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Formulation of Sulphate Resistant Super Sulphated Cement Using Fluorogypsum and Granulated Blast Furnace Slag

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Abstract: *Present studies deal with the formulation of sulphate resistant super sulphated cement using flourogypsum (83 %), granulated blast furnace slag (10 %) and OPC (7%) by grinding and blending in a ball mill. The setting time and compressive strength have been determined along with chemical composition. The effect of calcium chloride and supper plasticizer on hydration of super sulphated cement show increase in the compressive strength without affecting the setting time. The maximum compressive strength using 0.75 % of calcium chloride and 2 % Metflux 2651F superplasticizer are 47.8 and 39.0 MPa respectively after 28 days of hydration. The cement also shows good sulphate resistant properties for use in aggressive environment. Major hydration products of super sulphated cement evaluated by DTA, XRD and SEM show formation of ettringite, tobermorite and C_4AH_{13} responsible for strength development in the cement. The super sulphated cement thus produced is sulpahte resistant, possesses low heat of hydration and consumes less energy than ordinay Portland cement. The cement is recommended for general concrete construction and marine structures.*
Keywords: *Super sulphated cement, OPC, granulated blast furnace slag, fluorogypsum, sulphate resistant, compressive strength, setting time*.

I. Introduction

Portland cement has become the dominant binder used in construction industry due to its versatility, durability and economic value. Portland cement concrete is receiving increasing recognition for its relatively low embodied energy compared to other materials [1]. Vast quantities of Portland cement are manufactured worldwide which consumes enormous amount of energy annually [2] results in release of approximately 0.87 tonnes of carbon dioxide for every tonne of cement production [3]. The cement industry is under pressure to reduce both energy and emission of green house gases such as CO_2, SO_2 and NOx. To meet this objective, there is a need to find the alternative materials to this binder using low energy and emitting low green house gases. Industrial byproducts like fly ash, ground granulated blast furnace slag (GBFS), silica fume etc. is used to replace about 10-50 % cement in concrete to save energy, production cost and environment [3]. There is a need to develop new binders that promise to reduce the environmental impact of construction industry using greater proportion of industrial byproducts.

Super sulphated cement (SSC) is one such binder which is sulphate resistant cement and produced by intergrinding a mixture of GBFS and gypsum anhydrite with a small quantity of Portland cement or clinker or hydrated lime. The main constituents of supersulphated cement are 80-90 % GBFS, 10-15 % anhydrite and low quantities (<5 %) of an alkaline activator mainly Portland cement or clinker [4-8]. The SSC thus produced is sulphate resistant, possesses low heat of hydration and consumes less energy than Ordinary Portland Cement.

Blast-furnace slag cement (BFSC) is used in many countries for its characteristic property of improving sulphate and chloride resistance. GBFS is a byproduct generated from the iron manufacture process and it tends to be hydrated and hardened when activated with clinker and gypsum, anhydrite, lime or plaster of Paris [9-14] with or without additional activator. In a previous work [15], it was reported that the high slag content in concrete increases sulphate resistance due to a decrease in the permeability to different types of ions and water.

Improvement in the sulphate and chloride resistance of concrete with sufficient amount of slag content is mainly due to the reduction of C_3A with the increase of slag portion in cement concrete. In addition, the addition of slag to concrete decreases the formed $Ca(OH)_2$ by reaction with slag to form the additional amounts of CSH (tobermorite), which enhances the compressive strength. These hydration products fill up the open pores, leading to the formation of a more compact body. Therefore, blended cement paste resists sulphate and chloride attack.

The suitability of a slag for use in cementitious materials depends primarily on its reactivity. Generally, slags with high CaO, MgO and especially Al_2O_3 contents are suitable for making supersulphated cement [4-6]. Taneja et al. [16] worked on replacement gypsum by phosphogypsum after calcining it at 750^oC to obtain anhydrite phosphogypsum. Mechanical properties of SSC were also studied using different phosphogypsum in different environment buy different workers [9, 17-19]. Indian slags on an average are relatively poor in lime (26-38 %) and rich in alumina (20-30 %). Activation of low lime and high alumina slag for making SSC was carried out by Dutta and Borthakur [6] and it was observed that optimum amount of anhydrite gypsum required was in the range of 15-20 %. Hydration mechanism of SSC in different slag [4] and microstructure of the SSC [10] were studied which show that main hydration products are ettringite and CSH.

