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Experimental Studies on Fly Ash Gypsum Slurry in Aggressive Environment

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Abstract - This study investigates the properties of fly ash gypsum slurry with quarry waste in aggressive environment. The mix was adjusted for 400±25mm flow, including and excluding super plasticizer. The research focuses on a variety of factors, including wetting and drying resistance, sulphate resistance, salt resistance, and water absorption. For the purpose of accelerating up laboratory testing, specimens were soaked in 3% magnesium sulphate for sulphate attack 3.5% sodium chloride solution for salt attack. By adding super plasticizer, fly ash gypsum slurry with quarry waste requires 17% less water, resulting in a 400±25mm spread of flow. Following 28 days of curing, six cycle wetting and drying tests were performed. The findings of the investigation show that adding super plasticizer improves the durability of fly ash gypsum slurry. This enhancement is observed through the evaluation of mass loss and compressive strength in specimens exposed to different environmental conditions over a 28-day curing period.

***Keywords*:** fly ash gypsum slurry, quarry waste, aggressive environment, wetting and drying resistance, sulphate resistance, salt resistance, and water absorption.

1. Introduction

Flowable slurry is a flexible and ecologically friendly building material emphasized for its self-compacting properties. This cementitious material can be used in a highly fluid form, which has various advantages, notably in the large-scale usage of industrial byproducts. Its unique properties, falling between traditional concrete and soil, make it suitable for a wide range of building applications. The American Concrete Institute (ACI) 229 R, 2022, emphasizes the need of evaluating the qualities of flowable slurry as a guideline for its development and implementation in building projects [1].

Willam C. Krell (1989) examined the compressive strength, erosion test, permeability, segregation, and subsidence of flowable fly ash utilising class F fly ash and 4 to 5% Portland cement to construct ramps or embankments as structural backfill. The findings would be valuable for engineers and researchers looking for long-term and effective solutions to civil engineering projects [2].

Tarun R. Naik et al. (1990). Bruce and W. Ramme (1990) investigated the compressive strength and plastic characteristics of flowable fly ash in a variety of scenarios, including backfilling, filling abandoned subsurface facilities, and excavation [3-4]. Tarun Naik (1997) investigated the compressive strength and plastic characteristics of slurry made from class F fly ash, clean sand, and used foundry sand. The use of diverse supplementary materials, such as FGD ash, cement, lime, and admixtures, illustrates the variety of techniques in generating flowable materials adapted for individual building demands [5-6]. Revathi et al. (2005) investigated both fresh slurry and hardened mechanical characteristics of gypsum activated Class C fly ash [7].

Christine and her associates1998 using pond ash, Pierce et.al 2002 using crumb rubber as aggregate, Do & Kim using wood ash investigated the hardened and plastic properties of flowable slurry [8-10]. J.P.Won 2004, studied durability studies such as wetting and drying, freezing and thawing resistance, permeability using bottom ash [11].

Slurry that is flowable is usually not designed to resist aggressive chemicals, erosive forces, or water and heat [12] (Bruce.W.Ramme, 1997). Most often, flowable slurry is used for non-structural purposes such as the filling of holes in the ground or trenches. The durability requirements for flowable slurry depend on the specific application. According to Brewer, durability considerations depend on the intended use of the slurry. Consequently, flowable slurry performance expectations can vary depending on the context in which it is used [13]

If flowable slurry is used for the pavement base layer, freezing and thawing must be addressed. Erosion resistance is necessary to safeguard the embankment and culverts. Corrosion resistance is necessary when using flowable slurry as a pipe bedding material. When flowable slurry is utilised as a high-quality base material for foundations and road bases, it must be able to tolerate weather changes and the external chemical environment. In the presence of free weathering, particularly frequent changes in moisture and temperature as well as influences caused by external chemical environments (from water and soil), flowable slurry will have a dramatically reduced service life.

