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Durability Properties of Admixture of Fly ash, Bottom Ash and GBFS

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Abstract:

Fly ash, bottom ash and granulated blast furnace slag (GBFS) are industrial wastes materials from thermal power plants and steel industries. Due to stringent disposal laws, it is a challenging issue to dispose-off. It can be used as concrete admixture of above these raw materials by geopolymer techniques. It will reduce the cost of disposal, which includes treatment, transportation, and environmental cost and also reduce consumption of cement. Ordinary Portland cement concrete is very popular in construction but it is not an eco-friendly material because a huge amount of energy consumed for cement production and their consequent a huge amount of CO₂ is released in atmosphere. This article especially emphasized the use of raw material as eco-friendly and effect in compressive strength of their product concrete exposed in various chemical environments, various exposure duration and various silicon/aluminium ratios. The compressive strength of sample was determined in 1% and 3% of H₂SO₄, CH₃COOH, Na₂SO₄, MgSO₄, and NaCl solutions for exposure 7, 28 and 60 days. The reduction in compressive strength was found in the entire sample in each condition.

Key words: Bottom ash (BA), Compressive strength, Durability, Flyash (FA), Granulated blast furnace slag (GBFS), Geopolymer,

Introduction:

In India, the main source of energy is thermal power plant which is based on coal. During the production of power from thermal plant, huge amount of waste, fly ash (FA) and bottom ash (BA) were generated. Fly ash is composed of the particulates that are driven out of coal fired boilers together with flue gases. Ash that falls to the bottom of boiler combustion chamber is called bottom ash. About 43% fly ash is recycled [1] as a pozzolan to produce hydraulic cement. Granulated blast - furnace slag (GBFS) is obtained by quenching molten iron slag from a blast furnace in water or stream, to produce a glassy, granulated product that is then dried and ground into a fine powder. It is highly cementitious and high in calcium silicate hydrates (CSH) which is a strength enhancing material in concrete. Concrete is used construction materials with Portland cement as the main component. The global cement production is increasing day to day and expected the 4.83 billion metric tons up to 2030 [2]. Many researchers have reported that the production of one ton of Portland cement emits one ton of CO₂ into the atmosphere [2]. The cement industry itself responsible for about 6% overall carbon dioxide in atmosphere and contribute approximately 65% in global warming. Hence, alternative material for cement is required. The millions of tons of fly ash are generated every year by coal-fired power plants for satisfying the large demand of industrial and domestic energy [2-4]. But only about 60% of the generated fly ash is used as additive in cement and concrete, rest as filling material [5]. Aluminosilicates and alkaline activators are responsible materials for preparation of alkali-activated materials generally known as geopolymer which is eco-friendly and having excellent properties such as high early compressive strength, alkali & acid resistance etc[6-11]. Granulated blast furnace slag [12,13] fly ash [14] and metakaolin [15] are the major aluminosilicates materials, which are used as precursors, and the activators include caustic alkali, silicate, carbonate and sulfate, etc. [16]. Geopolymers contain mainly two types of gel system, one is high-calcium and other is low calcium system which is dominated by calcium alumina silicate hydrate (C-A-S-H) gels and alkali aluminosilicate (N-A-S-H) gels respectively. Low-calcium gel system, is also called 'geopolymers', in which alkali aluminosilicate (N-A-S-H) gels with pseudo-zeolitic structure are the main products, like alkali- activated fly ash and other low-calcium binders. The C-A-S-H and N-A-S-H gels could coexist in alkali-activated blended

systems [17,18], and the main reaction products could change from N-A-S-H to C-A-S-H gel with the increase of calcium content, which is beneficial to the composition design of alkali activated materials (AAMs)[19]. It is wide used in building components [20]. In geopolymerization, the chemical reaction between almino-silicate oxides with alkali formed polymeric Si-O-Al-O bonds[21] due to this bond amorphous 3-D structure of geopolymer described by the formula: $M_n [-(Si-O)_2 - Al - O]_n \cdot nH_2O$, Where in 'M' is a cation such as potassium, sodium or calcium, 'n' is a degree of polycondensation and z is 1, 2, 3. The Mechanical Properties improved at the early age of fly ash based geopolymer concrete cured at ambient condition [22] while, the significant change in concrete properties like thermal, mechanical and deformation at elevated temperature [23]. The concrete durability is a critical issue because it start deteriorate after 20 to 30 years in urban and coastal environment , though their life design was at least 50 years [24]. Geopolymer concrete has more resistance to sulphate and chloride attack[25-26] in comparison to OPC concrete. In sulfate environment, it make stable cross-linked aluminosilicate polymer structure. The compressive strength of geopolymer materials is superior to OPC paste in acid solution [27-29].

EXPERIMENTAL DETAILS:

Source of raw materials

Fly ash was collected from Usha Martin Ltd. situated at Tatisilwai, Ranchi. Bottom ash was collected from Bokaro thermal power Plant, Bokaro, & GBFS was collected from Tata steel, Tata. The chemical compositions of the raw materials were carried out using inductively coupled plasma-Optical emission spectroscopy (ICP-OES), Perkin-Elmer 3000 and by conventional method such as titration. The chemical compositions are given in table 1

Table 1: Chemical composition of the raw materials

Constituent	Fly Ash	Bottom Ash	GBFS Slag
SiO ₂ (%)	51.06	51.71	14.20
Al ₂ O ₃ (%)	29.71	16.20	2.69
Fe ₂ O ₃ (%)	9.60	13.71	25.34
CaO (%)	2.14	3.61	42.83
MgO (%)	0.75	1.56	5.05
Na ₂ O (%)	0.56	0.39	---
K ₂ O (%)	0.40	0.50	---

Physical Characterizations of raw materials:

The physical characterizations of raw material are shown in table 2. The specific gravity of the materials was determined as per IS 2386 (Part III) using Pycnometer and the particle size distribution of raw material was carried out as per IS 383 by sieve analysis. The sieve analyses of the raw materials are shown in Table 3. The particles were distributed in the range from - 325 mesh to +50 mesh.

