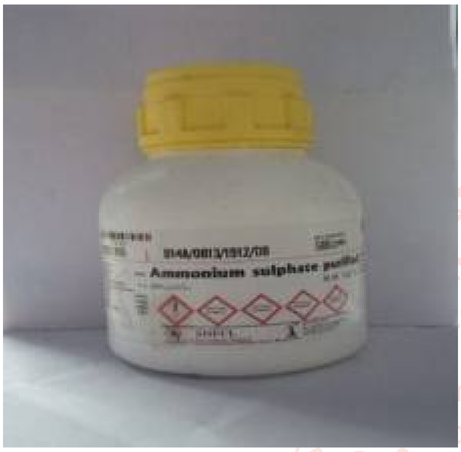


Calcium Sulphate

Magnesium Sulphate



Sodium Sulphate



Amonium Sulphate

Ferrous Sulphate

4. Methodology

Vicat's test: This test is carried out to find out initial as well as final setting time. Setting time of cement is generally a logical division of time viz. IST & FST.

IST: It is conceptually considered as the time elapsed from the addition of water to dry cement to the extent when cement paste loses its plasticity and start becoming hard.

FST: It is basically the period extent between addition of water to dry cement and stiffening of cement paste.

Apparatus: Vicat's Apparatus along with accessories.

Procedure:

1. Take 500 g of cement sample and gauge it with 0.85 times the water required to produce cement paste a standard consistency.
2. Start the stopwatch at the moment water is added to the cement the paste is prepared in the standard manner and filled into the vicat's mould within 3 to 5 minutes.
3. For measuring initial setting time attach the needle to the apparatus gently and bring it in contact with the surface of the test block and release quickly, allow it to penetrate into the test sample.
4. At the beginning the needle will completely pierce through the test block, repeat the test for every two minutes until it feels to push the block beyond 5.0±0.5 mm measured from the bottom of the mould. The time period lapsed between the time of addition of water in cement and the time at which the needle penetrate the test block to a depth equal to 5.0±0.5 mm from the top is taken as initial setting time.
5. For final setting time replace the needle of vicat apparatus by a circular attachment. This needle has two portions one is a central needle and other is a circular cutting edge of the attachments.
6. Upon lowering the circular attachment repeatedly for an equal intervals of time, at some particular point the central needle makes an impression while the circular cutting-edge fails.
7. The time period lapsed between the time of addition of water in cement and the time at which the circular attachment fails to make an impression is taken as final setting time.

Compressive strength test:

The strength of mix proportioned sample against compression is ascertained by this experiment.

Arithmetically, the value of strength of any material against compression is

Comp. Strength = Failure load/area of cross-section

Test specimen for this test is cube with the side length of 70.6 mm or 76 mm. Generally, the specimen larger than this should not be made as cement shrinks and cracks. The temperature of water and test room should be 27° ± 2° Celsius. Take cement and standard sand in the proportion of 1:3 by weight is mixed dry, with the trowel for one minute. Then add the water to form a standard consistency paste. The mix is filled in the mould completely and is placed on the vibration table.

Vibrations are imparted for about two minutes at a speed of 12000±400 rpm per minute. Three specimens are prepared and after 42 hrs removed from the moulds and submerged in clean freshwater. After a while the cubes are taken out for testing in a compression testing machine. The load is applied starting from 0 at the rate of 35 N per square millimetres per minute. Note down the maximum load at which the cube fails. The compressive strength is calculated from the crushing load divided by the average area over which the load is applied. The result is expressed in newton per millimetre square. Compressive strength is taken to be the average of the results of the three cubes.