Despite the fact that a lot of study has been done on the use of flowable slurry, there have been few studies on its durability. As a result, the current study focuses on properties such as wetting and drying resistance, sulphate resistance, salt resistance, and water absorption. The current investigation involved laboratory exposures to fly ash gypsum slurry containing quarry waste with and without superplasticizer.

2. Materials Used

2.1. Fly ash

This study employed fly ash collected from Neyveli Lignite Corporation (NLC). The physical and chemical characteristics are reported in Tables 1 and 2. It has been claimed that Neyveli fly ash contains a greater amount of CaO (14%), making it more reactive than other fly ash.

Table 1: Physical properties of fly ash.

Test Conducted	Specific gravity	Setting time (min)		Consistency %	Blain's Fineness, cm^2/g	Lime Reactivity, MPa
		Initial Set	Final Set			
Observed Values	2.47	45	280	35%	3550	7.1
Required as per IS 1320-1981	N/A	N/A	N/A	N/A	2500 to 3200	3 to 4

Table 2: Chemical properties of fly ash.

Test Conducted	Loss on Ignition	Silicon Dioxide (SiO_2)	Ferric Oxide (Fe_2O_3)	Aluminum Oxide (Al_2O_3)	Calcium Oxide (CaO)	Magnesium Oxide (MgO)	Sulfur Trioxide (SO_3)	Chloride (Cl)
Observed Values	3.74%	35.87%	4.00%	34.14%	14.25%	3.64%	3.4%	0
Required as per IS 1320 - 1981	12% max	35% max	N/A	24.09%	N/A	5% max	2.75%max	N/A

2.2. Gypsum

The gypsum utilised in this investigation was obtained from TANFAC Cuddalore. TANFAC is a fluoride industry. Gypsum is warmed to 200°C before disposal. Partially burned gypsum is recovered in powder form. The $CaSO_4$ percentage of the aforementioned gypsum is around 94%. The specific gravity was measured to be 2.70. The chemical characteristics of gypsum are shown in Table 3.

Table 3: Chemical properties of gypsum.

Loss on Ignition	Insoluble Residue (IR)	Ferric Oxide (Fe_2O_3)	Aluminum Oxide (Al_2O_3)	Calcium Oxide (CaO)	Magnesium oxide (MgO)	Sulfur Trioxide (SO_3)	Calcium Fluoride (CaF_2)
1.63	0.5	0.82	0.75	44.99	1.02	48.8	0.3

2.3. Quarry Waste

This study applied quarry waste with a dust fraction that passed through a 4.75mm filter and was retained on a 300 μm screen. Organic matters have been avoided with care. The fineness modulus of rock dust in its natural condition was determined to be 1.53. The specific gravity was measured to be 2.75.

2.4. Super Plasticizer

This study hired the superplasticizer CONPLAST 430, which has a specific gravity of 1.18. The base substance of the superplasticizer was sulphonated naphthalene formaldehyde.

2.5. Preparation of Mixes

The mix was designed for a flow of 400±25 mm. Based on previous research [14], the ideal quarry waste content for maximum compressive strength was 10% by weight of the total binder component (i.e., fly ash + gypsum). The earlier analysis for NLC fly ash determined that the optimal gypsum concentration was 10% by weight of fly ash [15]. The water content needed to achieve a 400±25 mm flow was found to be 60% by weight of binder without super plasticizer.

The performance of a flowable F-G slurry may be impaired due to its high water content. To lower the water content, another mixture was matched with superplasticizer with the same flow rate. According to an early assessment, the water content of 300 mm flow mix was 43% by weight of the binder, and the superplasticizer concentration was modified to provide a 400±25 mm flow. The superplasticizer concentration was determined to be 1% by weight of the binder, with 43% water. Mixing with super plasticizer not only decreases the water content, but also the amount of components. The mix without superplasticizer was termed the control mix. The specifics of the mixes are shown in Table 4. The flowable F-G slurry without super plasticizer is labelled as CM, whereas the F-G slurry with super plasticizer is designated as FG-SP.

