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Studies on Geopolymerisation Reaction using Synthesised Activators

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ABSTRACT: Red mud, a residue of Bayer's process was used synergistically with GGBS to develop geopolymer. Influence of 0-100% red mud addition on the reaction, durability, structure and properties of geopolymer was studied using mechanical testing. An improvement in intensity of reaction was observed with the red mud addition at all replacement level but improvement in compressive strength was observed only in the samples containing 30:70 (RM: GGBS) proportion. Structural characterization revealed that rate of reaction was dependent on the KOH concentration but the development of mechanical properties were related to the compact microstructure which was developed due to the combined effect of KOH concentration, solubility of silicates and the presence of iron oxides. Based on scientific understanding, concrete cubes, cylinders and prisms were cast using pure RM, GGBS mixtures and 30:70, 50:50 and 70:30 proportions for red mud and GGBS. Specimens were subjected to ambient curing and steam curing. It was observed that 30:70 (RM: GGBS) mix gave best mechanical strength relative to M60 grade OPC.

KEYWORDS: **:** Geopolymer concrete, Red mud, Ground Granulated Blast furnace Slag (GGBS), XRD, Microstructure, SEM, Strength.

I. INTRODUCTION

Concrete is the most predominantly used construction material in the world. The basic ingredient of concrete that is Ordinary Portland Cement (OPC) is a major contributor of global warming. The yearly global cement production of 1.6 billion tons is responsible for about 7% of the total CO2 emission into the atmosphere (Mehta, 2001) [1]. The raw materials required for cement production are non-renewable and depleting at a rapid rate. At the same time, a lot of industrial and agro wastes with inherent cementitious properties are produced abundantly but mostly dumped into landfills. Employing such by products as an alternative for cement has multiple benefits including conservation of environment, sustainability of resources and solving the disposal problem of by products. Extensive researches are being carried out to assess the feasibility of utilizing industrial wastes as complete replacement for OPC and generating quality binders from the same. One such successful attempt is Geopolymer concrete which entirely eliminates the use of OPC in concrete production.

Davidovits coined the term 'geo-polymer' in 1978 to describe a family of mineral binders that can be produced from the polymerization reaction between an alkaline liquid and a source material containing silicon (Si) and aluminum (Al), which possess a chemical composition similar to zeolites but exhibiting an amorphous microstructure (Davidovits, 2011) [2]. These zeolitic polymers possess high strength in comparison to OPC concrete (Jun and Oh, 2015) [3]. The polymerization process is a relatively quick chemical reaction involving alkaline liquids and Si-Al minerals. The end product is a three-dimensional polymeric ring structure comprising of Si-O-Al-O bonds, termed as 'silicon oxo aluminates' or 'sialates' in short. Contrasting to OPC, the principal binders in Geopolymer concrete are not calcium-silicate-hydrates. Instead, an aluminosilicate polymeric gel formed by tetrahedrally-bonded silicon and aluminium with oxygen atoms shared in between acts the binder.

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However, several studies have revealed that, when sources like GGBS or Metakaolin containing high amounts of soluble calcium silicates are added, calcium dissolution occurs at low alkalinity resulting in the formation of a C-S-H gel in conjunction with the geopolymeric gel (Yip et al., 2004[4]; Yip et al., 2008[5]; Bernal et al., 2011[6]; Ismail et al., 2014[7]). The geopolymeric gel remains the principal binder phase with small calcium precipitates scattered inside. This co-existence of two binder phases enhances the mechanical strength of the geopolymer concrete (Yip et al., 2004) [4]. Red Mud is an industrial waste generated from aluminum industry. In Geopolymer concrete, most of the research works have been made on Flyash based geopolymers and in this present study, industrial waste which is Ground Granulated Blast furnace Slag (GGBS) and Red Mud are used as source materials for Geopolymer concrete. In present study, RM: GGBS ratio was replaced at different percentages and its effect on the compressive, flexural and tensile strengths were studied.

