Experimental Study on Metakaolin and GGBS Based Geopolymer Concrete

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Abstract-Construction industry is dominated by new materials which are ecologically violable and feasible solution for ever growing architectural industry. Effort are in progress all over the world to develop environment friendly construction materials which minimizes the utility of natural resources and helps to decrease green house gas emissions in to the atmosphere. The green house gas releases in the atmosphere is increasing day by day due to ordinary Portland cement production. In this connection, Geopolymer is in need, where the binders used in the production of geopolymer concrete is inorganic polymers. Geopolymer concrete will be introduced as an alternative concrete which did not use any cement in its mixture and used Metakaoline and GGBS as alternative cement. NaOH and Na₂SiO₃ were used as activator solution. The fixed ratio of sodium silicate to sodium hydroxide is 2.5 and the concentration of sodium hydroxide is 8 Molar. The geopolymer concrete specimens are casted and tested in the laboratory for compressive strength, Split Tensile Strength and Flexural Strength for 3 Days, 7 Days and 28 days and cured at ambient temperature. This study helps in gaining knowledge about the morphological composition of geopolymer concrete which might result in path-breaking trends in research and construction industry.

Keywords - Geopolymer Concrete, Alkali Activators, Compressive Strength, Split Tensile Strength, Flexural Strength.

I. INTRODUCTION

Concrete is one of the most widely used construction material. Portland cement production is a major contributor to carbon dioxide emissions. The global warming is caused by the emission of greenhouse gases, such as carbon dioxide, to the atmosphere by human activities. Among the greenhouse gases, carbon dioxide contributes about 65% of global warming. Many efforts are being made in order to reduce the use of Portland cement in concrete. These efforts include the utilization of supplementary cementing materials such as fly ash, silica fume, granulated blast furnace slag, rice-husk ash and metakaolin, and finding alternative binders to Portland cement. In terms of reducing the global warming, the geopolymer technology could reduce the carbon-dioxide emission to the atmosphere caused by Cement about 80% [1]. In this paper, the effort was made to study the strength parameters of geopolymer concrete with GGBS and Metakaoline [2-3].

There are two main constituents of geopolymers, namely the source materials and the alkaline liquids. The source materials for geopolymers based on alumina-silicate should be rich in silicon (Si) and aluminium (Al). These could be natural minerals such as kaolinite, clays, etc. Alternatively, by-product materials such as fly ash, GGBS, etc could be used as source materials. The choice of the source materials for making geopolymers depends on factors such as availability, cost, type of application, and specific demand of the end users. The alkaline liquids are from soluble alkali metals that are usually sodium or potassium based. The most common alkaline liquid used in geo polymerization is a combination of sodium hydroxide (NaOH) and sodium silicate [4-7].

1.1 Origin of term ‘Geopolymer’

The term “Geopolymer” was first introduced to the world by Davidovits of France resulting in a new field of research and technology. Geopolymer also known as ‘inorganic polymer’ has emerged as a ‘green’ binder with wide potentials for manufacturing sustainable materials for environmental, refractory and construction applications [8].

1.2 Need for the Study

- To find an alternative for the ordinary Portland cement.
- To reduce CO₂ emission and produce eco-friendly concrete.
- To provide high strength concrete than ordinary Portland concrete.
1.3 Objectives of Present Study

- To make a concrete without using cement (i.e. Geopolymer concrete).
- To study the different Mechanical properties of geopolymer concrete with varying percentages of GGBS and Metakaolin.

II. MATERIALS

2.1 Metakaolin

It is obtained from the calcination of kaolinitic clay at temperatures ranging from 500 - 750°C. The further calcination of kaolinite at higher temperatures leads to the formation of more ordered crystalline phases, such as spinnel, mullite and cristobalite. It is suggested that firing kaolinite at lower temperatures (< 500°C) does not give sufficient energy to break the crystalline structure of kaolinite. As a result, amorphous metakaolinite is not formed. However, calcination at higher temperatures, i.e. higher than a threshold temperature turns the metastable phase, metakaolinite, into more ordered crystalline phases, which are non-reactive upon alkali-activation. The high amorphicity of metakaolinite leads to the high reactivity when it is activated in alkali solutions. MK has the smallest particle size in comparison to FA or GGBS. The fine and irregular particle shape of MK often mean that MK generally requires more solution for wetting and reaction to take place appropriately [9].