Table 4: Mix proportions.

Mix Ingredient	Quantity in kg/cum	
	Control Mix (CM)	F-G Slurry with Super Plasticizer (FG-SP)
Fly ash (kg/m^3)	907.88	798.83
Gypsum (kg/m^3)	90.78	79.8
Quarry waste (kg/m^3)	99.86	87.8
Water (kg/m^3)	599.08	377.84
Super Plasticizer (kg/m^3)	--	8.78
W/ (FA + G)	0.60	0.43
Flow/Spread (mm)	405.0	394

2.6. Test Procedure

The F-G slurry specimens were tested for compressive strength, wetting and drying, sulphate and salt resistance, and water absorption. All tests were carried out on cylindrical specimens with dimensions of 150 mm in diameter and 300 mm in height. For each test, three cylindrical specimens were utilised. Each test's average test value was calculated using three cylindrical specimens. The produced F-G slurry was cast in cylinder moulds with a diameter of 150 mm and a height of 300mm. After 24 hours, the specimens were demolded and humid-cured until they reached compressive strength. The compressive strength was measured at ages 7, 28, and 56 days.

To investigate the resistance of F-G slurry specimens to weathering action, an accelerated weathering cycle was performed. After 28 days of curing, cylindrical specimens were removed from the curing tank and weighed. The specimens were left in distilled water for 24 hours. The specimens were then dried in an oven at 110°C for 24 hours, completing one cycle of wetting and drying. At the end of each cycle, the specimens' weight was measured. A total of six cycles of alternating soaking and drying were performed. After six cycles, the specimens' compressive strength was assessed.

The soils contained sulphate values that ranged from 0.1 to 0.3%, with an occasional high of over 1%. To speed up laboratory experiments, research has customarily utilised 3% by mass magnesium sulphate dissolved in distilled water. Following demoulding, the specimens were weighed before being immersed in a sulphate solution for attack. A 3% magnesium sulphate solution was produced, and the specimens were submerged in it. After 28 days of immersion, samples were removed, surface dried, and weighed. The % weight decrease was computed. The specimens were next evaluated for compressive strength.

Specimens were weighed before being immersed in chloride solution for salt attack. A 3.5% sodium chloride solution was produced, and the specimens were immersed. After 28 days of immersion, samples were removed, surface dried, and weighed. The % weight decrease was computed. The specimens were next evaluated for compressive strength.

For the water absorption test, cylindrical specimens were removed from the curing tank after 28 days of curing and dried in an oven for 24 hours. The dried specimens were cooled to room temperature, and their dry weight was reported. The dried specimens were soaked in water. The saturated weight of the specimens was measured at specified intervals after cleaning the surface with a dry towel. This method was repeated for at least 48 hours or until the weight remained consistent in two consecutive assessments.

3. Results and Discussions

3.1 Compressive Strength of F-G Slurry Mix

Table 5 shows the test results for compressive strength. The compressive strength of CM was reported to be 1.82, 2.30, and 2.98 MPa after 7, 28, and 56 days. The compressive strength of FG-SP was 4.21, 5.80, and 5.97 MPa after 7, 28, and 56 days. Table 5 reveals that FG-SP's compressive strength increases by a strength factor of 2.3 during 7 days when compared to CM. The compressive strength of FG-SP increases by a factor of two at both 28 and 56 days. The addition of super plasticizer boosted the compressive strength of the F-G slurry. The apparent water-reducing effect was responsible for the increase in compressive strength. The binder-water reaction's hydration efficiency resulted in an extra improvement in compressive strength. The best performance was attained at both early and late ages. Thus, the application of super plasticizer proved effective.

Table 5: Compressive strength of F-G slurry mix.

