

# MODIFIED GUIDELINES FOR GEOPOLYMER CONCRETE MIX DESIGN USING INDIAN STANDARD

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## ABSTRACT

This experimental study is intended to identify the mix ratios for different grades of Geopolymer Concrete by trial and error method. A new Design procedure was formulated for Geopolymer Concrete which was relevant to Indian standard (IS 10262-2009). The applicability of existing Mix Design was examined with the Geopolymer Concrete. Two kinds of systems were considered in this study using 100% replacement of cement by ASTM class F flyash and 100% replacement of sand by M-sand. It was analyzed from the test result that the Indian standard mix design itself can be used for the Geopolymer Concrete with some modification.

**Keywords:** Alumina-silicate; fly ash; geopolymer concrete; M-sand; mix design.

## 1. INTRODUCTION

Geopolymers are inorganic polymeric materials with a chemical composition similar to zeolites but possessing an amorphous structure. Geopolymers may be seen as man-made rocks. They can be produced by reacting solid aluminosilicates with a highly concentrated aqueous alkali hydroxide or silicate solution. The chemistry and terminology of inorganic polymers was first discussed in detail by Davidovits [1]. Since the first mention of the term 'geopolymer' by Davidovits [1], Geopolymers form three-dimensional disordered frameworks of the tecto-aluminosilicate type with the general empirical formula  $Mn[-(SiO_2)_z-AlO_2]_n \cdot wH_2O$ , in which  $n$  is the degree of polycondensation, and  $M$  is predominantly a monovalent cation ( $K^+$ ,  $Na^+$ ), although  $Ca^{2+}$  may replace two monovalent cations in the structure [2].

The present work is carried out in the framework of a project aims to produce the geopolymeric Mix procedure of different grade of geopolymer concrete matrices, stronger and denser equal to the cement concrete obtained by using Portland Cement binders, that can be

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used for the long term stabilization of inorganic toxic waste i.e. flyash. The particular work presented in this paper deals with a study investigating the Mix design.

## 2. MATERIALS USED

Cementitious material used in this Experimental programme was low calcium Flyash (ASTM type F) [2]. The specific gravity Fly Ash was 2.3. The Specific gravity and Fineness modules for manufactured sand were 3.1 and 2.15 respectively. The chemical composition for cementitious material is shown in table 3. Locally available crushed granite stone aggregate of size 20 mm passing and retained in 10 mm, was used and the specific gravity and fineness modulus for the same are 2.6 and 6.4 as per IS: 2386- 1968 Part III. Both the Aggregates complied with the requirements of IS: 383-1970. Specific gravity of NaOH and  $\text{Na}_2\text{SiO}_3$  solutions were 1.47 and 1.6 respectively.

### 2.1 M-SAND

M-sand is crushed aggregates produced from hard granite stone which is cubically shaped with ground edges, washed and graded with consistency to be used as a substitute of river sand. M-Sand is superior quality manufactured sand with international standards [3].

### 2.2 Sodium hydroxide

Generally the sodium hydroxides are available in solid state by means of pellets and flakes. The cost of the sodium hydroxide is mainly varied according to the purity of the substance. Since our geopolymer concrete is homogenous material and its main process to activate the sodium silicate, so it is recommended to use the lowest cost i.e. up to 94% to 96% purity. In this investigation the sodium hydroxide pellets were used. Whose physical and chemical properties are given by the manufacturer is shown in Table 1 and 2.

Table 1: Physical Properties Sodium hydroxide

Colour	Colour less
Specific Gravity	
20%	1.22
30%	1.33
40%	1.43
50%	1.53

### 2.3 Sodium silicate

Sodium silicate is also known as water glass or liquid glass, available in liquid (gel) form. In present investigation sodium silicate 2.0 (ratio between  $\text{Na}_2\text{O}$  to  $\text{SiO}_2$ ) is used. As per the manufacture, silicates were supplied to the detergent company and textile industry as bonding agent. Same sodium silicate is used for the making of geopolymer concrete. The chemical

properties and the physical properties of the silicates are given the manufacture is shown in Table 3.