Table 1. Physical Properties of Metakaolin

| Property                        | Value     |
|---------------------------------|-----------|
| Colour                          | Pink / Off-white |
| Pozzolan Reactivity mg Ca(OH)₂/gm | 900       |
| Average Particle size           | 1.4 micron |
| Brightness (ISO)                | 75 ± 2    |
| Bulk Density (gms/Ltr)          | 320 to 370|
| Specific Gravity                | 2.5       |

Table 2. Chemical Properties of Metakaolin

| Al₂O₃                   | >39.0%    |
|------------------------|-----------|
| Fe₂O₃                   | <0.8%     |

2.2 Ground Granulated Blast Furnace Slag (GGBS)

GGBS is a by product of the manufacturing of iron in a blast furnace where iron ore, limestone and coke are heated up to 1500°C.When these materials melt in the blast furnace, two products are produced i.e. molten iron, and molten slag. The molten slag is lighter and floats on the top of the molten iron. The molten slag comprises mostly silicates and alumina from the original iron ore, combined with some oxides from the limestone. The process of granulating the slag involves cooling the molten slag through high pressure water jets. This rapidly quenches the slag and forms granular particles generally not larger than 5mm in diameter. The rapid cooling prevents the formation of larger crystals, and the resulting granular material comprises some 95% non-crystalline calcium alumino silicates. The granulated slag is further processed by drying and then ground to a very fine powder, which is GGBS (ground granulated blast furnace slag) cement it is another excellent cementitious material [10-11].
Wainwright and Ait-Aider (1995) examined the influence of the composition of OPC and the addition of up to 70% GGBS on the bleed characteristics of concrete and conclude that the partial replacement of OPC with 40% and 70% of GGBS. GGBS led to increases in the bleeding of the concretes, like fly ash, also GGBS can improve many mechanical and durability properties of concrete and it generates less heat of hydration [12 – 14].

Babu and Kumar (2000) determined the cementitious efficiency of GGBS in concrete at various replacement percentages (10–80%) through the efficiency concept by establishing the variation of strength to water-to-cementitious materials ratio relations of the GGBS concretes from the normal concretes at the age of 28 days. The 28-day compressive strength of concretes containing GGBS up to 30% replacement were all slightly above that of normal concretes [15], and at all other percentages, the relationships were below that of normal concretes. It was also observed that the variations due to the different percentages of slag replacement were smaller than the corresponding variations in the case of fly ash [16 - 17]. The result showed that the slag concretes based on overall efficiency factor (k), will need an increase of 8.6% for 50% replacement and 19.5% for 65% replacement in the total cementitious materials for achieving strength equivalent to that of normal concrete at 28 days [18].

### Table 3. Physical Properties of GGBS

| Parameter            | GGBS  | IS : 12089 – 1987 |
|----------------------|-------|-------------------|
| CaO                  | 37.34%| ---               |
| Al₂O₃                | 14.42%| ---               |
| Fe₂O₃                | 1.11% | ---               |
| SiO₂                 | 37.73%| ---               |
| MgO                  | 8.71% | Max. 17%          |
| MnO                  | 0.02% | Max. 5.5%         |
| Sulphide Sulphur     | 0.39% | Max. 2%           |
| Loss of Ignition     | 1.41% | ---               |
| Insoluble Residue    | 1.59% | Max. 5%           |
| Glass Content (%)    | 92%   | Min. 85%          |

### 2.3 Coarse Aggregate

Coarse aggregate are a broad category particulate inert materials used in construction. Hard stones are crushed to the required size and are used as coarse aggregate. The material that is retained on an IS sieve of size 4.75 is called coarse aggregate. Aggregate of essentially the same nominal maximum size and grading will produce concrete of satisfactory workability. These aggregates are bound together by the cement and fine aggregate in the presence of water to form concrete [19-21]. Coarse aggregates of sizes 12mm and 20mm and fine aggregate taken from a local supplier are used in the present study and the properties as shown in Table 4.