5. Results and Discussion

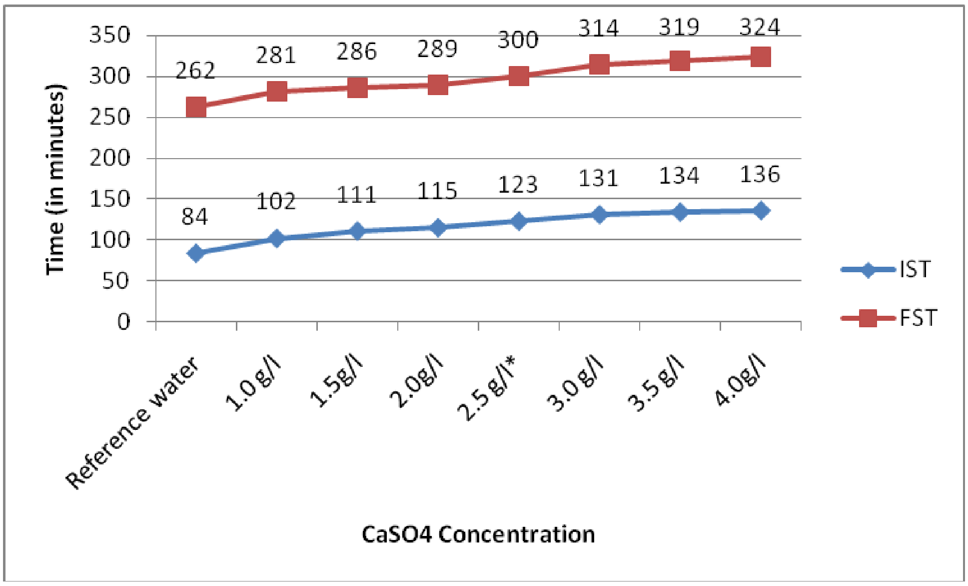
Effect of Calcium sulphate (CaSO4,)

Setting Time: This test is carried out to ascertain the setting time of different cement mix. Test is carried out with deionised water and calcium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Setting times of Fly ash Blended Cement (FBC) corresponding to CaSO4 Concentrations

Sr. No	Sample of water	S.T(in minutes), Difference and %age change					
		IST	% change	Variation	FST	% change	Variation
1	Deionised water	84	0	0	262	0	0
2	1.0 g/l	102	21.42	18	281	6.76	19
3	1.5g/l	111	32.14	27	286	9.16	24
4	2.0g/l	115	36.90	31	289	10.30	27
5	**2.5 g/l***	**123**	**46.42**	**39**	**300**	**14.50**	**38**
6	3.0 g/l	131	55.95	47	314	19.84	52
7	3.5 g/l	134	59.52	50	319	21.75	57
8	4.0g/l	136	61.90	52	324	23.66	62

*- Significant



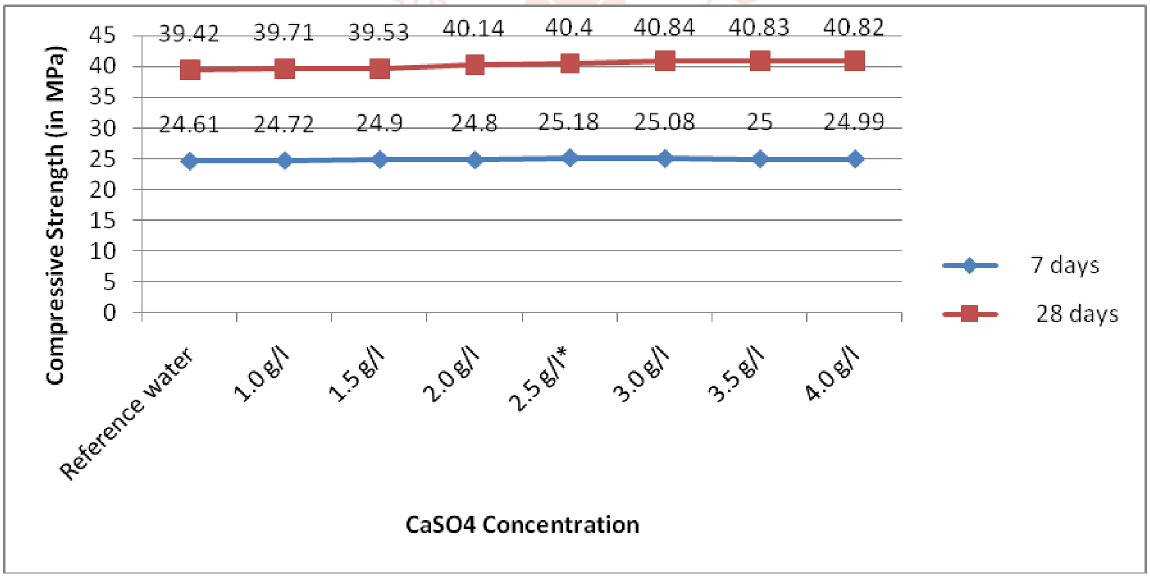
Setting time vs calcium sulphate concentration