In Belgium, France, Germany and South African countries SSC is extensively used for construction work. In South Africa SSC is used in sewerage underground works. SSC are attracting new attention for industrial applications [20-21], partly due to climate debate [3], but especially due to their very low heat of hydration and their good durability in chemically aggressive environments [20-21]. The super sulphated market in South Africa is about 250,000 Tonnes per annum.

There are also various repair mortar applications reported, such as rendering, injection mortars or masonry mortars [22-24], where especially the high sulfate resistance has been the main driving force to use SSC. Singh et al. [25] have shown that calcinating phosphogypsum, a waste of phosphoric industry, at 750 to 850^{o}C may produce suitable SSC. At present about 6.0 million Tonnes of by product gypsum are produced annually from several phosphoric acid and hydro-fluoric industries in India. Fluorogypsum, a waste from hydrofluoric industry contains the impurities of fluoride and free acidity which adversely affect the setting and strength development of plaster and its product. In India about 1.0 million tonne per annum of fluorogypsum is available from different hydrofluoric acid manufactuerers. In South Africa there is about 20,000 tonnes per annum consumption of fluorogypsum for making SSC.

In present studies, an attempt has been made to develop SSC by blending GBFS with alkali activated fluorogypsum received from South Africa and ordinary Portland cement. The SSC was evaluated for its physical, chemical and mechanical properties as per IS: 6909: 1990 [26]. The hydration characteristics of SSC were evaluated by differential thermal analysis (DTA), X-ray diffraction (XRD) and scanning electron microscopy (SEM). The effect of chloride and super plasticizer was also studied on physical properties along with determination of sulphate resistance of SSC.

II. Materials And Methods

The raw materials like fluorogypsum, GBFS and ordinary Portland cement were supplied by M/s Meta Dynamics, South Africa to formulate super sulphated cement.

2.1 Fluorogypsum

The sample of fluorogypsum having a fineness of 3200 cm^2/g, Blaine was dried at 42^{o}C to constant weight and cooled to ambient temperature. The sample was analysed for physio-chemical composition as per IS: 1288:1983 [27] and the results of analysis have been presented in Table 1. A perusal of Table 1 shows that fluorogypsum possesses high amount of calcium as $CaSO_4.2H_2O$ content besides presence of fluoride as a major impurity.

Table 1: Chemical composition of fluorogypsum

Chemical Composition							
CaF_2	SiO_2 + insoluble residue	$Al_2O_3 + Fe_2O_3$	CaO	MgO	SO_3	LOI	pH
1.98%	0.58%	0.65%	42.2%	0.05%	55.1%	0.31	8.2

2.2 Granulated blast furnace slag (GBFS)

The sample of GBFS having fineness of 4500 cm^2/g, Blaine was analysed as per IS: 4032:1985 [28] for chemical composition and values are shown in Table 2. A perusal of Table 2 shows that slag is rich in silica and calcium content which are 36.8 and 34.9 % respectively. The chemical composition of slag complied with IS: 12089:1987 [29] for the manufacture of Portland slag cement.

2.3. Cement

Ordinary Portland Cement (OPC) of 43 Grade having fineness of 4500 cm^2/g, Blaine and specific gravity of 3.14 was utilized for development of SSC. The initial and final setting times were 240 and 360 min. The mechanical properties shows that the 3, 7 and 28 days compressive strength were 33, 49 and 61 MPa respectively determined as per IS:4031:1999 [30]. The chemical composition of OPC analyzed as per IS: 4032: 1985 [28] has been shown in Table 3.

Table 2: Chemical composition of granulated blast furnace slag

Chemical Composition (%)								
CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O+ Na_2O	MnO	Insoluble residue
34.9	36.8	16.5	0.36	0.37	7.46	1.70	0.85	0.039

Table 3: Composition of ordinary Portland cement

Chemical Composition (%)								
CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	K_2O	Na_2O	LOI
62.62	24.20	3.39	3.20	1.80	3.21	1.15	0.51	0.45