II. MATERIALS USED

2.1 Red Mud

Red mud is an industrial waste product. It is obtained as a by-product during production of Aluminium from its ore, bauxite by Bayer's process. Red mud was procured from Hindalco industries ltd, Belagavi. Chemical composition of Red mud are given in table 1

2.2 GGBS

GGBS is an acronym for Ground Granulated Blast Slag, another industrial waste product. It is obtained as a by-product during production of iron from its ore, the slag formed during the process floats on surface of mixture. This slag is skimmed off in molten state and quenched using huge volume of water. Later the quenched slag granules are ground into fine powder, resulting in GGBS. GGBS was procured from RMC Ltd, Yelhanka branch, Bengaluru. Chemical composition of GGBS are tabulated in table 2

Compound	Percentage by Weight
Fe_2O_3	30-60%
Al_2O_3	10-20%
SiO_2	3-50%
Na_2O	2-10%
CaO	2-8%
TiO_2	Trace-25%

Table 1: Chemical Comp. of Red Mud

Compound	Percentage by Weight
CaO	30-50%
SiO2	28-38%
Al_2O_3	8-24%
MgO	1-18%
MnO	Trace-8%
TiO_2	Trace-25%

Table 2: Chemical Comp. of GGBS

2.3 Chemicals

In this research work the chemicals used to maintain alkaline reactive medium are 10M Potassium Hydroxide (KOH) and Potassium Silicate (K_2SiO_3).Calculated quantity of commercially available KOH pellets were dissolved in water to produce KOH solution. Potassium silicate was synthesized freshly for the work and used for mixing after 24 hours of synthesis. The governing reaction for preparation of alkaline solution is

$$KOH + K_2SiO_3 \rightarrow \text{GPC Alkaline solution.}$$

Potassium silicate was synthesized by mixing Silicic acid ($SiO.(OH)_2$) and potassium hydroxide (KOH) in an externally cooling medium, as the reaction is highly exothermic in nature. The following chemical reaction takes place

$$2KOH + H_2SiO_3 \rightarrow K_2SiO_3$$

2.4 Aggregates

The crushed sand is of cubical shape with grounded edges, washed and graded to as a construction material. The size of manufactured sand (M-Sand) is less than 4.75mm. Natural basalt rock pieces of 20mm downsize was used as Coarse aggregate.

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III. MIX DESIGN

Control mix was designed using IS 10262:2009 with target strength for M60 grade concrete. Since there are no codal provisions available for the mix design of geopolymer concrete, the density of Geopolymer concrete was assumed as 2400 Kg/m3 and other calculations were made based on the density of concrete as per the design given by Lloyd and Rangan (2010)[8]. The combined total volume occupied by the coarse and fine aggregates was assumed to be 77%. The alkaline liquid to binder ratio was taken as 0.35. Target strength of 60 MPa was fixed considering as a regular strength concrete. The mix proportions were taken as per Table. 3.

Quantities	Control Mix	Mix I	Mix II	Mix III
Cement (in Kg/m^3)	420	NA	NA	NA
Red Mud (in Kg/m^3)	NA	122.4	204	285.6
GGBS (in Kg/m^3)	NA	285.6	204	122.4
Coarse Aggregate(in Kg/m^3)	1123	1294	1294	1294
Fine Aggregate (in Kg/m^3)	710	555	555	555
% KOH solids	NA	35.89%	35.89%	35.89%
% K2SiO3 Solids	NA	37.5%	37.5%	37.5%
Super plasticizer (% of binders)	2.5%	2.5%	2.5%	2.5%

Table 3: Mix Proportion of various mixes

IV. PREPARATION OF TEST SPECIMENS

The materials for the mixes were weighed and first mixed in dry condition for 3-4 minutes. Then the alkaline solution which is a combination of Potassium Hydroxide along with Potassium silicate which includes required water quantity is added in 2-3 instalments. Then super plasticizer along with little water was added to improve the workability if required. The mixing was continued for about 6-8 minutes. After the mixing, the concrete was placed in moulds and subjected to vibration so as to compact it. Precautions were taken to ensure uniform mixing of the ingredients. Two types of curing were used for the specimens to be tested for the compressive strength. One set of cubes was steam cured and the other set was cured under ambient condition. For steam curing, the cubes were de-moulded and placed in the oven at 60°C respectively for 24 hours. Then they were taken out and allowed to cure in the room temperature till the required day of testing. For the ambient curing, the cubes were kept in room temperature after casting and de-moulded after 1 day and further cured in the room temperature till the day of testing. The cubes were then tested at 3, 7 and 28 days from the day of casting. It was found that steam cured specimen performed better then ambient curing specimen in compressive strength, so for all the specimens steam curing procedure at 60°C was adopted

