MORPHOLOGICAL SCRUTINY OF CEMENT MATRIX IN IMPROVISED CONCRETE INFUSED WITH SILICA FUME AND CEMENT SLUDGE

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Abstract- Improvising performance of concrete mix by infusing waste by products has become the order of the day in sight of expediting material substitution coupled with environmental protection. The technical feasibility and degree of performance by an improvised cement matrix impregnated with silica fume and cement sludge was test verified at selected optimum proportions of cement replacement. As relatively new material for partial substitution with cement, the optimal percentage of replacement by the waste cement sludge was frozen at 4%. As the usage of silica fume is already in vogue its optimal percentage proportion in tandem with the initialized 4% cement sludge has been experimented with 8% silica fume. The experimental results could be fortified by an empirical regression modelling approach. The morphological changes within the cement matrix of improvised concrete mix were exemplified by a SEM analysis.

Keywords: Cement matrix, waste cement sludge, Silica fume, industrial by-product, Improvised concrete

1 INTRODUCTION

Owing to rapid industrialization, and geometrically progressing population, the accumulation of waste growths every year [1-3,7,9]. Waste disposal becomes a influential environmental concern [5,6]. Waste management by recycling is the primary issue contemplated by the developing countries throughout the world, in order to transform the wastes polluting the environment into beneficial by products that are compatible to the ecology without causing a threat in the form of pollution or contamination of the atmosphere or lithosphere or the hydrosphere [4,8,10-14]. Ready mix concrete (RMC) has more benefits in large quantity of concrete production but the disposal of the sludge that is generated from cleaning activities become a problem. Cleaning process of batching plant, concrete trucks, concrete mixing drums and others, enormous quantity of cement sludge has been produced [20,24-26].

Due to higher pH value, unsafe disposal of sludge becomes hazardous to the environment [21,23]. Disposal of sludge in land surface will change the soil properties that may pollute ground water and cause land pollution [15-19]. In most of the cases cement sludge is dumped either at construction sites, or at landfills, sometimes in open yards [30,31].

Many of the sludge cleareance methods are not environmentally compatible. Therefore, recycling of sludge becomes obligatory now-a-days. If sludge reused as an ingredient in concrete, it has economic benefit because it is treatment free. Silica fume also used in this study along with cement sludge. Silica fume highly pozzolanic material from ferrosilicon industry [28]. It may enhance the properties of concrete. It can be utilized as a constituent or blended with portland cement in concrete[22,27, 29].

2 EXPERIMENTAL PROGRAMME

2.1 Materials

(1) Cement : OPC of grade 53 was used throughout the study to prepare the concrete specimens.

(2) Cement Sludge: Cement sludge produced during cleaning process was collected and Owen dried at 110°C temperature.

(3) Silica fume: Silica fume was collected from Magna ferrosilicon industry Chennai. Cement was partially substituted with Silica fume.

Properties of silicafume, dried cement sludge and Portland cement were furnished in Table.1.

Table 1 dried cement sludge and Portland cement

Description	OPC	Dried	Silicafume
1		Sludge	
Specific	3.15	2.62	2.39
Gravity			
CaO	62.11	36.62	0.55
Fe_2O_3	3.58	2.74	0.92
Na ₂ O	0.22	0.29	0.51
SiO ₂	23.61	11.95	91.3
Al_2O_3	6.02	5.9	2.6
MgO	1.49	6.42	0.82
K ₂ O	0.24	0.91	
LOI	0.91	24.88	2.93
Free CaO	0.54	0.31	

(4) Aggregates: Natural sand Crushed stone of size 20 mm was used as fine and coarse aggregate in concrete.(5) Potable water was used to prepare mixes.

2.2 Concrete Mixes

Conventional and two improvised categories of mixes (TS and TSF) were prepared. Where, the treatment category TS stands for treatments involving cement added with dried cement sludge powder and TSF stands for treatments involving cement added with frozen proportion of dried cement sludge powder at 4% and silica fume. Concrete specimens in accordance with these categories were cast and subjected to standard tests for assessing the mechanical properties of concrete and corrosion resistance of the steel embedded in the corrosion of steel for both mixes. The test results were investigated with the performance of conventional concrete designated as M_{40} .