Table 2: Chemical properties sodium hydroxide

Assay	97%	Min
Carbonate (Na <sub>2</sub> CO <sub>3</sub> )	2%	Max
Chloride (Cl)	0.01%	Max
Sulphate (SO <sub>2</sub> )	0.05%	Max
Lead (Pb)	0.001%	Max
Iron (Fe)	0.001%	Max
Potassium (K)	0.1%	Max
Zinc (Zn)	0.02%	Max

#### 2.4 Alkaline liquid

Generally alkaline liquids are prepared by mixing of the sodium hydroxide solution and sodium silicate at the room temperature. When the solution mixed together the both solution start to react i.e. (polymerisation takes place) it liberate large amount of heat so it is recommended to leave it for about 24 hours thus the alkaline liquid is get ready as binding agent [4].

#### 2.5 Preparation of alkaline liquids

##### 2.5.1 Sodium hydroxide

Sodium hydroxide pellets are taken and dissolved in the water at the rate of 16 molar concentrations. It is strongly recommended that the sodium hydroxide solution must be prepared 24 hours prior to use and also if it exceeds 36 hours it terminate to semi solid liquid state. So the prepared solution should be used within this time.

##### 2.5.2 Molarity calculation

The solids must be dissolved in water to make a solution with the required concentration. The concentration of Sodium hydroxide solution can vary in different Molar. The mass of NaOH solids in a solution varies depending on the concentration of the solution.

For instance, NaOH solution with a concentration of 16 Molar consists of  $16 \times 40 = 640$  grams of NaOH solids per litre of the water, were 40 is the molecular weight of NaOH. Note that the mass of water is the major component in both the alkaline solutions.

The mass of NaOH solids was measured as 444 grams per kg of NaOH solution with a Concentration of 16 Molar. Similarly, the mass of NaOH solids per kg of the solution for other concentrations was measured as 10 Molar: 314 grams, 12 Molar: 361 grams, and 14 Molar: 404 grams [4].

Table 3: Physical and chemical properties sodium silicate

Chemical formula	Na <sub>2</sub> O x SiO <sub>2</sub> Colour less
Na <sub>2</sub> O	15.9%
SiO <sub>2</sub>	31.4%
H <sub>2</sub> O	52.7%
Appearance	Liquid (Gel)
Colour	Light yellow liquid (gel)
Boiling Point	102 C for 40% aqueous solution
Molecular Weight	184.04
Specific Gravity	1.6

## 2.6 Experimental programme

### 2.6.1 Testing the concrete specimens

Twenty five 150×150mm cubes and 150mm diameter 300mm high were cast out of which three cubes each were used to determine the compressive strength and three cylinders each were used to determine the split tensile strength at 24 hours for different grades of Geopolymer Concrete. A total of 300 numbers of specimens were tested in this study to find out the optimized grades of Geopolymer Concrete incorporating M-sand. Optimized mix ratios were shown in Table 4. All geopolymer concrete were made with mix design procedure using IS 10262-2009 [5]. It is recommended to have necessary precaution on workers because of acidic nature of the concrete. The aggregates were prepared in saturated-surface-dry (SSD) condition. Geopolymer concrete can be manufactured by adopting the conventional techniques used in the manufacture of Portland cement concrete. Then the components of concrete ingredients are collected and mixed with the pan mixture for about 5 min. Then Alkaline liquid were then added to the mixture and the mixing was done for another 5 min. After the mixing, the flow value of fresh geopolymer concrete was determined in accordance with slump test IS 516-1959. The fresh concrete could be handled up to 120 minutes without any sign of setting and without any degradation in the compressive strength. Flow test (workability) was carried out by slump cone test as described for cement concrete. After the flow test, fresh concrete were placed in respected mould as described in the IS 516-1959. The fresh concrete was cast and compacted by the usual methods used in the case of Portland cement concrete. The specimens were left standing for 1 hour and then cured at 60°C in the curing chamber for about 24 hours. Demoulding was done at 24 hours at the time of curing age. After the curing period the specimens left at the room temperature for about an hour and ready for testing. Thus the compressive strengths and tensile strength of concrete were tested at the same day in accordance with IS 516-1959 [6]. The reported strengths were the average of the three specimens shown in Table 5.