V. TESTS CONDUCTED

5.1 Compressive Strength Test

The compressive strength of the geopolymer concrete was tested as per IS 516:1959. Cube specimens of size 100 mm were cast for each proportion and tested for their compressive strength at the ages of 3, 7 and 28 days. All the specimens were steam cured and tested using Compression Testing Machine (CTM) of 1000 KN capacity under a uniform rate of loading of 140 Kg/Cm2/Min until failure and the ultimate load at failure was taken to calculate the compressive strength.

5.2 Split Tensile Strength Test

The split tensile strength test was carried out as per IS 5816:1999. Cylindrical concrete specimens of size 100 mm diameter and 200 mm height were cast. The specimens were steam cured and then tested for their splitting tensile strength using Universal Testing Machine (UTM) at the ages of 3, 7 and 28 days.

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5.3 Flexural Strength Test

The flexural strength of the geopolymer concrete was carried out as per IS 516:1959. Beams of size 75 mm × 75 mm × 450 mm size were cast, steam cured and and then subjected to the flexural strength test using Universal Testing Machine (UTM) at the ages of 3, 7 and 28 days.

VI. RESULTS AND DISCUSSION

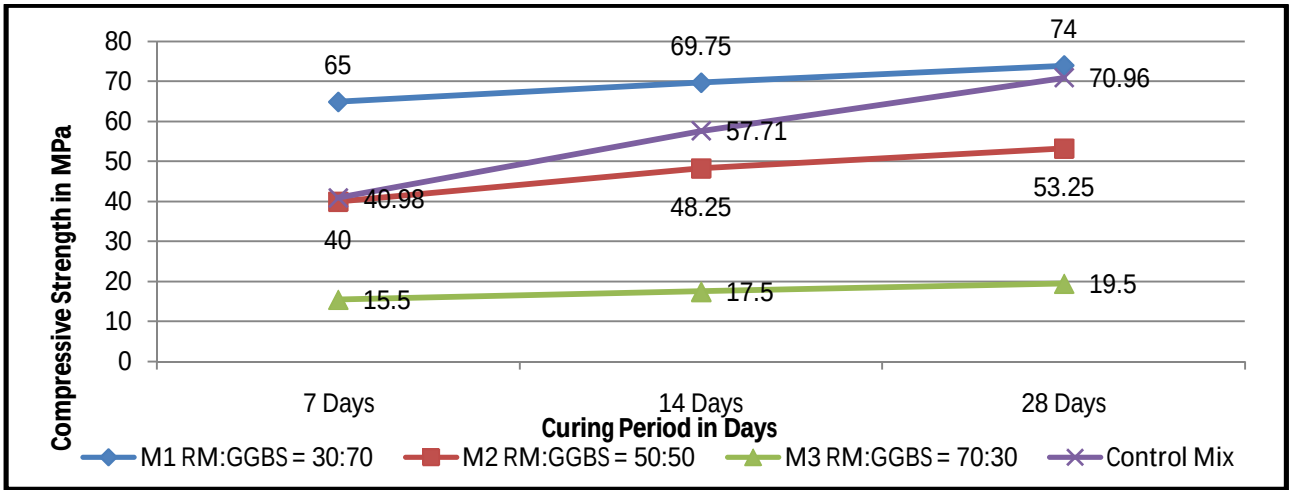
6.1 Compressive strength

The compressive strength test results of the Geopolymer concrete on 3, 7 and 28 days are shown in Table 4.The effect of RM and GGBS on the compressive strength of the Geopolymer concrete at different curing temperatures is discussed. Graphs 1 and 2 represent the variation of compressive strength of the specimens kept under Steam cured condition and Ambient cured temperature at different curing ages. Geopolymer concrete made with M60 was kept as the control specimen.