### Table 4. Physical Properties of Coarse Aggregate

| Sieve Size (mm) | Requirement as per IS: 383-1970 | Percentage passing | Requirement as per IS:383-1970 | Percentage passing |
|-----------------|---------------------------------|--------------------|---------------------------------|--------------------|
| 20 mm           |                                 |                    | 12 mm                          |                    |
| 80.00           | ----                            | ----               | ----                            | ----               |
| 63.00           | ----                            | ----               | ----                            | ----               |
| 40.00           | 100 %                           | 100 %              | ----                            | ----               |
| 20.00           | 85 – 100 %                      | 94.60 %            | ----                            | ----               |
| 16.00           | ----                            | ----               | 100 %                          | 100 %              |
| 12.50           | ----                            | ----               | 85-100 %                       | 93.4 %             |
| 10.00           | 0 – 20 %                        | 14.30 %            | 0 – 45 %                       | 39.23 %            |
| 4.75            | 0 – 05 %                        | 2.85 %             | 0 – 10 %                       | 6 %                |
| 2.36            | ----                            | ----               | ----                            | ----               |
| Specific gravity| 2.82                            | -                  | 2.79                            |
| Water Absorption%| 0.40                           | -                  | 0.48                            |
| Aggregate Impact Value | 12 %                           | -                  | 13 %                            |
| Bulk Density (kg/m³)| 1660                           | -                  | 1655                            |
| Flakiness       | 14 %                            | -                  | 15 %                            |
| Elongation      | 15 %                            | -                  | 16 %                            |
2.4 Fine Aggregate

Fine aggregate should consist of natural sand or crushed stone sand. It should be hard, durable and clean and be free from organic matter etc. Fine aggregate should not contain any appreciable amount of clay balls and harmful impurities such as alkalis, salts, coal, decayed vegetation etc[22]. The silt contents should not exceed 4% [23].

| I.S. Sieve (mm) | Percentage Passing through I.S. Sieve | Percentage Passing I.S. Sieve as per IS 383 |
|----------------|--------------------------------------|--------------------------------------------|
| 10             | 100                                  | 100                                        |
| 4.75           | 99.6                                 | 90-100                                     |
| 2.36           | 99                                   | 75-100                                     |
| 1.18           | 92.6                                 | 55-90                                      |
| 600 micron     | 48.6                                 | 35-50                                      |
| 300 micron     | 8.2                                  | 8-30                                       |
| 150 micron     | 2                                    | 0-10                                       |
| Zone           | II                                   |                                             |

Fineness modulus = 2.2
Specific Gravity = 2.50
Bulk Density =1625 Kg/m³
Bulking of Sand = 23%
Silt Content = 0.25%

2.5 Sodium Hydroxide

The most common alkaline activator used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate. The type and concentration of alkali solution affect the dissolution of fly ash. Leaching of Al³⁺ and Si⁴⁺ ions are generally high with sodium hydroxide solution compared to potassium hydroxide solution. Therefore, alkali concentration is a significant factor in controlling the leaching of alumina and silica from fly ash particles, subsequent geo polymerization and mechanical properties of hardened geopolymer[16]. In presence of NaOH in the activating solution the reaction proceeds more rapidly and the gel is less smooth. The gel composition analysed in the sample activated with the mixture of sodium silicate and NaOH is enriched in Na and Al [24].

2.6 Sodium Silicate

Palomo et al (1999) concluded that the type of activator plays an important role in the polymerization process. Reactions occur at a high rate when the alkaline activator contains soluble silicate, either sodium or potassium silicate, compared to the use of only alkaline hydroxides. A study conducted by Xu and Van Deventer showed that the addition of sodium silicate solution to the sodium hydroxide solution as the alkaline activator enhanced the reaction between the source material and the solution[21]. Tempest et al (2009) state that the sodium silicate activator dissolves rapidly and begins to bond fly ash particles. Open porosity can be observed and is rapidly filled with gel as soon as the liquid phase is able to reach the ash particles. The liquid phase is important as a fluid transport medium permitting the activator to reach and react with the fly ash particles [21], [31 – 32].

2.7 Casting Procedure

Generally the fine aggregate, coarse aggregate and fly ash are weighed to the required quantities and then they are mixed in dry condition foe 2-3 minutes and then the alkaline solutions prepared (combination of sodium hydroxide and sodium silicate) are to be taken to required quantity is added to the dry mix. This mixing is done for 5-7 minutes in the mixer for proper bonding of all the materials [21, 25]. After the mixing is done the mix is filled in the cube moulds of size 150mm x 150mm X 150mm in 3 layers with equal compacting and the cubes are kept on a vibrating table so that no voids are formed [26].