Keeping the conventional concrete mix as the control for comparison the following designations the following designations were given to the treatments belonging to the categories namely cement with dried sludge (TS) and cement with dried cement sludge + silica fume (TSF).Treatments under the category TS were shown in table.2

Table 2 Furnishes the various treatments under the category of TS

Designation	Description
TS-2	2% dried cement sludge with cement
TS-4	4% dried cement sludge with cement
TS-6	6% dried cement sludge with cement
TS-8	8% dried cement sludge with cement
TS-10	10% dried cement sludge with cement

By the same token the treatments under category of TSF were shown in table.3

Table 3 Furnishes the various treatments under the category of TSF

Designation	Description	
TSF-3	3% silica fume + 4% dried cement	
	sludge	
TSF-6	6% silica fume + 4% dried cement	
	sludge	
TSF-8	8% silica fume + 4% dried cement	
	sludge	
TSF-10	10% silica fume + 4% dried cement	
	sludge	
TSF-12	12% silica fume + 4% dried cement	
	sludge	

2.3 Test Methods

The mechanical properties of the conventional concrete and TS, TSF category of concretes were determined by appropriate tests. $100 \times 100 \times 100$ mm size specimens were subjected to compressive strength test for 3, 7, 28 and 56 days curing. For determination of flexural strength $100 \times 100 \times 500$ mm size of concrete beams were cast and tested after 7, and 28 days of curing for both series and compared with conventional concrete. 100×200 mm size steel embedded concrete cylinders were cast to find corrosion resistance of steel.

In order to determine the corrosion rate of reinforcement, concrete samples were submerged in 3.5% NaCl and reinforcement was connected with 12 Volt DC supply. The rate of corrosion was determined by obtained potential difference. The morphology of cement matrix during hydration was studied using scanning electron microscopy (SEM).

3 RESULTS AND DISCUSSIONS

Morphological stimulus of impregnating Silica fume and cement sludge at an optimal replacement proportion for cement was investigated.

3.1 Freezing the Optimum Mix Proportion of Dried Cement Sludge in the Concrete Mix

To identify the latent usage of dried cement sludge in concrete the optimum replacement percentage was identified and frozen. TS category of mixes showed higher compressive strength as well as flexural strength was observed up to 4% partial substitution of dried sludge by weight of cement for the given mix grade of M₄₀. Strength decrease was noted at replacement percentages of 6, 8 and 10%. By trial and error 4% was frozen as an optimum replacement percentage of dried cement sludge for cement in concrete. The experimental results construed from the experimentation were modelled with simple linear regression analysis using the Principle of the Least Squares and the best fitting lines indicated the reliability of the relationships at the respective correlation levels for the independent variable viz., percentage of dried sludge content in cement matrix and the corresponding model dependent variables viz., the compressive strength, flexural strength and the corrosion rates respectively. Compressive and flexural strength values of TS category of mixes for different curing ages were shown in following figures[1-13].

3.2 Regression Model on Compressive Strength Vs Various % Proportions of Dried Cement Sludge Incorporation in Cement Matrix

The simple linear regression models for different combination percentages of dried cement sludge with cement are furnished as follows.



Figure 1 Compressive strength values of TS category of mix after 3 days of curing

In harmony with empirical model, compressive strength value of control mix after 3 days of curing was found to be 19.23 N/mm² and for the specimens TS-2, TS-4, TS-6, TS-8 and TS-10 the compressive strength was found to be 20.03, 19.36, 17.96, 15.83 and 14.6 N/mm² respectively. An increase in compressive strength of 0.68% than control was noted in the mix TS-4 after 3 days curing at a dependable correlation of 97%.



Figure 2 Compressive strength values of TS category of mix after 7 days of curing.

Escalating strength found in 7 days Compressive strength of the specimens TS-2 and TS-4. This shows that the sludge replacement up to 4% can be preferable. The compressive strength values of mix TS-2, TS-4, TS-6, TS-8 and TS-10 was found to be 27.26, 26.96, 25.03,

22.96 and 21.83 N/mm² respectively and the compressive strength value of control concrete was 26.83 N/mm² at a dependable correlation of 96% of sample data analyzed.



Figure 3 Compressive strength values of TS category of mix at 28 days.

In case of 28 days compressive strength concrete without any trace of substitute materials got 40.7 N/mm². At 2% and 4% dried sludge replacement levels slight increase in compressive strength was noted. The 28 days compressive strength values of mix TS-2, TS-4, TS-6, TS-8 and TS-10 was found to be 41.13, 40.93, 39.73, 36.43 and 33.9 N/mm² respectively. It was scrutinized that beyond 4% sludge replacement a dip in compressive strength was observed. Compressive strength increase of 0.56% than control concrete was observed in TS-4 specimens and 90 % correlation was obtained from sample data analyzed.



Figure 4 Compressive strength values of TS category of mix at 56 days.