Compressive strength test: This test is carried out to ascertain the compressive strength of different cement mix. Test is carried out with deionised water and calcium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Compressive strength of FBCM corresponding to CaSO4 concentrations.

Sr. No	Sample Description	Compressive strength (MPa)		Compressive strength variation (%)	
		7 days	28 days	7 days	28 days
1	Deionised water(Control)	24.61	39.42	0.00	0.00
2	1.0 g/l	24.72	39.71	0.44	0.73
3	1.5 g/l	24.90	39.53	1.17	0.27
4	2.0 g/l	24.80	40.14	0.77	1.82
5	**2.5 g/l***	**25.18**	**40.40**	**2.31**	**2.48**
6	3.0 g/l	25.08	40.84	1.90	3.60
7	3.5 g/l	25.00	40.83	1.58	3.57
8	4.0 g/l	24.99	40.82	1.54	3.55

*- Significant



Compressive strength vs calcium sulphate concentration

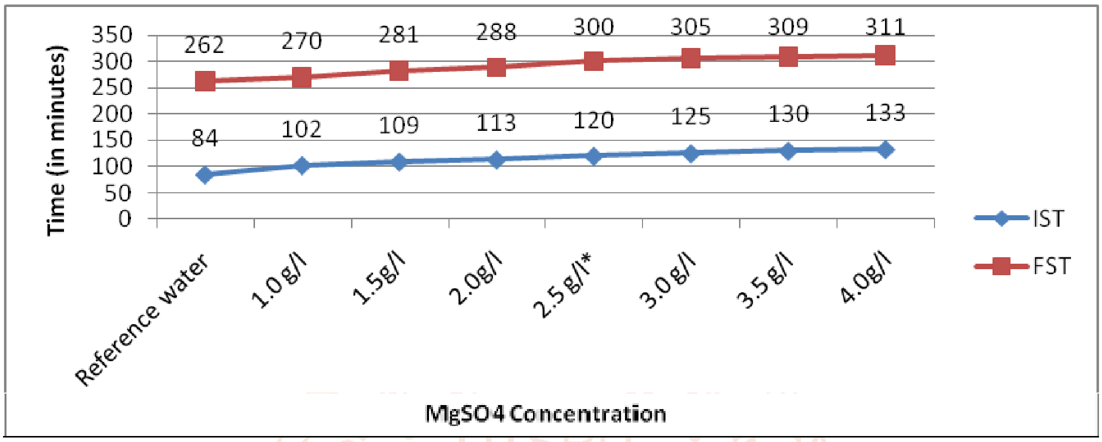
Effect of Magnesium sulphate (MgSO4,)

Setting Time: This test is carried out to ascertain the setting time of different cement mix. Test is carried out with deionised water and magnesium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Setting times of Fly ash Blended Cement (FBC) corresponding to MgSO4 Concentrations.

Sr. No	Sample of water	S.T(in minutes), Difference and %age change					
		IST	% change	Variation	FST	% Change	Variation
1	Deionised water(control)	84	0	0	262	0	0
2	1.0 g/l	102	21.4	18	270	3.05	8
3	1.5g/l	109	29.76	25	281	7.25	19
4	2 g/l	113	34.52	29	288	9.92	26
5	**2.5 g/l***	**120**	**42.85**	**36**	**300**	**14.50**	**38**
6	3 g/l	125	48.80	41	305	16.41	43
7	3.5 g/l	130	54.76	46	309	17.93	47
8	4 g/l	133	58.33	49	311	18.70	49