III. EXPERIMENTAL DETAILS

3.1 Experimental Procedure

3.1.1 Mix Design of Geopolymer Concrete

In the design of geopolymer concrete (GPC mix), coarse and fine aggregates together were taken as 75% of entire mixture by mass. This value is similar to that used in OPC concrete in which it will be in the range of 75% to 80% of the entire mixture by mass. Fine aggregate was taken as 30.8% of the total aggregates1. From the past literatures it is clear that the average density of Cementitious materials based geopolymer concrete is similar to that of OPC concrete (2400kg/m³) [22]. Knowing the density of concrete, the combined mass of alkaline liquid and cementitious materials can be arrived. By assuming the ratios of alkaline liquid to cementations materials as 0.45, mass of metakaolin and mass of alkaline liquid was found out. To obtain mass of sodium hydroxide and sodium silicate solutions, the ratio of sodium silicate solution to sodium hydroxide solution was fixed as 2.5. Extra water (other than the water used for the preparation of alkaline solutions) used respectively to achieve workable concrete [1,20]. The mix proportion for geopolymer concrete is given in Table 6 and the different combinations of GGBS and Metakaolin investigated are given in Table 7.
Table 6. Mix Proportions

| Materials Used | Cementitious Materials | Fine Aggregate | Coarse Aggregate | Sodium Hydroxide | Sodium Silicate |
|----------------|------------------------|----------------|------------------|------------------|----------------|
| Quantity of materials in kg/m³ | 414 | 660 | 1136 | 53 | 133 |

Table 7. Combinations of GGBS and Metakaolin

| Mix ID | Metakaolin (%) | GGBS (%) |
|--------|----------------|----------|
| M1     | 100            | 0        |
| M2     | 90             | 10       |
| M3     | 80             | 20       |
| M4     | 70             | 30       |
| M5     | 60             | 40       |
| M6     | 50             | 50       |
| M7     | 40             | 60       |
| M8     | 30             | 70       |
| M9     | 20             | 80       |
| M10    | 10             | 90       |
| M11    | 0              | 100      |

3.2 Preparation of Geopolymer Concrete

To prepare 8 molarity concentration of sodium hydroxide solution, 320 grams (molarity x molecular weight) of sodium hydroxide flakes was dissolved in distilled water and makeup to one litre. The sodium hydroxide solution thus prepared is mixed with sodium silicate solution one day before mixing the concrete to get the desired alkaline solution. The solids constituents of the GPC mix i.e. Metakaoline, GGBS and the aggregates were dry mixed in the mixer for about three minutes. After dry mixing, alkaline solution was added to the dry mix and wet mixing was done for 4 minutes [1, 20]. Finally extra water was added to achieve workable GPCC mix [17].

In this experimental work a total of 297 numbers of Geopolymer concrete specimens were cast with various mix ratios as shown in Figure 1. The specimens are of 150 mm side. Before casting machine oil was smeared on the inner surfaces of the cast iron mould. Concrete was poured into the moulds and compacted thoroughly using a tamping rod. The top surface was finished using a trowel. The GPC specimens were removed from the mould after 1 or 2 days based on setting of specimens. The specimens were left at room temperature till the day of testing. Compressive strength test was conducted using a 3000kN Compression testing machine. The test was conducted as per the relevant Indian standard specifications [1], [26 – 27].

Table 8. Details of Test Specimen

| S. No. | Name of Test                  | Size of the Specimen (mm) | No. of Specimen |
|--------|-------------------------------|---------------------------|-----------------|
| 1      | Compressive Strength Test     | 150 x 150 x 150           | 99              |
| 2      | Split Tensile Test            | 150 x 300                 | 99              |
| 3      | Flexural Strength Test        | 500 x 100 x 100           | 99              |
|        | Total                         |                           | 297             |

IV. RESULTS AND DISCUSSIONS

4.1 Compressive strength

The compressive strength of Metakaolin based geopolymer concrete at the age of 3 days 7 days and 28 days are presented in the Figure -3

Table 9. Compressive Strength (MPa)

| Mix ID | M1    | M2    | M3    | M4    | M5    | M6    | M7    | M8    | M9    | M10   | M11   |
|--------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 3 Days | 6.01  | 9.46  | 12.03 | 14.77 | 19.17 | 19.76 | 24.96 | 30.51 | 31.68 | 33.75 | 40.50 |
| 7 Days | 8.68  | 12.83 | 15.08 | 20.56 | 22.02 | 28.27 | 30.98 | 33.06 | 37.55 | 38.42 | 45.31 |
| 28 Days| 13.17 | 16.36 | 20.32 | 24.13 | 26.01 | 29.67 | 33.84 | 38.50 | 40.22 | 42.68 | 48.04 |
Compressive strength test on cubes is the most common test conducted on hardened concrete because it is an easy test to perform and most of the desirable properties of concrete are comparatively related to its compressive strength. The compression test was carried out on cubical specimen of size 150mm in a compression testing machine of capacity 3000 kN. The strength is determined at 3, 7 and 28 days of casting [28]. The variation of compressive strength at the age of 7th and 28th days with optimum percentage of GGBS and Metakaolin were given below in Table 10. From the test results, it was observed that the maximum compressive strength was obtained for mix M11 with 100% GGBS and least compressive strength is obtained to 100% Metakaolin and it is also it is observed that the percentage increase in strength from 7 days to 28 days is more in mix M8 i.e. 70% GGBS and 30% Metakaoline [25],[31-32].