It was observed that 56 days curing compressive strength of TS-series specimens slightly increased at the frozen value of 4% dried cement sludge replacement level without detrimental to the quality of conventional concrete. At 96% dependability of data the compressive strength of control concrete at 56 days of curing was reckoned at 42.03 N/mm² whereas for the specimens TS-2, TS-4, TS-6, TS-8 and TS-10 the same was found to be 42.96, 42.16, 40.1, 37.03 and 34.13 N/mm² respectively. Concrete specimens of mix TS-4 apparently gained 0.30% higher compressive strength than control concrete.

3.3 Regression Model on Flexural Strength Vs % of Dried Cement Sludge

Flexural strength test was conducted for 2 different time lapses namely 7 days and 28 days. The flexural strength of the material substituted concrete mix (Dried cement sludge) depends on the percentage proportion with cement as a control standard for comparison with the conventional concrete mix. The flexural strength upon plotting against the cement sludge replacement proportion indicates that up to 4% dried cement sludge addition the flexural strength has shown an ascending trend but afterwards suddenly started showing a dip for any further addition of dried cement sludge. The relative percentage increase in flexural strength at 4% addition of dried cement sludge after 7 and 28 days of curing are 0.42% and 0.46% respectively. Hike in flexural strength is due to proliferation of fines in sludge that fill the pore present in cement matrix and restrict the propagation of cracks thereby withstand more load.



Figure 5 Flexural strength values of TS category of concrete after 7 days of curing

The increase in percentage of dried sludge content decreased the flexural strength of the mix TS-6, TS-8 and TS-10. In case of mix TS-2 and TS-4, flexural strength is slightly increased with respect to control concrete. 7 days Flexural strength of mix TS-2, TS-4, TS-6, TS-8 and TS-10 was found to be 4.87, 4.79, 4.59, 4.13 and 3.81 N/mm2 respectively and for control concretes reckoned as 4.77 N/mm2. Mix TS-4 gained 0.42% higher flexural strength than control concrete. The R2 value shows 93.9% reliability.



Figure 6 Flexural strength values of TS category of mix at 28 days

Flexural strength of specimens TS-2, TS-4, TS-6, TS-8 and TS-10 was found to be 6.63, 6.54, 6.36, 5.97 and 5.81 N/mm2 respectively and the control concrete obtained flexural strength value of 6.51 N/mm2. Similar trend was observed in 28 days flexural strength as 7 days. Except mixes TS-2 and TS-4 all other mixes showed a waning trend in rate of flexural strength gain. Mix TS-4 gained 0.46% higher flexural strength at 28 days than control concrete. From the co-efficient of determination value the predicted values are 95.4% accurate to the actual one. The reduction in strength may be due to leaching of lime and less CaO content in sludge. Presence of carbon particles is porous in nature which lowers the strength.

3.4 Silica Fume Addition with Frozen Proportion of Dried Cement Sludge in the Concrete Mix

In the second category i.e TSF, the properties of concrete were enhanced by adding silica fume with the dried sludge percentage frozen at 4%. Silica fume particles are 100 times smaller than cement particles which facilitate filling the minute pores of concrete. Due to nuclei present in cement significantly improves the hydration. C-S-H gel formation is restricted in pore spaces of control concrete but in silica fume concrete, C-S-H gel form onto the micro particles of silica fume and spread on pores of concrete. C-S-H gel formation enriches the bonding between and improved the properties of concrete.

3.5 Regression Model on Compressive Strength Vs 4% of Dried Cement Sludge + Silica Fume Incorporation

From the test observations compressive strength of TSF category mix was higher than TS category and conventional concrete mix up to 8% replacement of silica fume with 4% initialized dried sludge powder. Compressive strength of TSF category of mixes was computed with respect to control concrete. Compressive strength of improvised concrete for replacement proportion of 8% silica fume with 4% dried sludge powder at 3 days stands at 22.03 N/mm2 whereas the control concrete attained compressive strength of 19.23 N/mm²



Figure 7 Compressive strength values of TSF category of mix at 3 days.

Compressive strength of mixes TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 at 3 days was found to be 20.66, 21.33, 22.03, 19.86 and 18.6 N/mm2 respectively. Mix TSF-8 gained 14.56%, 13.79% higher compressive strength than control and TS-4 category of concrete after 3 days of curing.



Figure 8 Compressive strength values of TSF category of mix at 7 days.

As like 3 days compressive strength same trend was observed in 7 days compressive strength. Specimens TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 after 7 days of curing gained compressive strength of 27.53, 28.26, 29.13, 27.36 and 25.43 N/mm2 respectively. Peak compressive strength was observed for the mix TSF-8. The compressive strength increase of 8.57% than control concrete and 8.04% than the mix TSF-8 was computed

and correlation was obtained with 28.2% confidence level.