*- Significant



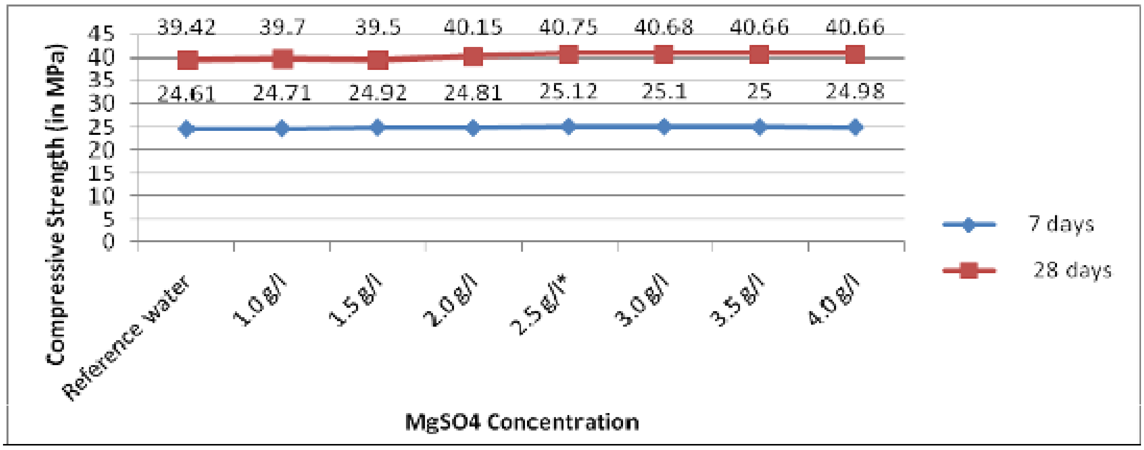
Setting time vs magnesium sulphate concentration

Compressive strength test: This test is carried out to ascertain the compressive strength of different cement mix. Test is carried out with deionised water and magnesium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Compressive strength of FBCM corresponding to MgSO4 concentrations.

Sr. No	Sample Description	Compressive strength (MPa)		Compressive strength variation (%)	
		7 days	28 days	7 days	28 days
I	Deionised water(Control)	24.61	39.42	0.00	0.00
Ii	1.0 g/l	24.71	39.70	0.40	0.71
Iii	1.5 g/l	24.92	39.50	1.25	0.20
Iv	2.0 g/l	24.81	40.15	0.81	1.85
V	**2.5 g/l***	**25.12**	**40.75**	**2.07**	**3.37**
Vi	3.0 g/l	25.10	40.68	1.99	3.19
Vii	3.5 g/l	25.00	40.66	1.58	3.14
Viii	4.0 g/l	24.98	40.66	1.50	3.14

*- Significant



Compressive strength vs Magnesium sulphate concentration

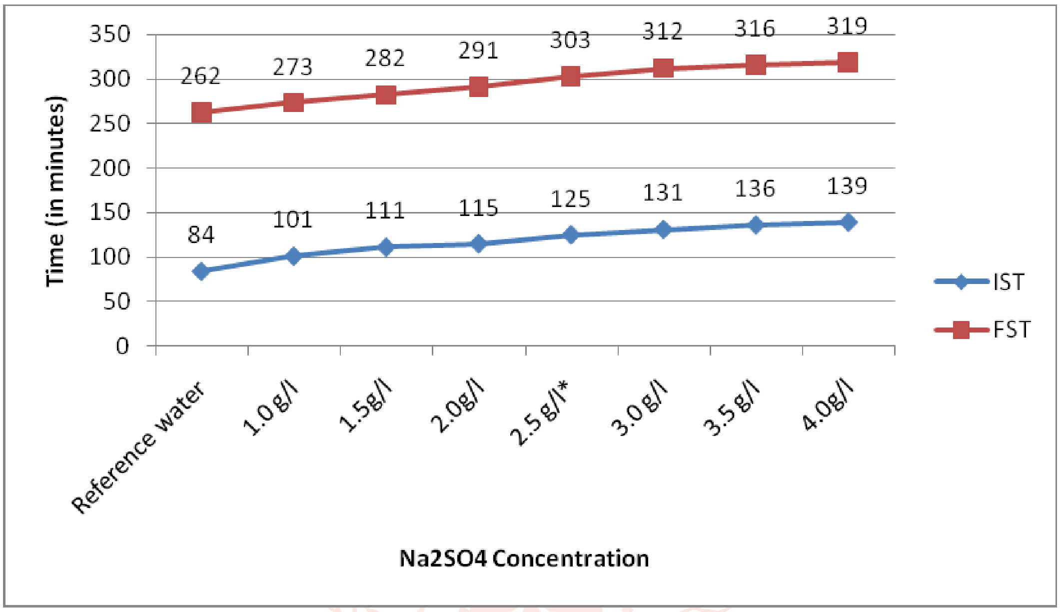
Effect of Sodium sulphate (Na_2SO_4,)