Table 2: Physical properties of raw materials

Physical property	Fly ash	Bottom ash	GBFS
Color	Gray	Gray	Light Gray

Form	Powder	Powder	Powder
Plasticity	Non plastic	Non plastic	Non plastic
Apparent Specific Gravity	1.972	1.957	2.88

Table 3: particle size distribution of raw materials.

Sieve No.	Retained Wt. (%) Fly ash	Retained Wt. (%) GBFS	Retained Wt. (%) bottom ash
+229 μ	20	42	98
-229 μ , +149 μ	38	10	2
-149 μ , +74 μ	30	26	0
-74 μ , +44 μ	6	8	0
-44 μ	2	10	0
6% Fly ash particle are loosed during Sieve analysis. 4% GBF slag particle are loosed during Sieve analysis			

X ray diffraction Analysis: XRD analysis was carried out using X-ray BRUKER diffractometer (Model D8 Discover) using $K\alpha$ radiation for the mineralogical analysis of raw materials. The diffraction pattern was recorded between angle of 10^0 to 80^0 using the scanning speed of 2^0 /min.

Scanning Electron Microscopy analysis: The main purpose of SEM is to provide information about the surface morphology. EDAX was used to analyze the elemental composition of each micro-phase present in the structure. The scanning electron microscope used in this study was JOEL Scanning Electron Microscope (JSM-6390LV) with an EDAX attachment.

Preparation of geopolymer samples: The geopolymer samples were prepared by blending GBFS with fly ash and bottom ash in various concentration of activator solution (6 M, 10M and 14 M NaOH) by maintaining different Si/Al ratio 2,3 and 4 (by mass) as shown in table 4. Fly Ash, bottom ash and GBFS were spread on the mixing pan in the fixed proportion and dry mixed using trowels for 3-4 minutes. After properly dry mixing the ingredients, required NaOH + Sodium Silicate solution was added to make the paste. The dimensions of the cylindrical samples were: diameter = 3.475cm, height = 4.25cm and cross sectional area of the contact surface = 976 mm^2

Table 4: Composition of Fly Ash-Bottom Ash-GBFS (FBG) Geopolymers Sample

Sample Nomenclature	Sample compositions				
	Fly ash (gram)	Bottom ash (gram)	GBFS (gram)	Molarity of NaOH	Silica/alumina ratio
2FBG6M	2400	800	800	6M	2
3FBG6M	2400	800	800	6M	3
4FBG6M	2400	800	800	6M	4
2FBG10M	2400	800	800	10M	2
3FBG10M	2400	800	800	10M	3
4FBG10M	2400	800	800	10M	4
2FBG14M	2400	800	800	14M	2
3FBG14M	2400	800	800	14M	3
4FBG14M	2400	800	800	14M	4

Where 2, 3, 4 numbers is Si/Al ratio, 6M, 10M and 14 M is molarity of NaOH solution and F, B, G are fly ash, bottom ash and GBFS respectively.

Compressive Strength Test: The compressive strength of prepared samples was determined as per the American Society for testing and materials (ASTM) specifications, by using compression testing machine. The loading rate of compression testing machine was 2 MPa/min. Triplicate samples were used for the compressive test after curing at 7, 28 and 60 days. The results were reported in MPa.

Durability Test: The durability test of samples were conducted by immersing the samples in 1% and 3% H₂SO₄, CH₃COOH, Na₂SO₄, MgSO₄ and NaCl solutions for 7, 28 and 60 days respectively. After 7, 28 and 60 days the compressive tests were done to know the extent of reduction in compressive strength caused by the acid and salt solutions. The solutions represent adverse conditions of atmosphere.

Result of mineralogical composition of raw material: The mineralogical composition of fly ash, bottom ash and blast furnace slag were determined by using XR. XRD pattern of fly ash, bottom ash and milled blast furnace slag are shown in Figure 1(a), (b) and (c) respectively. XRD results revealed that fly ash and bottom ash are mainly crystalline in nature. Fly ash and bottom ash have similar mineral phases like mullite and quartz. While the granulated blast furnace slag showed that Gehlinite mineralogical phase which was mostly amorphous in nature.

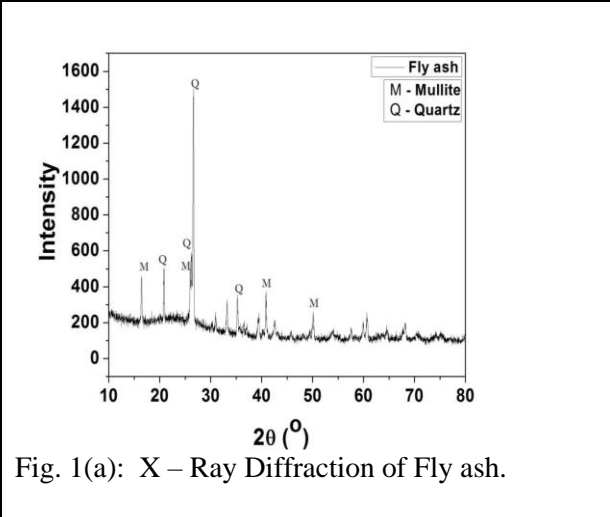


Fig. 1(a): X – Ray Diffraction of Fly ash.