2.4 Formulation of super sulphated cement

The samples of fluorogypsum and GBFS were grinded separately in a ball mill to fineness of 4500 cm^2/g, Blaine and blended with OPC in different proportions as shown in Table 4. The final fineness of SSC was 4500 cm^2/g, Blaine conforming to the IS: 6909 [26] having minimum requirement of 3000 cm^2/g, Blaine. The SSC mixes thus prepared were tested for physical properties like setting time and compressive strength as per IS: 4031 [30]. To determine the compressive strength, super sulphated cement and Ennore standard sand were used in 1:3 ratio and cubes of 50 cm^3 were cast by vibrating method as per IS: 650:1970 [31]. Water was taken equivalent to (P/4+3) % where p is the consistency of the cement. The cubes were demoulded after 24 h and cured in water at 27 ± 2^oC and compressive strength were determined on 3, 7 and 28 days of hydration. The physical and chemical properties of final SSC mix showing best performance were determined as per IS 4031 and IS: 4032 respectively and selected for further investigations. To study the effect of calcium chloride and super plasticizer 50 cm^3 cubes were prepared using the vibrating method and tested.

Table 4: Composition of mixes for super sulphated cement

Mixes	Fluorogypsum (%)	Granulated blast furnace slag (%)	OPC (%)
SSC1	15	80	5
SSC2	10	85	5
SSC3	13	82	5
SSC4	10	83	7
SSC5	10	80	10

2.5 Effect of Calcium chloride ($CaCl_2$)

Effect of various concentration of $CaCl_2$ (0.50 to 2.0 %) was studied on setting time, compressive strength (3, 7 and 28 days) and soundness of super sulphated cement of final mix as per IS:4031. The soundness was determined using Le Chatlier method.

2.6 Determination of sulphate resistance

The sulphate resistance of final mix for SSC was determined as per method described in IS: 12330:1998 [32]. A mixture of SSC and natural gypsum was prepared to achieve total SO_3 content 7.0 % by mass. The fineness of natural gypsum was 100 % passing from 150 µm IS sieve and minimum 75 % passing from 45 µm IS sieve. Bars of size 250 mm x 25 mm x 25 mm were cast using the prepared mix and sand taken in the proportion 1:2.75 at a w/c ratio of 0.485. The bars were demoulded after 24 h and immersed in water horizontally for curing. The average expansion of three bars was recorded after 14 days of curing.

2.7 Effect of super plasticizer on hydration of SSC

To study the effect of super plasticizer on the setting time and compressive strength of hydrated super sulphated cement, super plasticizer Metflux 2651F received from M/s. Meta Dynamics was used as water reducer. The effect of different concentrations 0.075, 0.20 and 0.30 % of Melflux 2651F was studied on the final mix of SSC.

2.8 Heat of hydration

The heat of hydration is the quantity of heat in calories per gram of unhydrated cement, evolved upon complete hydration at a given temperature. It is largely influenced by the mineralogical composition of cement. The heat of hydration of final mix of SSC was determined as per method given in IS: 4031 [30].

2.9 Hydration of super sulphated cement

Strength development and identification of hydration products in SSC of final mix were evaluated by differential thermal analysis (DTA Perkin Elmer Diamond model), X-ray diffraction (Rigaku, Japan) and SEM (LEO 438 VP, UK). Hydrated samples were immersed in acetone to stop hydration and grounded to a particle size of <45 µm for this purpose. X-ray diffraction was recorded on an X-ray Diffractometer equipped with a rotating anode, utilizing CuK_a radiation at 40 kV and 30 mA.

III. Results And Discussion

3.1. Properties of Super sulphated cement

The results of physical properties like setting time and compressive strength of SSC mixes are shown in Table 5 along with limits mentioned in IS:6909, specification for super sulphated cement. . A perusal of Table 5 depicts that setting times of all SSC mixes and 28 days compressive strength conformed to IS: 6909. It is observed that with increase in curing period, compressive strength increases. However, the SSC4 mix shows best performance as compared to other SSC mixes formulated and conform to IS; 6909 for early strength (3 and

7 days) as well as in delayed strength (28 days). Therefore, SSC4 mix was finalized and selected for further studies. The chemical composition of SSC4 is shown in Table 6.