Table 4: Shows the optimised mix proportions for M-sand

Content	M 1 kg/m <sup>3</sup>	M 2 kg/m <sup>3</sup>	M 3 kg/m <sup>3</sup>	M 4 kg/m <sup>3</sup>	M 5 kg/m <sup>3</sup>	M 6 kg/m <sup>3</sup>
Fly ash	364.9	419.7	482.6	555.0	638.2	483.7
Coarse aggregate	1049.5	1018.0	987.5	957.9	929.1	882.2
Water	36.5	44.2	44.2	44.2	44.2	14.2
NaOH solution	52.7	68.5	68.5	68.5	68.5	89.8
Na <sub>2</sub> SiO <sub>3</sub>	184.5	171.1	171.1	171.1	171.1	224.6
<b>M-sand</b>	613	608.1	589.9	572.2	615.6	652.1
Content	M 7 kg/m <sup>3</sup>	M 8 kg/m <sup>3</sup>	M 9 kg/m <sup>3</sup>	M 10 kg/m <sup>3</sup>	M 11 kg/m <sup>3</sup>	M12 kg/m <sup>3</sup>
Fly ash	554.7	483.7	554.7	554.7	364.9	447.0
Coarse aggregate	832.8	882.2	832.8	832.8	964.0	907.5
Water	12.7	28.3	14.2	28.3	14.2	28.3
NaOH solution	103.0	82.2	89.8	82.2	83.0	89.8
Na <sub>2</sub> SiO <sub>3</sub>	257.5	205.5	224.6	205.5	207.3	224.6
<b>M-sand</b>	615.6	652.1	615.6	615.6	713	670.8

Table 5: Test results for manufactured sand

Mix	Compressive strength (N/mm <sup>2</sup> )	Tensile strength (N/mm <sup>2</sup> )
M1	21	1.592
M2	25	2.865
M3	22	2.865
M4	24	2.865
M5	24	2.865
M6	33	2.865
M7	20	2.4
M8	22	2.4
M9	21	2.65
M10	23	2.73
M12	20	2.73

M13	21	2.865
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### 3. DESIGN OF GEOPOLYMER CONCRETE MIXTURES

#### 3.1 Data for mix design

The following basic data are required to be specified for design of a concrete mix:

- Characteristic compressive strength of Geopolymer Concrete at 24 hours curing at the temperature of 60°C (f<sub>ck</sub>).
- Maximum size and Type of Fine aggregate and Coarse Aggregate to be used.
- Specific gravity of ingredients of concrete.
- Selection of Alkaline liquid, Flyash Ratio to the Compressive ratio.

Aggregates size, grading, surface texture, shape and other characteristics may produce secreted of different compressive strength for the same tiled ratio, the relationship between strength and free Alkaline liquid, Extra Water, Fly ash Ratio should preferably be established for the materials [7,8,9].

#### 3.2 Estimation of air content

Approximate amount of entrapped air to be expected in normal (non-air-entrained) concrete is given in Table 6.

Table 6: Approximate of Air Content

Nominal maximum size of aggregate mm	Entrapped air, as percentage of volume of concrete
10	3
20	2

For the desired workability, the quantity of mixing per unit volume of concrete and the ratio of fine aggregate to total aggregate by absolute volume are to be estimated from Tables as applicable, depending upon the nominal maximum size and type of aggregates.