Setting Time: This test is carried out to ascertain the setting time of different cement mix. Test is carried out with deionised water and sodium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Setting times of Fly ash Blended Cement (FBC) corresponding to Na_2SO_4 Concentrations

Sr. No.	Sample of water	S.T(in minutes), Difference and %age change					
		IST	% change	Variation	FST	% change	Variation
1	Deionised water(control)	84	0	0	262	0	0
2	1.0 g/l	101	20.23	17	273	4.19	11
3	1.5g/l	111	32.14	27	282	7.63	20
4	2.0g/l	115	36.90	31	291	11.06	29
5	**2.5 g/l***	**125**	**48.80**	**41**	**303**	**15.64**	**41**
6	3 g/l	131	55.95	47	312	19.08	50
7	3.5 g/l	136	61.90	52	316	20.61	54
8	4 g/l	139	65.47	55	319	21.75	57

*- Significant



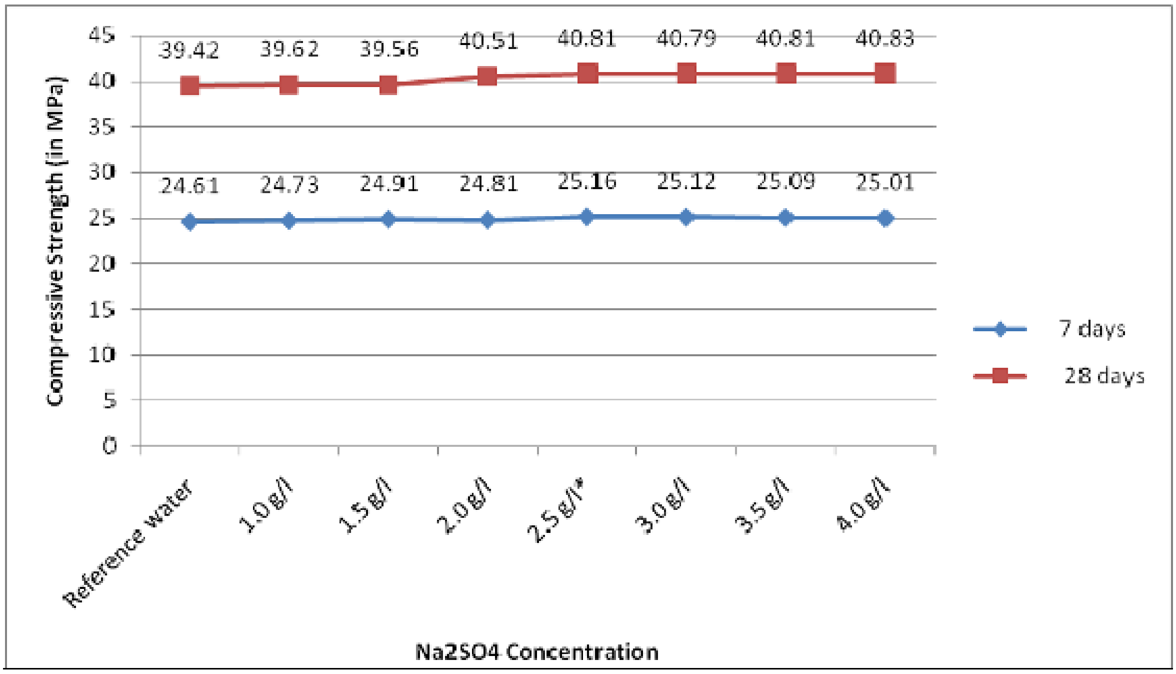
Setting time vs Sodium sulphate concentration

Compressive strength test: This test is carried out to ascertain the compressive strength of different cement mix. Test is carried out with deionised water and sodium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Compressive strength of FBCM corresponding to Na_2SO_4 concentrations.