Table 5: Physical properties of super sulphated cement mixes

Mixes	Setting time(min)		Compressive strength (MPa)		
	Initial	Final	3 days	7 days	28 days
SSC1	275	415	10.9	19.3	31.1
SSC2	263	380	12.1	18.5	30.0
SSC3	280	378	13.8	19.8	31.2
SSC4	270	350	15.5	22.6	32.0
SSC5	273	355	14.8	20.2	30.0
IS:6909:1990 limits	Not less than 30	not more than 600	15.0	22.0	30.0

Table 6: Chemical composition of super sulphated cement (SSC4)

Chemical Composition (%)								
CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	S	Insoluble residue	LOI
46.2	28.35	11.20	0.20	6.20	3.98	0.85	2.80	0.80

3.2 Effect of Calcium chloride ($CaCl_2$)

The effect of different concentration of anhydrous calcium chloride on the properties of SSC4 mix has been studied and results are shown in Table 7. A perusal of Table 7 shows with the addition of calcium chloride, improvement in compressive strength has been observed at all ages. Maximum strength has been observed on 28 day of curing at a calcium chloride concentration of 0.75 % and on further increase in $CaCl_2$ concentration, the strength almost remains constant. Therefore, $CaCl_2$ may be considered as strength enhancer or strength activator for super sulphated cement (SSC4). Moreover, the chloride concentration calculated in the cement has been recorded within the limit (<0.45 %) determined by IS: 12423: 1988 [33].

3.3 Determination of sulphate resistance

The results of sulphate resistant test conducted for SSC4 mix show the average expansion of 0.023 % after 14 days against the maximum specified value of 0.045 % as per IS:12330. It is relatively higher sulphate resistant vis-à-vis Portland cement making it more suitable for use in marine structures and for construction of sewage pipes.

3.4 Effect of super plasticizer on hydration of SSC

The results of different concentrations of super plasticizer on setting time and compressive strength of SSC4 are shown in Table 8. It has been observed that the setting times are not much affected by variation of Metflux 2651F concentrations. Further, the results show that on increase in Metflux 2651F concentration from 0.075 to 0.3 %, the compressive strength increases. However, the strength is comparable for 0.2 and 0.3 % of super plasticizer concentration which is 39.0 and 38.8 MPa respectively.

Table 7: Effect of calcium chloride on physical properties of super sulphated cement (SSC4)

Calcium chloride (%)	Setting time(min)		Compressive strength (MPa)			Soundness (mm)
	Initial	Final	3 days	7 days	28 days	
0	270	350	15.5	22.6	32.0	1.70
0.5	205	315	22.0	29.3	38.0	1.65
0.75	221	302	22.7	32.3	47.7	1.68
1.25	230	296	20.0	29.2	48.0	1.69
2.0	185	301	19.0	31.8	47.5	1.71
IS:6909:1990 limits	Not less than 30	not more than 600	15.0	22.0	30.0	5.0

Table 8: Effect of super plasticizer on physical properties of super sulphated cement (SSC4)

Super plasticizer (%)	Setting time(min)		Compressive strength (MPa)		
	Initial	Final	3 days	7 days	28 days
0	270	350	15.5	22.6	32.0
0.075	331	361	18.5	23.0	32.2
0.20	334	368	20.3	29.5	39.0
0.30	340	370	20.7	30.0	39.8
IS:6909:1990 limits	Not less than 30	not more than 600	15.0	22.0	30.0

3.5 Heat of hydration

Table 9 summarizes the heat of hydration of super sulphated cement (SSC4) and it is observed that the mix SSC4 complies with the specification of IS:6909 which specify a maximum limit of 60 and 70 cal/g heat of hydration at 7 and 28 days respectively. Due to low heat of hydration, SSC is eminently suitable for mass concrete construction.

Table 9: Heat of hydration of super sulphated cement (SSC4)

Heat of dissolution (cal/g)				Heat of hydration (cal/g)		
Unhydrated SSC	hydrated SSC					
	3days	7 days	28days	3days	7 days	28days
532.2	568.0	577.4	592.7	35.8	45.2	60.5
IS: 6909:1990 limits					Max. 60.0	Max. 70.0

3.6 Hydration of super sulphated cement

Strength development in super sulphated cement (SSC4) was studied by DTA, XRD and SEM. The thermograms of SSC4 at 3, 7 and 28 days of curing shown in Fig. 1 indicate that endotherms at 155^o, 180^o, 230^o, 500^o, 700-800^{o}C and exotherms at 845-920^{o}C which may be assigned to the formation of ettringite phase, dehydration of calcium sulphate hemihydrate and calcium aluminate hydrate (C_4AH_{13}). DTA does not show the evidence of direct formation of CSH gel, however, an unexplained endotherm at 700^{o}C at 7 and 28 days of hydration may be the CSH gel. Another way to explain the non appearance of CSH could be that it has intermingled with the intensity of ettringite phase. The high intensity of ettringite is responsible for strength development with time.