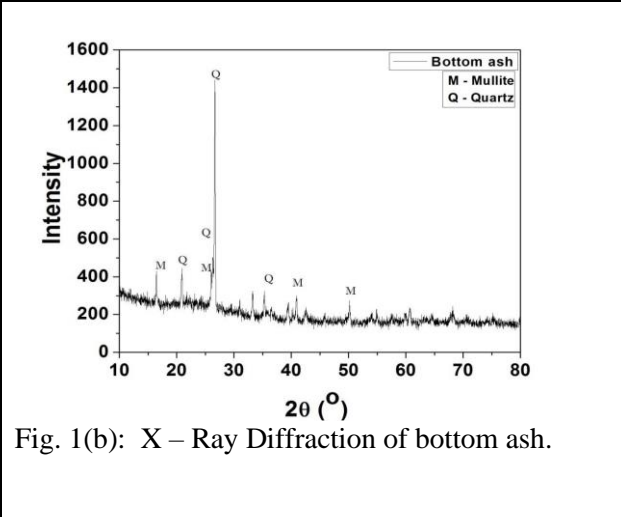


Fig. 1(b): X – Ray Diffraction of bottom ash.

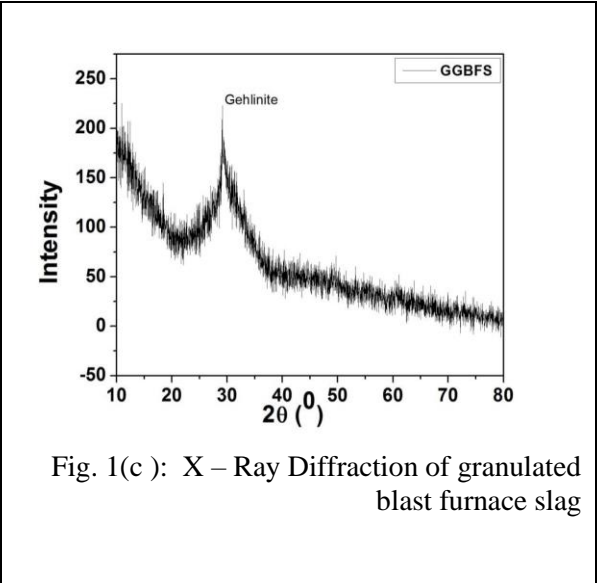


Fig. 1(c): X – Ray Diffraction of granulated blast furnace slag

Results of Compressive strength:

The compressive strength of the FBG samples, after curing for 28 days was the function of Si/Al ratio and concentration of activator solution (NaOH). The minimum strength was 15.71 MPa for 6M NaOH solution with Silica/Alumina ratio 2 and the maximum strength was 25.26 MPa for 10M NaOH solution with Silica/Alumina ratio 4 in FBG samples as shown in Fig. 2. The strengths of the geopolymers depend on the concentration of the activator used in specimen preparation and Si/Al ratio. The specimens that were prepared with high concentration sodium hydroxide and high Si/Al ratio gained more compressive strength than specimens prepared with low concentration sodium hydroxide activator and low Si/Al ratio.