Age (Days)	Compressive Strength (MPa)		Strength Factor (FGSP/CM)
	CM	FGSP	
7	1.82	4.21	2.3
28	2.30	5.80	2.0
56	2.98	5.97	2.0

3.2. Wetting and Drying of F-G Slurry Mix

The wetting and drying findings are depicted in Tables 6 and 7. No obvious change in appearance of F-G slurry specimens was noticed following exposure in either of the F-G slurry control mix (CM) or mix with superplasticizer (FG-SP) specimens. After six cycles, there was no sign of a damaged surface. Drying did not result in a substantial change in mass for the F-G slurry control mix (CM) or mixes with superplasticizer. However, the weight of cylindrical specimens decreases continuously as the number of cycles increases. After drying and soaking, CM and FG-SP lost 11.2% and 9.2% of their mass, respectively. In addition, the compressive strength of the control mix (CM) was 2.06 MPa, whereas the mix with super plasticizer (FG-SP) was 5.24MPa. The percentage drop in compressive strength was 10.4% and 9.6% for CM and FG-SP mix specimens, respectively.

Table 6: Relative weight after repeated wetting and drying of F-G slurry.

Type of Mix	Cycle	Weight (kg)			Average Weight (kg)	Relative Weight (%)
		1	2	3		
CM	0	8.54	8.59	8.61	8.58	100
	1	8.39	8.49	8.5	8.46	98.6
	2	8.46	8.22	8.14	8.27	96.4
	3	8.12	8.24	8.1	8.15	95.0
	4	8.2	8.11	7.98	8.10	94.4
	5	8	7.85	7.65	7.83	91.3
	6	7.83	7.64	7.24	7.57	88.2
FG-SP	0	8.70	8.67	8.65	8.67	100.0
	1	8.61	8.60	8.59	8.60	99.2
	2	8.54	8.41	8.32	8.42	97.2
	3	8.36	8.23	8.27	8.29	95.6
	4	8.25	8.24	8.24	8.24	95.1
	5	7.98	8.12	8.05	8.05	92.8
	6	7.59	8.17	7.86	7.87	90.8

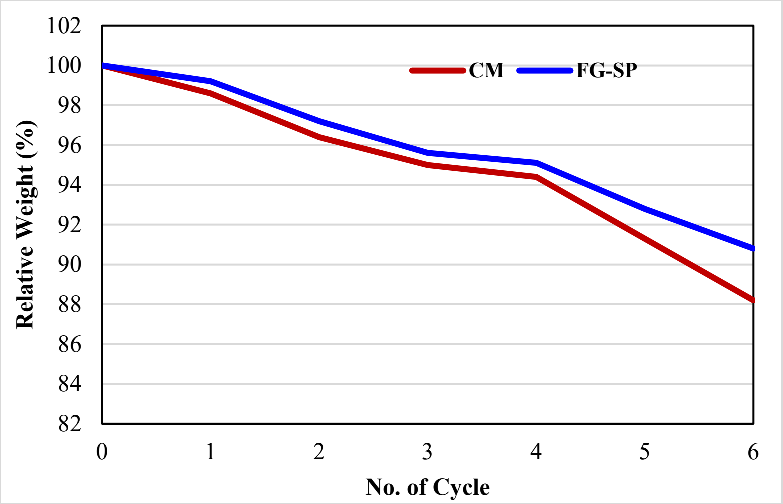

Fig. 1: Relative weight after repeated wetting and drying of F-G slurry.

Table 7: Compressive strength of F-G slurry mix with and without super plasticizer after 6 cycles of repeated wetting and drying.

Mix	Compressive Strength (MPa)		Compressive Strength Loss (%)
	Moist Curing	After 6 Cycles of Wetting & Drying	
CM	2.30	2.06	10.4
FG-SP	5.80	5.24	9.6

3.3 Sulphate Attack of F-G Slurry Mix with and without Super Plasticizer

Table 8 summarises the results of sulphate attack on an F-G slurry mix with and without superplasticizer. The specimens' sulphate resistance was determined by calculating their weight loss and compressive strength reduction. The percentage reduction in weight of the control mix was around 7.95. The weight loss of F-G slurry with superplasticizer was 6.84%. The percentage loss of compressive strength following immersion in sulphate solution for the control mix was 7.82%, whereas the F-G slurry with super plasticizer was 5.68%. The addition of super plasticizer significantly lowers the impact of sulphate. The specimen's surface showed no signs of scale development. Similarly, no damage or degradation was seen. This shown that combining F-G slurry with super plasticizer increased its durability.