Sr. No	Sample Description	Compressive strength (MPa)		Compressive strength variation (%)	
		7 days	28 days	7 days	28 days
I	Deionised water(Control)	24.61	39.42	0.00	0.00
Ii	1.0 g/l	24.73	39.62	0.48	0.50
Iii	1.5 g/l	24.91	39.56	1.21	0.35
Iv	2.0 g/l	24.81	40.51	0.81	2.76
V	**2.5 g/l***	**25.16**	**40.81**	**2.23**	**3.52**
Vi	3.0 g/l	25.12	40.79	2.07	3.47
Vii	3.5 g/l	25.09	40.81	1.95	3.52
Viii	4.0 g/l	25.01	40.83	1.62	3.57

*- Significant



Compressive strength vs Sodium sulphate concentration

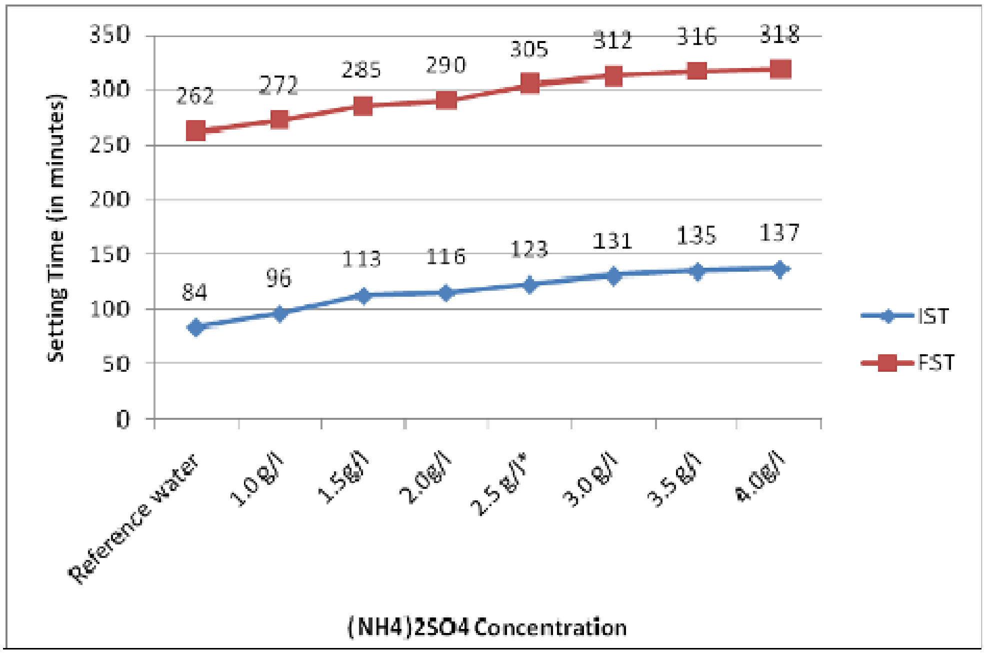
Effect of Ammonium sulphate ((NH4)2SO4,)

Setting Time: This test is carried out to ascertain the setting time of different cement mix. Test is carried out with deionised water and amonium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Setting times of Fly ash Blended Cement (FBC) corresponding to(NH4)2SO4 concentrations