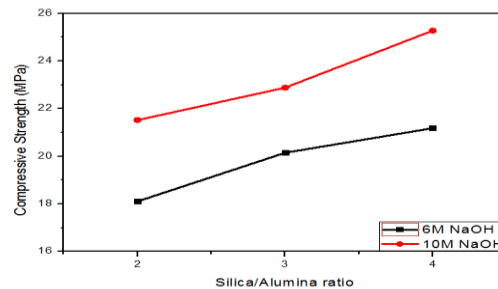
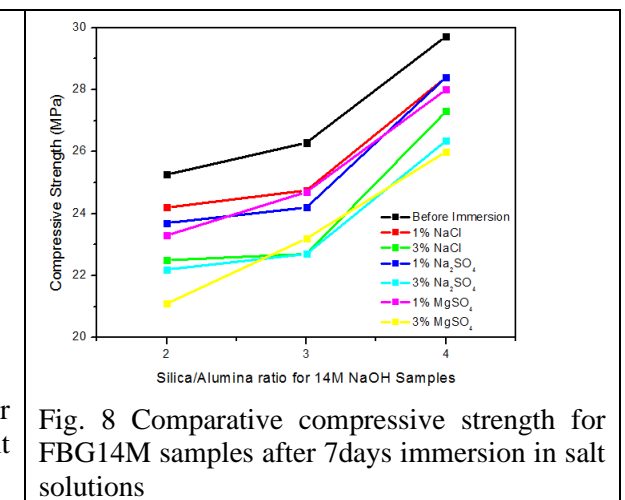
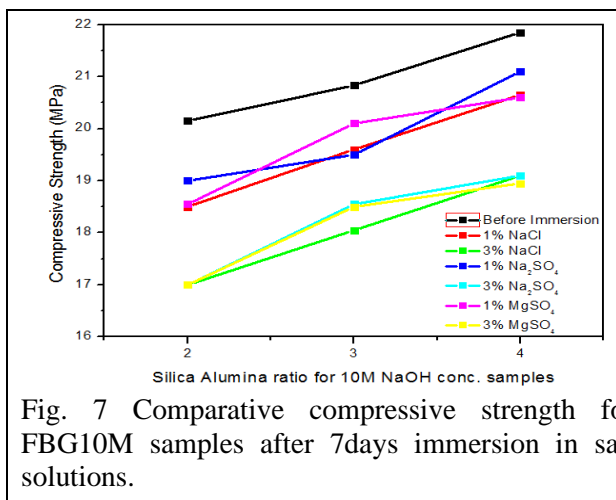
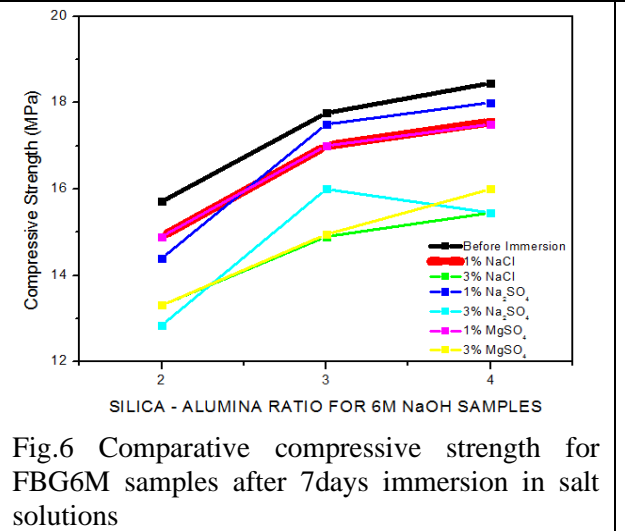
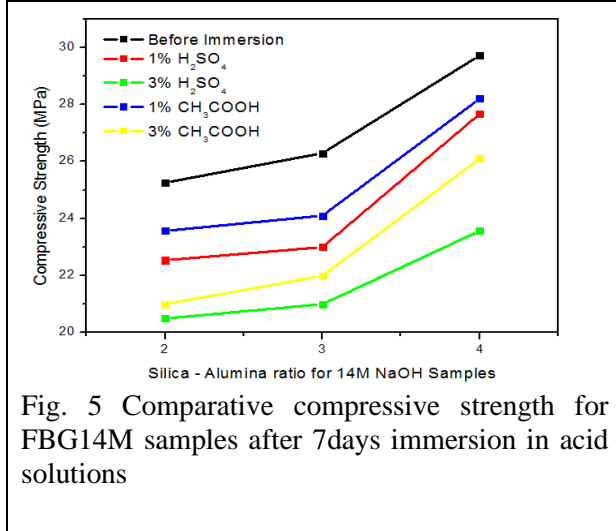
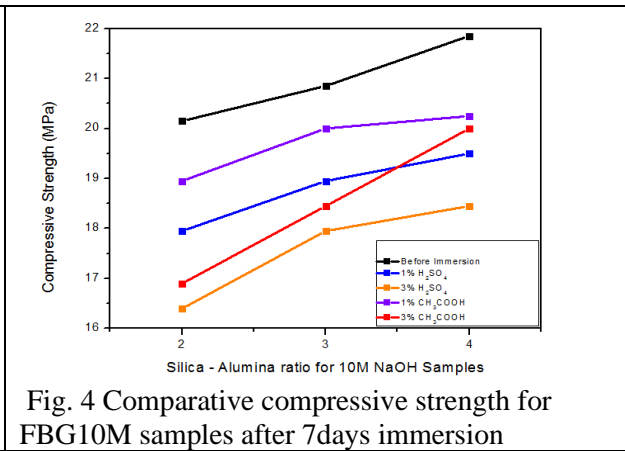
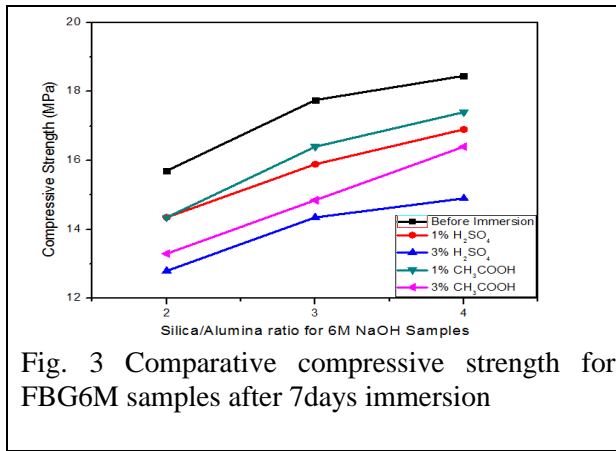


Fig 2: Variation in Compressive strength of Geopolymer samples (FBG)

Result of durability Test:

Durability test of samples were conducted by immersing the samples in 1% and 3% H_2SO_4 , CH_3COOH , Na_2SO_4 , $MgSO_4$ and $NaCl$ solutions for 7, 28 and 60 days respectively according to the Indian standard IS456:2000. After 7, 28 and 60 days the compressive tests were done to find to how much extent of reduction in compressive strength occurred due to the acid and salt solutions. The change in compressive strength for 7 days is shown in the Fig.3 to 8. The results showed that the compressive strength of the specimens which were immersed in sulphuric acid decreased more in comparison to the specimens that were immersed in acetic acid. It also depends on the concentration of the acid. Concrete requires different degrees of durability depending upon its use [12]. The compressive strength of the immersed samples in acids decreased in comparison to bare samples. The graphs also showed that the compressive strength is a function of concentration of activator solution in each case. Geopolymer FBG 14M specimens with Si/Al ratio 4 were the most resistant in acid as well as salt solution in all aged 7, 28 and 60 days. Generally, all FBG samples were acidic resistant as shown in Fig.3 to Fig. 8. In sodium chloride solution, generally, the compressive strength of the FBG samples decreased in comparison to the specimens that were immersed in other salt. Geopolymer FBG 14 M specimens with Si/Al ratio 4 was provide maximum compressive strength 30 Mpa and more resistant in salt and acid solutions as shown in Fig.5, 8, 11, 14, 17 and 20. The compressive strength of the specimens decreased with the increase of sulphate and chloride concentration. The change in compressive strength of FBG samples after immersion in acid and salt solutions for 28 days are shown in Fig. 9 to Fig.14. The compressive strength of specimens decreased with increase in time of immersion. The change in compressive strength of FBG Geopolymer samples after immersion in acid and salt solutions for 60 days are shown in Fig. 15 to Fig.20. Almost similar trends were observed. In acidic medium, the surface texture of the samples was changed due to deposition of acid ions. It also reacted with Si-O-Al skeleton bonds. Formation of Si-OH and Al-OH groups in geopolymers are due to the breakage of geopolymer skeleton structure and increased amount of silicic acid ions and dimers in solution and this process provide to the weight loss of the geopolymer materials. After immersion in the H_2SO_4 solution the surface texture of samples changed

from smooth to rough. There is no much change in the surface texture of samples after immersion in acetic acid. In the salt solution; there was no change in surface. It was as smooth as before the test. Depositions of salt layers were observed on the samples after immersion in salt solutions.



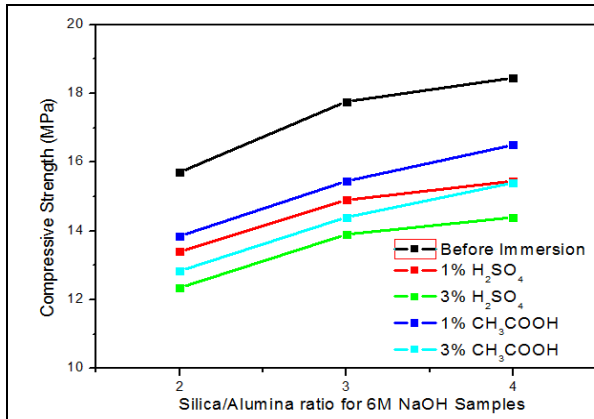


Fig.9 Comparative compressive strength for FBG6M samples after 28days immersion in acid solutions

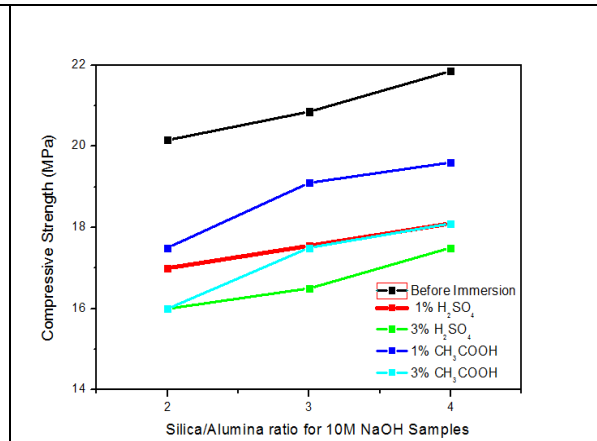


Fig. 10 Comparative compressive strength for FBG10M samples after 28days immersion in acid solutions

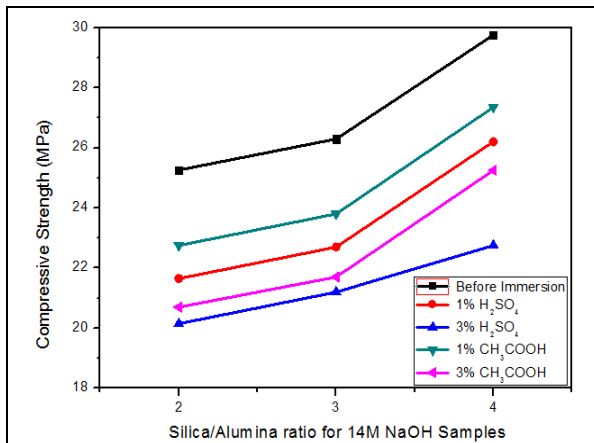


Fig. 11 Comparative compressive strength for FBG14M samples after 28days immersion in acid solutions

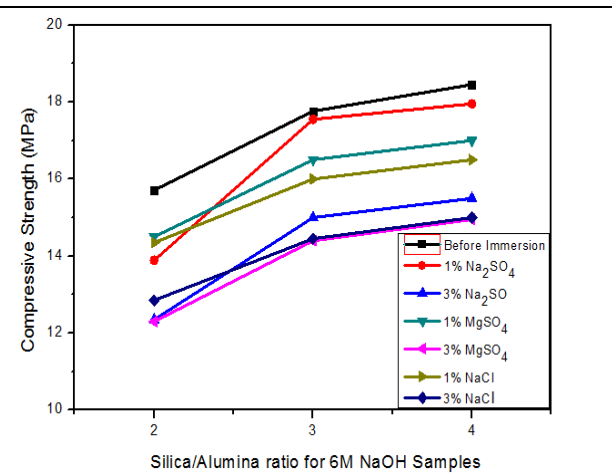


Fig. 12 Comparative compressive strength for FBG6M samples after 28days immersion in salt solutions