Mix	Steam Cured Cubes			Ambient Cured Cubes		
	7 Days	14 Days	28 Days	7 Days	14 Days	28 Days
M1	65	69.75	74	57.75	63.5	69
M2	40	48.25	53.25	38	45	52.3
M3	15.5	17.5	19.5	15.5	16	18
Control	40.98	57.71	70.96	53.46	56.41	63.76

Table 4: Compressive Strength of Steam Cured and Ambient Cured Mixes at different curing periods.

The initial 7 days strength is very low for M3 mix, just below par for M2 mix, whereas M1 mix shows that the early onset strength is very high (i.e., 65 MPa) in comparison to initial strength of Control mix (40.98MPa). At the curing age of 14 days, although the strength of M1 mix is dominant one but, the strength gain in Control Mix is greater in relation to all other mixes, this is evident by looking at the slope of all 4 lines. At the end of curing period (28 days curing) we can see that control mix has almost near to M1 mix, but M1 mix has slightly more final strength then control and other mixes. We can infer that RM:GGBS = 30:70 gives better compressive strength then M60 grade concrete and other two mixes.



Graph 1: Compressive strength of steam cured cubes

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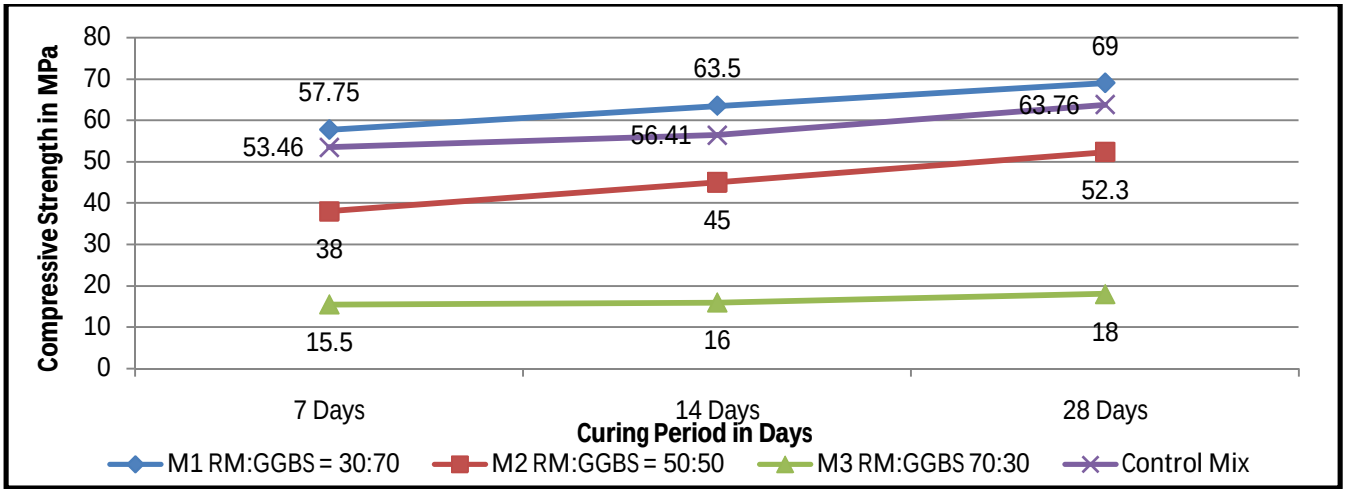

Graph 2: Compressive strength of cubes subjected to ambient curing

On testing cubes with 7 days curing age subjected to ambient curing we can see from graph that early onset strength of M1 mix is slightly higher than control mix and very high than M3 and nearly 1.5 times M2 mix. The strength values at 14 days curing and 28 days curing follow nearly same trend, M1 attains slightly higher final strength (69 MPa) in comparison to control mix (63.76 Mpa).

Looking at both the graphs, we can see that initial and final strengths between the two temperature shows that compressive strength is relatively higher in Steam cured specimen. It is observed that temperature is directly proportional to strength to certain extent. Mix M1 has better compressive strength at all stages of curing and temperature of curing with respect to control mix, looking at graph trends of M2 and M3 we can infer that steam curing aids in attain strength at all stages of curing.

6.2 Split tensile strength

The results of Split tensile strength test of the geopolymer concrete on 3, 7 and 28 days are tabulated in Table 5. Graphs 3 represent the variation of tensile strength of the specimens kept under Steam cured condition at different curing ages. Geopolymer concrete made with M60 was kept as the control specimen.