### 4.2 Split Tensile Strength

Split Tensile Strength of concrete cylinders 150mm diameter and 300mm long were tested as per the procedure explained in IS 5816 [28].

| Mix ID | M1 | M2 | M3 | M4 | M5 | M6 | M7 | M8 | M9 | M10 | M11 |
|--------|----|----|----|----|----|----|----|----|----|-----|-----|
| 3 Days to 7 days (%) | 30.81 | 26.26 | 20.24 | 28.15 | 12.95 | 30.07 | 19.42 | 7.70 | 15.64 | 12.14 | 10.63 |
| 7 Days to 28 days (%) | 34.10 | 21.60 | 25.75 | 14.79 | 15.35 | 4.72 | 8.46 | 14.15 | 6.64 | 9.99 | 5.68 |

Table 10. Percentage Change in Compressive Strength

| Mix ID | M1 | M2 | M3 | M4 | M5 | M6 | M7 | M8 | M9 | M10 | M11 |
|--------|----|----|----|----|----|----|----|----|----|-----|-----|
| 3 Days | 1.23 | 1.26 | 1.37 | 1.77 | 2.11 | 2.6 | 3.2 | 4.2 | 4.4 | 5.4 | 6.03 |
| 7 Days | 1.30 | 1.31 | 1.52 | 1.90 | 2.15 | 2.6 | 3.81 | 4.4 | 4.65 | 5.61 | 6.53 |
| 28 Days | 1.37 | 1.41 | 1.55 | 1.96 | 2.21 | 2.94 | 4.14 | 4.53 | 4.9 | 5.87 | 6.8 |

Table 11. Split Tensile Strength (MPa)
The values of split tensile strength of cylindrical specimens subjected to ambient curing conditions. The variation of split tensile strength at the age of 28th days with optimum percentage of GGBS and metakaolin were given below. It was observed that the maximum split tensile strength was obtained for mix M11. Also, the split tensile strength decreases with increase in Metakaoline [28].

### 4.3 Flexural Strength

#### Table 12. Flexural Strength (MPa)

| Mix ID | M1   | M2   | M3   | M4   | M5   | M6   | M7   | M8   | M9   | M10  | M11  |
|--------|------|------|------|------|------|------|------|------|------|------|------|
| 3 Days | 0.31 | 0.41 | 0.62 | 0.73 | 0.69 | 0.78 | 1.12 | 1.01 | 1.33 | 1.56 | 2.15 |
| 7 Days | 0.33 | 0.59 | 0.63 | 0.93 | 0.75 | 0.94 | 1.28 | 1.48 | 1.66 | 2.07 | 2.65 |
| 28 Days| 0.44 | 0.65 | 0.68 | 1.01 | 0.98 | 1.04 | 1.33 | 1.55 | 1.71 | 2.31 | 2.97 |

The results of flexural strength of concrete at the age of 3, 7 and 28 days are presented in Table 12. The variations in flexural strength at the age of 28 days with different percentage of GGBS and metakaolin were plotted. From the test results, it was observed that when the percentage of GGBS increases, the flexural strength of concrete also increases. On the contrary, the strength decreases when the percentage of metakaolin increases [28-30].
V. CONCLUSION

The conclusions based on the limited observations from the present investigation on properties of fresh and hardened metakaolin and GGBS based concrete are:

1. Workability of geopolymer concrete decreased as the metakaolin content increases with GGBS. But increase in GGBS does not affect the workability.

2. Mechanical properties such as compressive strength, split tensile strength and flexural strength shows increasing trend with the decrease of metakaolin.

3. Mix with 30% of metakaolin and 70% of GGBS and seems to have good compressive, split and flexural strengths, this may be due to increase in alkaline reaction between GGBS particles and calcium in Metakaoline.

4. Nearly 90% of total strength of GPC is achieved within the age of 7days.

5. Then increase in strength of GPC between 7days and 28days appeared to be high when compared with 3days and 7days. It shows that even after 7days geopolymer reaction is taking place but at a higher rate.

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