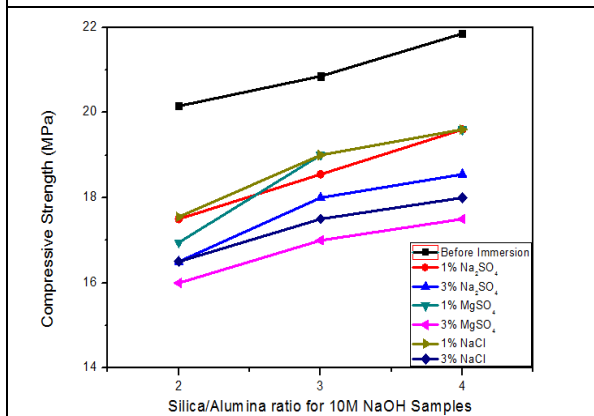


Fig. 13 Comparative compressive strength for FBG10M samples after 28days immersion in salt solutions

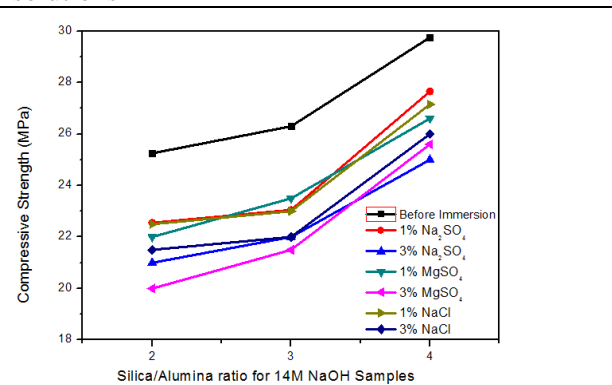


Fig. 14 Comparative compressive strength for FBG14M samples after 28days immersion in salt solutions

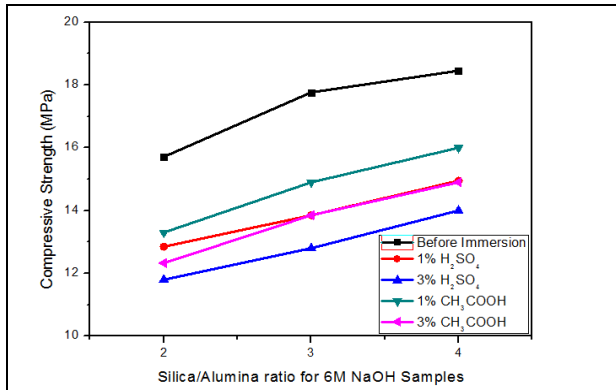


Fig. 15 Comparative compressive strength for FBG6M samples after 60 days immersion in acid solutions

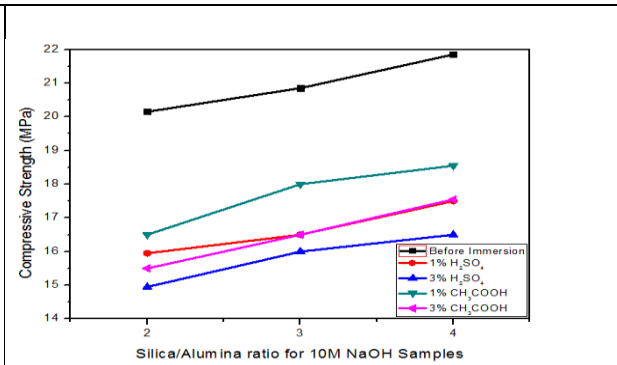


Fig. 16 Comparative compressive strength for FBG10M samples after 60 days immersion in acid solutions

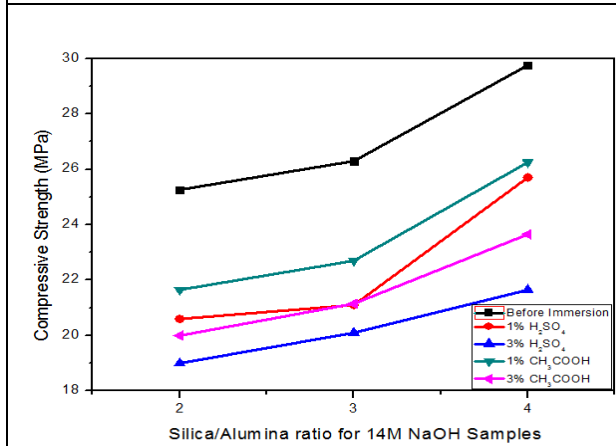


Fig. 17 Comparative compressive strength for FBG14M samples after 60 days immersion in acid solutions

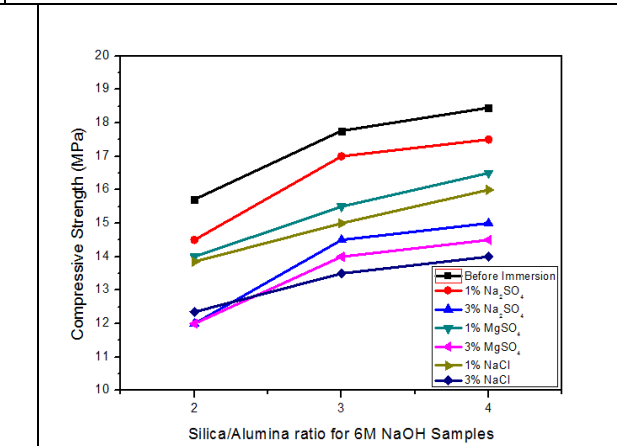


Fig.18 Comparative compressive strength for FBG6M samples after 60 days immersion in salt solutions