Mix	7 Days	14 Days	28 Days
M1	3.2	3.53	4.06
M2	2.87	3.15	3.59
M3	2.18	2.43	3.04
Control	2.81	3.75	4.68

Table 5: Split tensile Strength of Cylinders at different curing periods.

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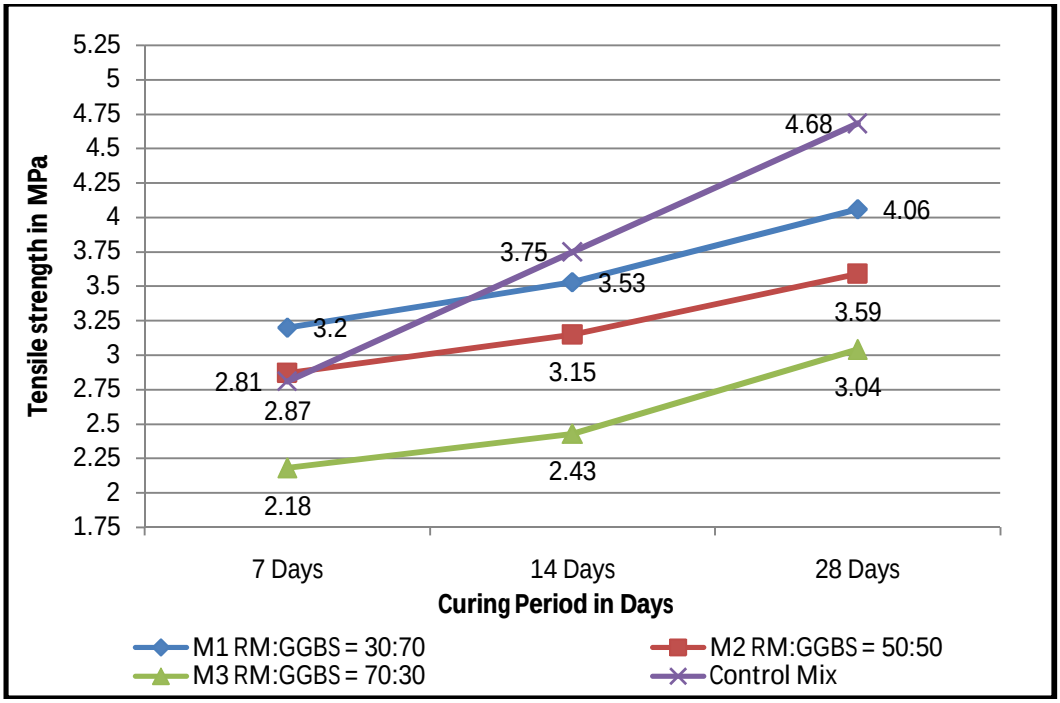

Graph 3: Split Tensile strength of Cylinders

From graph 3 we can see that the tensile strength of M3 mix is very low, M2 is nearly equal to control mix and M1 is nearly 85% more than control mix. At 14 days we can see that all three mixes gain strength gradually, whereas control mix has gained strength with a steady slope, surpassing M1 mix, which was initially greater. The final strength attained by control mix is 15% higher than M1 mix.
The trend set by graphs dictates that control mix although has low initial strength but keeps on gaining strength at a steady pace, whereas the other three mixes gain strength but at a slower pace.

6.3 Flexural strength

We can see various strengths of different mixes at different time intervals in Table 6. In this test, prisms we used to test the flexural strength of different mixes. The values have been plotted in graph 4 for 7,14 and 28 days

Mix	7 Days	14 Days	28 Days
M1	3.66	4.24	5.51
M2	2.96	3.95	4.88
M3	2.33	2.62	3.72
Control	5.11	5.22	5.74