Sr. No	Sample of water	S.T(in minutes), Difference and %age change					
		IST	% Change	Variation	FST	% change	Variation
1	Deionised water(control)	84	0	0	262	0	0
2	1.0 g/l	96	14.28	12	272	3.81	10
3	1.5g/l	113	34.52	29	285	8.77	23
4	2.0g/l	116	38.09	32	290	11.06	29
5	**2.5 g/l***	**123**	**46.42**	**39**	**305**	**16.41**	**43**
6	3.0 g/l	131	55.95	47	312	19.08	50
7	3.5 g/l	135	60.71	51	316	20.61	54
8	4.0g/l	137	63.09	53	318	21.37	56

*- Significant



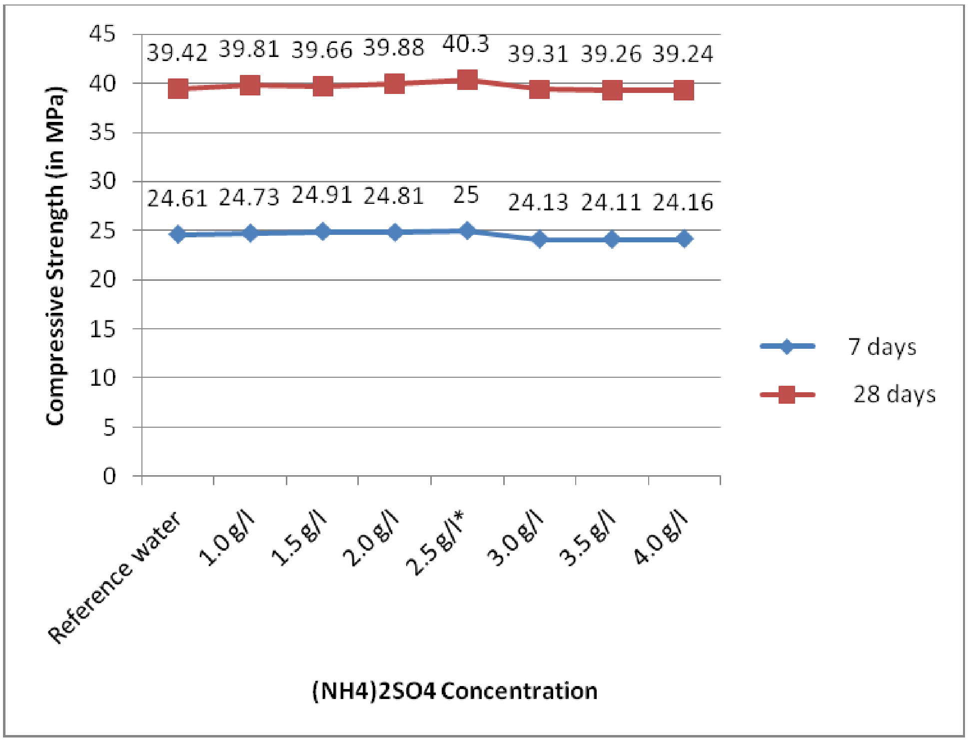
Setting time vs ammonium sulphate concentration

Compressive strength test: This test is carried out to ascertain the compressive strength of different cement mix. Test is carried out with deionised water and amonium sulphate water. Results from reference sample and sulphate altered samples are tabulated in table below and results from table are compared in the preceding line graph:

Compressive strength of FBCM corresponding to (NH4)2SO4 conentrations

Sr. No	Sample Description	Compressive strength (MPa)		Compressive strength variation (%)	
		7 days	28 days	7 days	28 days
I	Deionised water (Control)	24.61	39.42	0.00	0.00
Ii	1.0 g/l	24.73	39.81	0.48	0.98
Iii	1.5 g/l	24.91	39.66	1.21	0.60
Iv	2.0 g/l	24.81	39.88	0.81	1.16
V	**2.5 g/l***	**25.00**	**40.30**	**1.58**	**2.23**
Vi	3.0 g/l	24.13	39.31	-1.95	-0.73
Vii	3.5 g/l	24.11	39.26	-2.03	-0.40
Viii	4.0 g/l	24.16	39.24	-1.82	-0.45