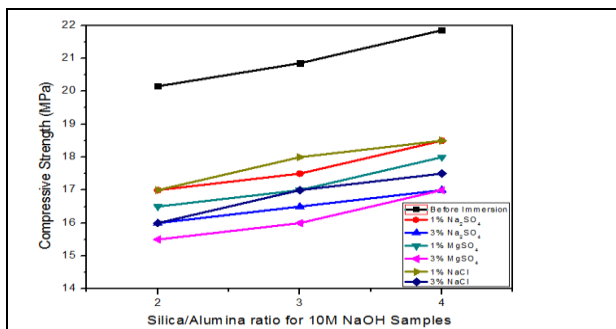


Fig.19 Comparative compressive strength for FBG10M samples after 60 days immersion in salt solutions

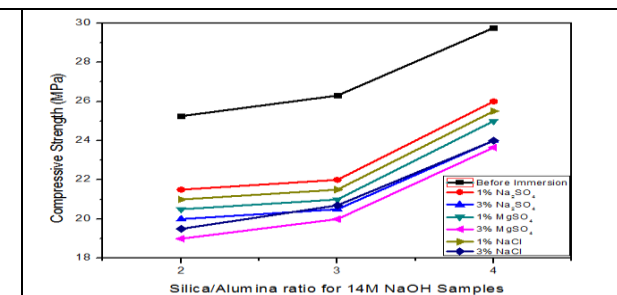


Fig. 20 Comparative compressive strength for FBG14M samples after 60 days immersion in salt solutions

Result of SEM and EDAX Analysis:

Morphological characterizations of the fractured samples were carried out by JEOL scanning electron microscope with an EDAX attachment. Samples were coated with a 2 nm layer of carbon for imaging in the scanning electron microscope. **Reactive geopolymeric** phase of FBG6M Samples (Si/Al = 2) show highly heterogeneous material contain fly ash and slag grain shown in Fig. 21. Fly ash particle are spherical while slag (GBFS) are angular. Fig 22. Show the microanalysis of geopolymeric gel phase of FBG6M samples, which contain 26% silica. The gel phase appears as a homogeneous phase

and reaction also enhanced by increasing concentration of Si/Al ratio (fig.23) and their EDAX analysis (fig. 24). It is clear that the reaction products consist of fly ash geopolymer phase and the remnant of a slag grain phase. The morphology and EDAX analysis of FBG10M samples are shown in Fig. 25-26. The thin walled hollow sphere was clearly shown in Fig. 26. The SEM images and their EDAX analysis which clearly indicates that homogeneity and reactivity increase by increasing concentration of alkali and ratio of silica and alumina as shown in Fig. 27 to Fig. 30. According to EDAX analysis, the result was the atomic percentage of Si/Al ratio 2.88. The microstructure of the sample as shown in Fig. 29 was cenosphere (thin walled hollow spheres) and texture of the surface is smooth and dense to highly porous. The shape of GBFS is not really spherical; it varies according to different grinding techniques. It is predominantly in anomalous shape with edges and angles. The reason for this shape is inter-impacting and inter rubbing between steel balls in ball mill. Aluminosilicate gel was formed by the reaction between the fly ash and sodium silicate (alkaline activator) which covered the fly ash particles and produced a dense matrix (Fig. 29).The cementitious phase of the geopolymers was observed in the Fig.27. The improvement in micro structural homogeneity and reactivity is the main reason for the increase in mechanical properties at higher Si/Al ratios and higher concentration of alkali.

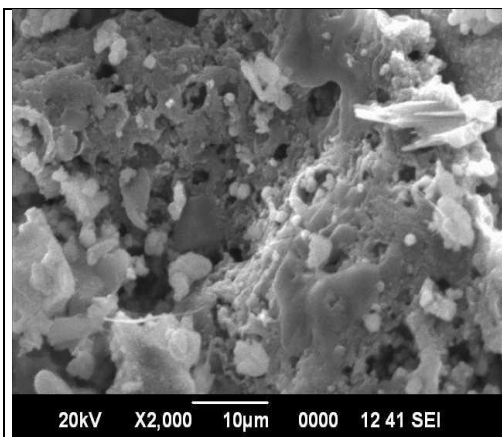


Fig. 21: Reactive geopolymeric phase of FBG6M Samples (Si/Al = 2)

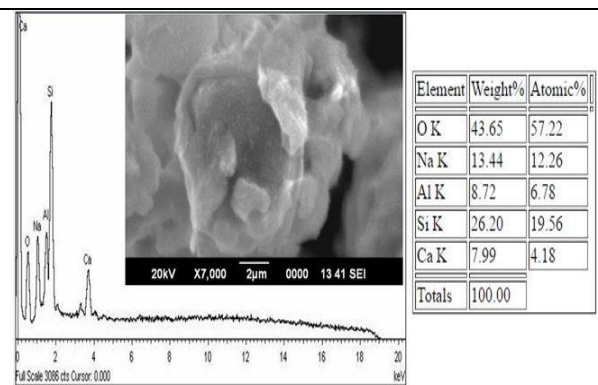


Fig. 22 Geopolymeric gel phase of FBG6M samples (Si/Al = 2) & their EDS

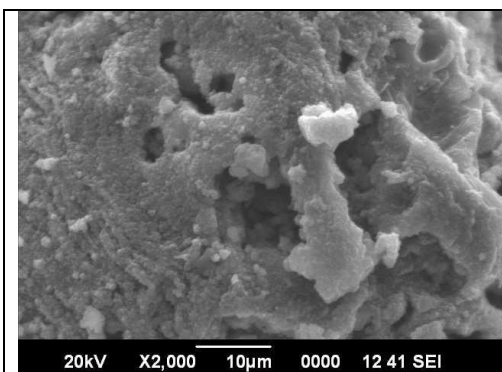


Fig. 23 Reactive geopolymeric phase of FBG6M Samples (Si/Al = 4)