Table 6: Split tensile strength of Prism at different curing periods

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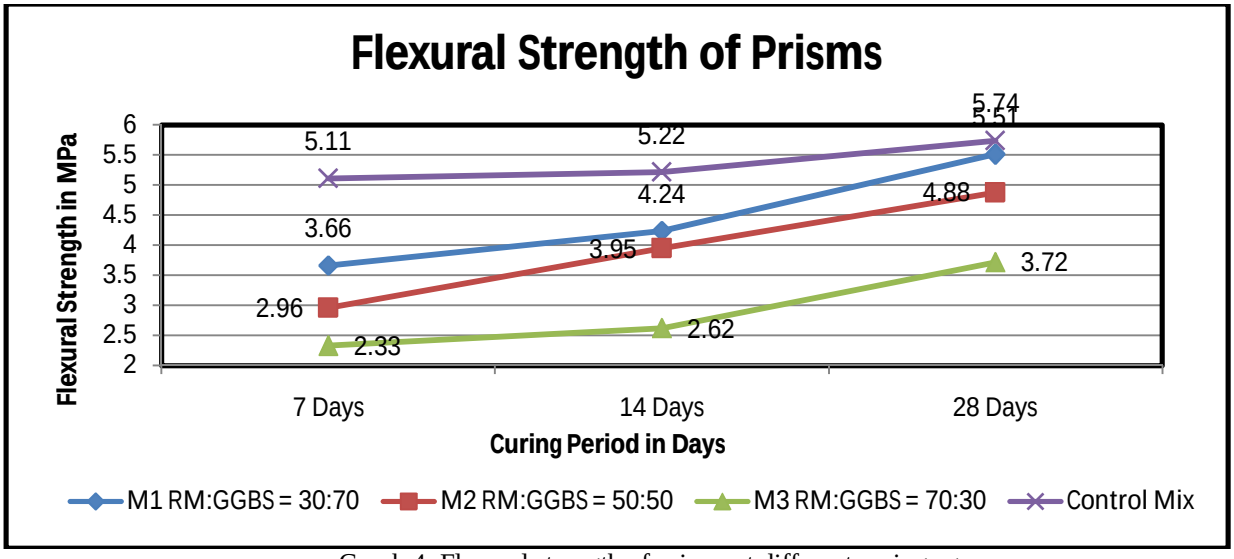

Graph 4: Flexural strength of prisms at different curing ages

In case of split tensile strength at least initial strength at M1 was greater than control mix, but in this flexural test we can see that the control mix dominates throughout its life in relation to all three mixes. Although M1 mix strives to attain strength at a faster pace and reach the mark of 5.51MPa, which is slightly less than value of control mix i.e, 5.74 Mpa. In this test the slopes of mixes M1, M2 and M3 is higher than control. We can notice that the strength gained during 14 to 28 days gap is high for geopolymer concrete.

VII. CONCLUSION

This paper presents preliminary study on the effect of addition of various percentages of red mud and GGBS on Geopolymer concrete. Various mechanical parameters were recorded for 7, 14 and 28 days for each test. Based on the resultes obtained from the tests following conclusions can be derived:

1. Irrespective of the percentage of Red mud and GGBS, the results of cube compressive strength of steam cured and ambient cured cube shows that the specimen with steam curing performed better then ambient dried cubes. It signifies that steam promotes geopolymerisation reactions.

2. M1 mix performs better in compressive strength and on par with split tensile strength but Control mix out weights in flexural strength. It signifies that Lower percentage of red mud (upto 30%) gives higher compressive strength, looking at the trend set by graph, we can observe that as percentage of red mud increases the strengths decrease (seen in M2 and M3 mix) at all stages of curing. In fact, Mix with 30:70 RM: GGBS gives slightly better results than M60 Grade concrete (Control Mix) in both the cases of curing.

3. Initial tensile strength of M1 and M2 mixes are better than control, but strength of M3 is very low. As time passes, Conventional concrete attains strength at a far faster pace compared to all other 3 Geopolymer mixes, and superseeds all of them at 14 days. At 28 days, the difference in Split tensile strength between control and M1 mix is around 18%. So we can conclude that, addition of redmud is not very effective in case of split tensile strength, as it performs below par to control mix.

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4. In case of flexural strength, the trend is similar to split tensile strength. M1 mix being the best among the 3 geopolymer mixes, has relatively low strength since 7 days (nearly 40%) and attains strength until 28 days, which is just 5% less then control mix.

5. We can conclude that addition of red mud upto 30% and GGBS upto 70% is fine for compressive strength, but any other further increase In addition of Red mud and/or GGBS leads to declination of strength in all mechanical parameters.

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