Table 8: Test results of F-G Slurry mix with and without super plasticizer for sulphate attack.

Type of Mix	Weight Loss after 28-days Attack (%)	Compressive Strength		Compressive Strength Loss (%)
		Moist Curing	Sulphate Solution Curing	
CM	7.95	2.30	2.12	7.82
FG-SP	6.46	5.80	5.47	5.68

3.4. Salt attack of F-G Slurry Mix with and without Super Plasticizer

Salt attack was assessed by measuring weight loss and compressive strength reduction. This was computed similarly to the sulphate attack. Table 9 summarises the salt attack findings for F-G slurry mixes with and without super plasticizer. There was no apparent staining in any of the specimens. The absence of any staining was interpreted as a sign of anticipated long-term performance. There was no significant difference in weight between the control mix and the F-G slurry mix with super plasticizer. The percentage reduction in weight of the control mix was around 7.21. The weight loss of F-G slurry with superplasticizer was 6.84%. The percentage loss in compressive strength of the control mix was approximately 5.22%. The percentage drop in compressive strength of F-G slurry with superplasticizer was just 2.75%. This indicates a significant increase in the quality of F-G slurry with superplasticizer.

Table 9: Test results of F-G slurry mix with and without super plasticizer for salt attack.

Type of Mix	Weight Loss after 28 days Attack (%)	Compressive Strength at 28 days		Reduction in Compressive Strength (%)
		Moist Curing	Chloride Solution Curing	
CM	7.21	2.30	2.18	5.22
FG-SP	6.84	5.80	5.64	2.75

3.5. Comparison of FG Slurry in Different Exposures

Table 10 shows the % retention of compressive strength and weight following exposure to various environments. The percentage retention of compressive strength after exposing the specimens in salt solution was 94.8% for CM and 97.2% for FG-SP. The percentage retention of compressive strength after exposing the specimens in sulphate solution was 92.2% for CM and 94.3% for FG-SP. The compressive strength of specimens after 6 cycles of soaking and drying showed 89.6% retention for CM and 90.4% retention for FG-SP.

The percentage weight retention following exposure to salt solution was 92.74% for CM and 93.54% for FG-SP. The percentage weight retention following exposure to sulphate solution was 92.05% for CM and 93.16% for FG-SP. The weight of specimens after 6 cycles of soaking and drying showed 88.2% retention for CM and 90.8% retention for FG-SP.

When FG slurry specimens were treated to alternating wetting and drying, they demonstrated a lower resistance to keeping their quality than previous exposures. FG-SP combination demonstrated greater resistance to salt deteriorate than other exposures.

Table 10: Percentage retention of compressive strength of F-G slurry mix with and without super plasticizer after different environment.

Type of Mix	Percentage Retention of compressive Strength			Percentage Retention of Weight		
	In Sulphate Solution	In Chloride Solution	After 6 Cycles Wetting and Drying	In sulphate Solution	In Chloride Solution	After 6 Cycles Wetting and Drying
CM	92.2	94.8	89.6	92.1	92.8	88.2
FG-SP	94.3	97.3	90.4	93.5	93.2	90.8

4. Conclusion

The findings drawn from the aforementioned test results are summarised below.

The inclusion of super plasticizer lowered the water required to generate a 400±25mm spread by 17%. In addition to an improvement in strength, superplasticizer and decreased water/binder showed an increase in strength across all ages. The fly ash gypsum slurry mix without superplasticizer had almost the same durability as the mix without superplasticizer.