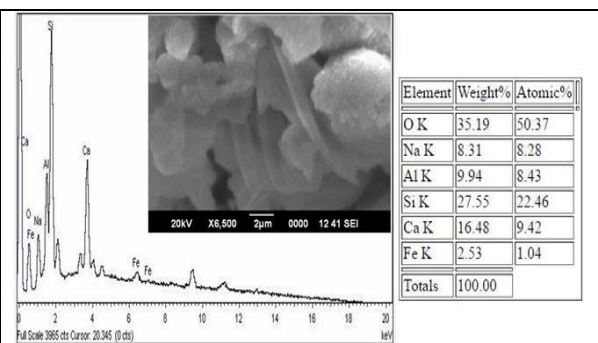


Fig. 24 Geopolymeric gel phase of FBG6M samples (Si/Al ratio 4) & their EDS

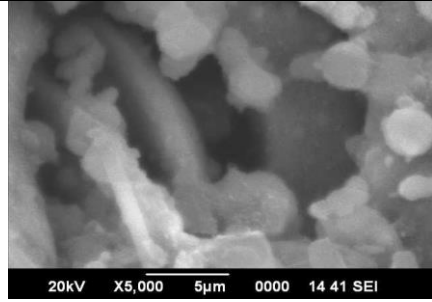


Fig.25Geopolymeric gel phase of FBG10M Samples (Si/Al = 4)

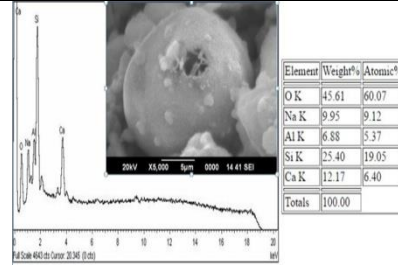


Fig. 26 Reactive fly ash surface phase of FBG10M Samples(Si/Al = 4)& their EDS

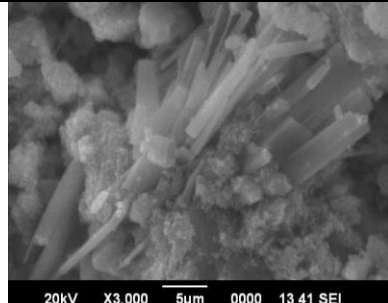


Fig. 27Geopolymeric cementite phase of FBG14M Samples (Si/Al = 2)

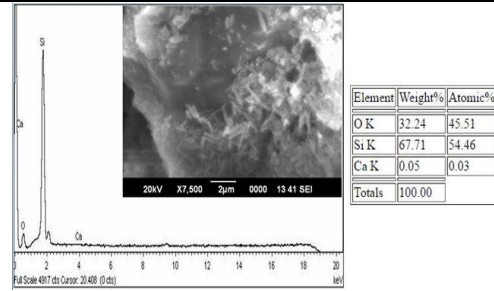


Fig.28Geopolymeric reactive phase of FBG14M Sample (Si/Al = 2)& their EDS

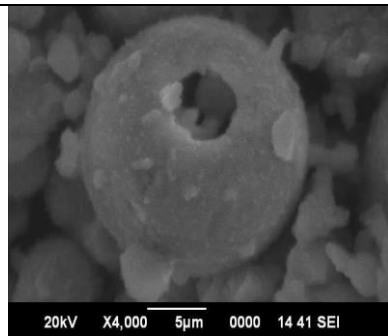


Fig. 29Geopolymeric fly ash surface phase FBG14M Samples (Si/Al = 4)

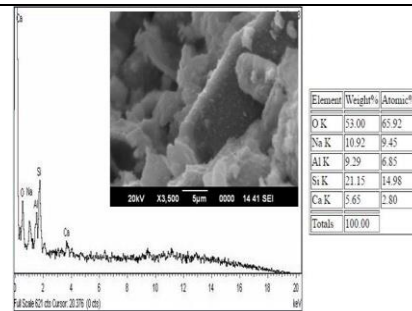


Fig. 30Geopolymeric reactive phase of FBG14M sample (Si/Al = 4)& their EDS

Conclusions:

The compressive strength of samples was depending on concentration of sodium hydroxide activator and Si/Al ratio. The maximum Compressive strength was 30 MPa found in 4FBG14M samples. As per IS code SP 23- 1982. The sample may be used as geopolymer concrete. The geopolymer samples were good resistance in acid and sulphate solution and their resistivity increased with increasing the concentration of alkali. The surface texture of the samples were changed from smooth to rough in exposure of H₂SO₄ solution and acetic acid solution. The reduction in compressive strength of the samples were more in sulfuric acid in comparison to acetic acid and also with increasing exposure duration (7, 28 and 60 days). The loss of compressive strength in FBG geopolymer samples were measured in the range of 15-25%, 12-22%, 14-20%, 11-17% and 13-17% after exposure of 1-3% in sulfuric acid, acetic acid, magnesium sulfate, sodium sulfate and sodium chloride solution respectively. On the basis of results, it can be concluded that geopolymer samples were durable in 60 days exposure of various types of acids and salt solutions.

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