Table 7: Approximate sand per cubic metre of concrete

Nominal size of coarse aggregate	Sand as percentage of total aggregate by absolute volume
10mm	40
20mm	35

Table 8: Adjustment of values in and sand percentage for zone factor i.e. fineness modulus

Change in condition	Adjustment in sand percentage
For sand conforming to grading Zone I, Zone III or Zone IV of	+1.5 percent for Zone I - 1.5 percent for Zone III

Table 4 of IS : 383-1970\*

3.0 percent for Zone IV

**3.3 Calculation of aggregate content**

The total aggregate content per unit volume of concrete may be calculated from the following equations:

$$V = \left[ \frac{SO}{S_{so}} + \frac{S}{S_s} + \frac{F}{S_F} + \frac{1}{P} \frac{F_a}{SF_a} \right] \times \frac{1}{1000} \text{ and}$$

$$V = \left[ \frac{SO}{S_{so}} + \frac{S}{S_s} + \frac{F}{S_F} + \frac{1}{1-P} \frac{C_a}{SC_a} \right] \times \frac{1}{1000}$$

Where,

- $V$  = Absolute volume of fresh concrete, which is equal to gross volume minus the volume of entrapped air.
- $S$  = Sodium Silicate Solution (kg) per m<sup>3</sup> of concrete.
- $SO$  = Sodium Hydroxide Solution (kg) per m<sup>3</sup> of concrete
- $F$  = Weight of Flyash (kg) per of m<sup>3</sup> of concrete
- $S_F$  = Specific gravity of Flyash
- $P$  = Ratio of fine aggregate to total aggregate by absolute volume
- $F_a, C_a$  = Total masses of fine aggregate and coarse aggregate (kg) per m<sup>3</sup> of concrete respectively
- $SF_a, SC_a$  = Specific gravity of saturated surface dry fine aggregate and coarse aggregate respectively.
- $S_s$  = Specific gravity of Sodium Silicate solution.
- $S_{so}$  = Specific gravity of Sodium hydroxide solution.

#### 4. ILLUSTRATIVE EXAMPLE FOR GEOPOLYMER CONCRETE MIX DESIGN

An example illustrating the mix design for a Geopolymer concrete of M 30 grade is given below:

**4.1 Design stipulations**

- |  |   |          |
|--|---|----------|
| a) Characteristic compressive strength required at age of 36hours at the temperature of 60°C | = | 30MPa    |
| b) Maximum size of aggregate (angular)   | = | 10mm     |
| c) Specific gravity of fly ash   | = | 2.3      |
| d) Specific gravity of coarse aggregate  | = | 2.6      |
| e) Specific gravity of fine aggregate  | = | 3.1      |
| f) Sand conforming   | = | zone III |
| g) Specific gravity of NaOH  | = | 1.47     |

h) Specific gravity of  $\text{Na}_2\text{SiO}_3$  = 1.6

#### 4.2 Solution

*Step 1: Selection of fly ash to the compressive ratio*

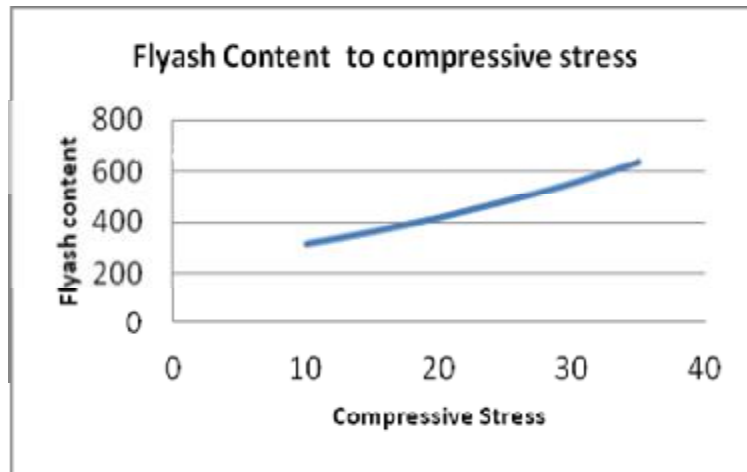


Figure 1. Shows flyash content to the compressive strength

The amount of flyash required for M30 grade  $= 550 \text{ Kg/m}^3$  is Derived from the above Figure 1.