When compared to F-G slurry without superplasticizer, F-G slurry with superplasticizer has higher resistance to sulphate attack, salt attack, and wetting and drying. The weight loss following sulphate and salt attack was much greater for F-G slurry with superplasticizer compared to the mix without superplasticizer. There was no damage seen after repeated soaking and drying. The wet-dry cycle produced good results. Water absorption in F-G slurry with super plasticizer was 1.3% lower than in F-G slurry without super plasticizer.

F-G slurry performed satisfactorily in wetting and drying, as well as sulphate and salt attack. Fly ash, as a pozzolanic substance, enhanced resistance to aggressive attacks by sulphate and salt solutions. The relative improvement was larger when mixed with superplasticizer. The addition of super plasticizer to fly ash gypsum slurry increased its mechanical and durability.

References

[1] ACI Committee 229R, (1994) Controlled Low Strength Materials (CLSM). ACI Concrete International, Vol.16, No.7, pp. 55-64

[2] William. C. Krell, "Flowable Fly ash," *ACI Concrete International*, vol.11, no.11, pp. 54-58, 1989.

[3] Tarun, R.Naik. Bruce, W.Ramme, Henry.J.Kolbeck, "Filling Abandoned Underground Facilities with CLSM Fly ash Slurry," *ACI Concrete International*, vol. 12, no.7, pp.19-25, 1990.

[4] Bruce.W. Ramme, Tarun, R.Naik. Henry. J.Kolbeck, "Use of Fly Ash Slurry for Underground Facility Construction," *Construction and Building Materials*, vol.8, no.1, pp.63-67, 1990

[5] Tarun, R.Naik, Shiw, S.Singh, "Flowable Slurry Containing Foundry Sands," *Journal of Materials in Civil Engineering*, pp.93-102, 1997.

[6] T.S. Butalia, W.E Wolfe, J.W. Lee, "Evaluation of a Dry FGD Material as A Flowable Fill," *Fuel.*, vol.80, pp.845-850, 2001.

[7] V. Revathi, V. L. Narasimha, "Development and Characterization of Flowable High –Calcium Fly ash-Gypsum Slurry," *Indian Concrete Journal*, vol.79, no.1, pp.49-53, 2005.

[8] A. Christine Langton, N. Rajendran, Stanley. E.Smith, "Use of Pond Ash in CLSM," *ACI Concrete International*, pp.58-62, 1998

[9] C.E. Pierce, M.C. Blackwell, "Potential of Scrap Tire Rubber as Lightweight Aggregate in Flowable Fill," *Waste Management*, vol. 123, no.3, p.197-208, 2003

[10] T.M. Do, Ys. Kim, "Engineering properties of controlled low strength material (CLSM) incorporating red mud," *Int.Journal of Geo-Engineering* 7, 7, pp.1-17, 2016.

[11] J.P. Won, Y.S. Lee, C.G. Park, H.G. Park, "Durability Characteristics of Controlled Low Strength Materials Containing Recycled Bottom Ash," *Magazine of Concrete Research*, vol. 64, no.7, pp.429-436, 2004.
[12] Bruse.W.Ramme, "Progressive in CLSM: Continuing Innovation," *Concrete International.*, pp.32 -33, 1997.
[13] W.E. Brewer, "Durability Factors Affecting CLSM," *SP 150-3*, American Concrete Institute, Detroit, MI, USA, pp.39 – 51, 1994
[14] V. Revathi, V. L. Narasimha, S.Jayanthi, "Studies on the Performance of Quarry Waste in Flowable Fly ash-Gypsum Slurry," *Modern Applied Science*, vol. 3, no. 2, pp147-153, 2009.
[15] V.L. Narasimha, T.Sundararajan, V.Revathi , :High Calcium Fly ash Gypsum Slurry: A Potential Material for High Volume Utilization of Fly ash," in *Proceedings of CBIP International Conference*, New Delhi India, Feb 19-21, 2003, pp 19-21.