The relative increase in compressive strength for the mixes TSF-3, TSF-6 and TSF-8 was observed at 28 days. At 28 days, mix TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 attained compressive strength of 41.86, 43.93, 44.76, 42.9 and 40.96 N/mm2 respectively. The optimum strength was observed for the mix TSF-8. The strength increase of 9.98% than control concrete and 9.36% than mix TS-4 was observed in improvised concrete.



Figure 9 Compressive strength values of TSF category of of mix at 28 days.

TSF-8. Strength improvement is due to the pozzolanic activity. The 56 days compressive strength of specimens TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 was found to be 44.1, 45.43, 46.16, 43.83 and 41.9 N/mm2 respectively. Improvised concrete additionally gained the compressive strength of 9.83% and 9.49% than control concrete and mix TS-4 respectively and the samples data were analyzed at dependable correlation of 26.6%.



Figure 10 Compressive strength values of TSF category of concrete after 56 days of curing

3.6 Regression Model on flexural Strength Vs 4% of dried cement sludge + Silica fume incorporation

The variations in flexural strength of silica fume and 4% dried cement sludge added concrete is shown in Fig. 11. Rising trend in flexural strength was observed for the mixes TSF-3, TSF-6 and TSF-8, for other mixes declining trend was observed. The 7 days flexural strength of specimens of mix TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 was found to be 4.98, 5.26, 5.52, 4.76 and 4.8 N/mm² respectively. The percentage gain in flexural strength for the mix TSF-8 with respect to control and dried sludge impregnated concrete was 15.72% and 15.24% at 7 days.



Figure 11 Flexural strength values of TSF category of mix at 7 days

Silica fume addition significantly contributed in improvement of flexural strength. The 28 days flexural strength of specimens of mix TSF-3, TSF-6, TSF-8, TSF-10 and TSF-12 was found to be 6.92, 7.26, 7.48, 6.7 and 6.48 N/mm² respectively. The flexural strengths almost follow the same trend as the compressive strength of improvised concrete does. The optimum silica fume replacement percentage with 4% dried sludge was found to be 8%. For the mix TSF-8, at 28 days, the flexural strength increase of 14.9% and 14.37% with respect to control and dried sludge impregnated concrete was observed. The co-efficient of determination from the curve fit equation developed for 7 and 28 days flexural strength prediction is 0.416 and 0.413 respectively.



Figure 12 Flexural strength values of TSF category of mix at 28 days.

3.7 Corrosion rate for steel embedded in concrete mix

Figure.13 shows the variations of rate of corrosion induced in steel embedded with control, TS-4 and TSF-8 category concrete. The rate of corrosion in TS-4 category concrete is higher than control concrete. The main reason may be due to the porous nature of TS-4 concrete mix. Which will increases chloride penetration through pores, results in higher rate of corrosion. In case of TSF-8 mix the rate of corrosion induced in embedded steel with respect to control mix was less. Addition of slica fume improved the corrosion resistance. Micro particles of silica fume proliferates through the pores of concrete and reduces the porosity of concrete and forms a denser micro structure thereby reduces the rate of corrosion ofsteel.



Figure 13 Rate of corrosion in control, TS-4 and TSF-8 category concrete

3.8 Scanning Electron Microscopy (SEM) analysis of the cement matrix

From the SEM image it was observed that a needle like structure called ettringite (3CaO.AI2O3.3CaSO4.32H2O) is present in the cement matrix along with Calcium Hydroxide (CH) and Cement sludge. Ettringite is a hydrous calcium alumino sulfate mineral.



Figure 14 SEM of fracture surface at 10,000 time magnification of improvised Concrete after 28 days of hydration.

The formation of ettringite in cement matrix is due to the reaction of calcium aluminate with calcium sulfate. Higher replacement of cement sludge may generate excess ettringite in cement matrix which affect the bonding and reduced the compressive strength. Calcium silica hydrated gel (C-S-H) formation is intended to enhance the bonding between the ingredients of the improvised concrete matrix.

4 CONCLUSION

The test verification upon the feasibility of impregnating silica fume with cement sludge has proved the morphological performance on par with control concrete at 4% replacement of dried cement sludge with 8% of silica fume. Replacement of cement sludge alone increased the porosity of concrete. Impregnation of dried sludge along with silica fume has been improved the performance of cement matrix and quality of concrete. Besides reutilization of waste by product as cement replacement material in concrete is a pragmatic solution to reduce the cost in totting up with environmental protection from the waste dumping.

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