The XRD of super sulphated cement at 3, 7 and 28 days of hydration are shown in Fig. 2 which indicates that major peaks are ettringite (E), tobermorite (T) and calcium aluminate hydrate (CAH). The intensity of ettringite and tobermorite peaks increase with increase in curing period and are responsible for strength development. However, the intensity of gypsum (G) and calcium hydroxide (CH) are reduced with time.

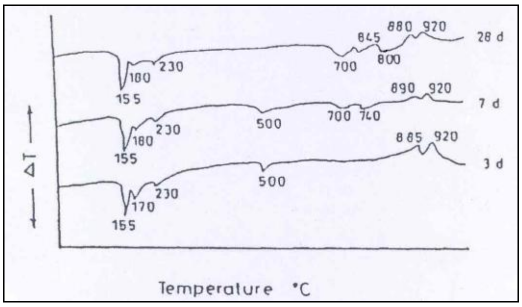

Fig. 1: DTA of hydrated SSC after 28 days

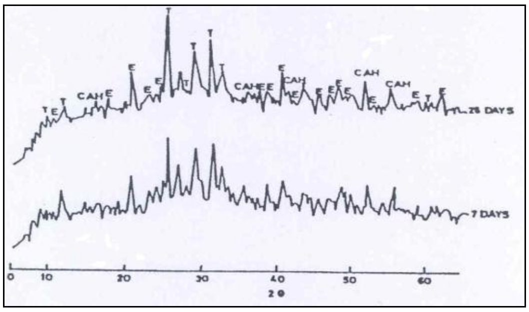

Fig.2: XRD of hydrated SSC after 28 days

The hydration studies were supplemented by the scanning electron microscopy (SEM) of SSC at 7 and 28 days as shown in Fig. 3 and 4 respectively. The microstructures show anhedral to subhedral fibrous and prismatic crystals of variable sizes and staking indicating the formation of ettringite and platy agglomerates of CSH bodies. These findings corroborate the results of DTA and XRD and confirm prove the results of earlier workers [10].

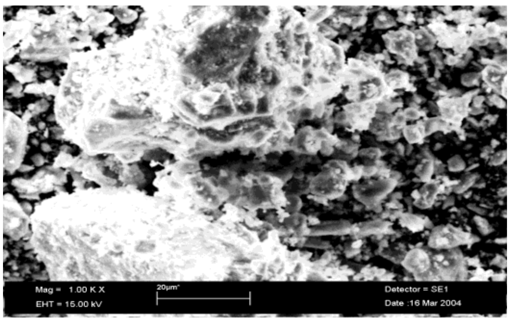

Fig. 3: SEM of SSC after 7 days of hydration

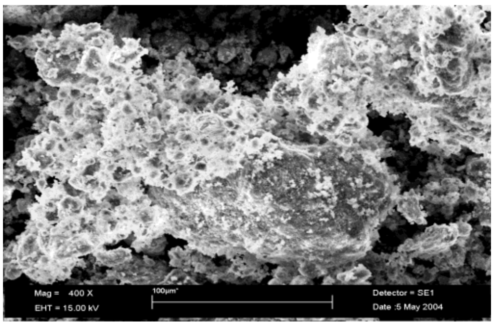

Fig. 4: SEM of SSC after 28 days of hydration

IV. Conclusions

The Following conclusions can be drawn from the work carried out on formulation of sulphate resistant cement using raw materials like fluorogypsum, granulated blast furnace slag and OPC:

1. The super sulphated cement can be produced from the fluorogypsum, GBFS and OPC by grinding and blending it in suitable composition. Out of several mix tried, the mix having 83 % of fluorogypsum, 10 % of GBFS and 7 % of OPC shows best performance. The physical and chemical properties comply with the specification of IS: 6909.
2. The effect of various concentration of anhydrous calcium chloride shows enhancement in strength and a concentration of 0.75 % is found to be optimum. Therefore calcium chloride may be used as strength activator.
3. The sulphate resistant of SSC is 0.023 % showing better durability characteristics in marine environment.
4. Mixing of super plasticizer Metflux 2651F also increases the compressive strength without affecting the setting time of SSC.
5. The heat of hydration determined at 7 and 28 days of SSC is low and comply with the requirements of IS: 6909. It may be recommended use for mass concrete formation.
6. The DTA, XRD and SEM studies confirm that strength development in hydrated SSC is due to formation of
7. ettringite, tobermorire and C_4AH_{13}.

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