*- Significant



Compressive strength vs ammonium sulphate concentration

6. Conclusion

Total three hundred twenty four samples were tested out of which one hundred eight were reference sample casted out of de-ionisd water i.e potable/pure water and rest two hundred sixteen samples were underpropped by sulphates. Different combinations were used for five sulphates under consideration. Sulphates used are- ferrous, calcium, ammonium, magnesium and sodium sulphates. The concentrations used for each sulphate is-1.0g/l, 1.5g/l, 2.0g/l, 2.5g/l, 3.0g/l, 3.5g/l and 4.0g/l. Two tests are detailed and discussed in current investigation namely setting time and compressive strength test. With the help of the obtained experimental test result, the following conclusions are drawn:

1. The initial and final setting time of conventional reference cement mix marked 84min and 262 min resp. There seen a significant increase in setting time value with increase in any of the sulphate content. When the content of these sulphates is 2.5g/l, the difference in IST and FST from the IST and FST of reference cement mix is more than 30 minutes. As per IS 456 if the increment in IST and FST is above 30minutes, then it is considered significant. So, mortars with high sulphate content mark higher values of IST and FST.
2. In a nutshell it is not wrong to say that sulphate increment resulted in explicit enhancement of setting time, thus making cement slow hardening

cement and making it compatible for hot weather concreting.

3. The compressive strength of conventional reference cement mix after 7 and 28 days marked 24.61 MPa and 39.42 MPa resp. There seen a slight increase in compressive strength value upto 2.5g/l concentration of respective sulphates and after that there seen a decline in compressive strength with further increase in sulphate content.
4. The increase in compressive strength is maximum when concentration of individual sulphate is 2.5g/l,
5. Higher the content of ferrous sulphate results in improvement in the split tensile and flexural strength of concrete structure.
6. When the content of $FeSO_4$ is 2.5g/l, the %age increase in compressive strength after 7 and 28 days are 2.19 % and 3.67 % respectively. After that if content of sulphate is increased further it results in improvement in the split tensile and flexural strength of concrete structure but reduces compressive strength.
7. When the content of $(NH_4)_2SO_4$ is 2.5g/l, the %age increase in compressive strength after 7 and 28 days are 1.58 % and 2.23 % respectively. After that if content of sulphate is increased further it results in porous structure thus reducing initial compressive strength.
8. When the content of $CaSO_4$ is 2.5g/l, the %age increase in compressive strength after 7 and 28 days are 2.31% and 2.48% respectively. After that if content of sulphate is increased further expansion and cracking increases thus reducing initial compressive strength.
9. When the content of $MgSO_4$ is 2.5g/l, the %age increase in compressive strength after 7 and 28 days are 2.07 % and 3.37% respectively. After that if content of magnesium sulphate is increased further it reduces initial compressive strength. This is due to the fact that, in this process $MgSO_4$ react with C-S-H (calcium Silicate Hydrate) and causes M-S-H (Magnesium Silicate Hydrate) which drastically is a non adhesive and harmful substance as shown in the following equation.

   $MgSO_4$ + C-S-H → M-S-H + $Ca(OH)_2$

10. When the content of Na_2SO_4 is 2.5g/l, the %age increase in compressive strength after 7 and 28 days are 2.23 % and 3.53% respectively. After that if content of sulphate is increased further expansion and cracking increases thus reducing initial compressive strength.
11. Thus to sum up the setting tine is enhanced to a significant extent, whereas there is slight increase or decline in compressive strength. Sulphate increment resulted in explicit enhancement of setting time, thus making cement slow hardening cement and making it compatible for hot weather concreting. Whereas there seen a slight increase in compressive strength value upto 2.5g/l concentration of respective sulphates and after that there seen a decline in compressive strength with further increase in compressive strength. Thus the optimum dosage of individual sulphates is 2.5g/l.

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[13] ASTM C94 –1992. Standard specification for ready-mixed concrete, American society for testing and materials, Philadelphia.

[14] American Concrete Institute(ACI) Manual of concrete Practice, Cement and Concrete Terminology- ACI 116R-78.

[15] BS 3148: 1980, Tests for water for making Concrete (including notes on the suitability of water), British Standards, London.