*Step 2: Selection of alkaline liquid ratio*

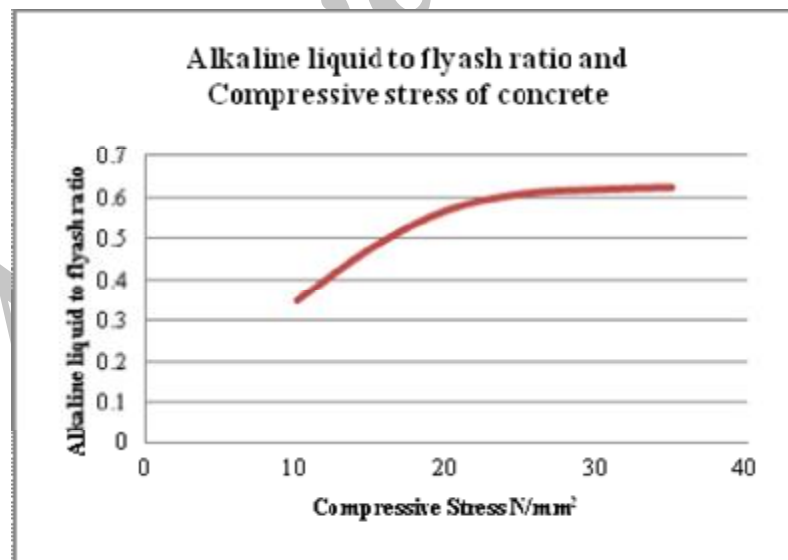


Figure 2. Generalised relation between free alkaline liquid to fly ash ratio and compressive strength of concrete

The ratio between Sodium hydroxide to sodium silicate is 1:2:5 From the table 9

The amount of alkaline liquid required accordance to compressive stress from the Figure 2.



The amount of Alkaline liquid =  $0.61 \times \text{flyash content} = 0.61 \times 550 = 335.5 \text{ Kg/m}^3$

Amount of Sodium silicate Solution =  $239.64 \text{ Kg/m}^3$

Amount of Sodium Hydroxide Solution =  $95.86 \text{ kg/m}^3$

Molarity to be used in the concrete is 16 molar in which 444 grams of NaOH solids dissolved in 556 grams of water. Solids =  $42.56 \text{ kg/m}^3$  Water =  $53.3 \text{ kg/m}^3$

Table 9: Sodium hydroxide to sodium silicate ratio accordance to compressive Strength

Compressive strength	Sodium hydroxide	Sodium silicate
10	1	3
15	1	2.5
20	1	2.5
25	1	2.5
30	1	2.5
35	1	2.5
40	1	2.5
45	1	2.5

*Step 3: Selection of water content*

The maximum water content to add extra is 0.06 Water to flyash ratio

The minimum water content to be added extra is 0.02 water to flyash ratio

According to workability extra water can be added this is due to flyash is arrived from various plant which have different properties in absorption of water in order to match extra water is added.

Amount of water add extra 0.02 to water flyash ratio =  $0.03 \times 550 = 16.5 \text{ kg/m}^3$

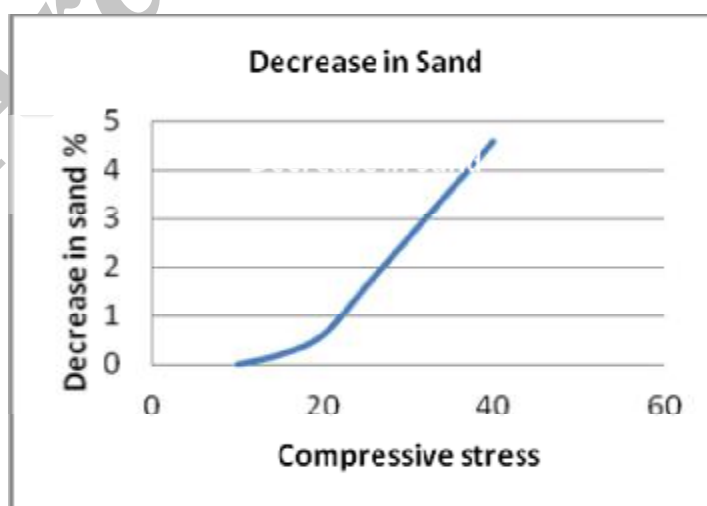


Figure 3. Adjustment of values in sand content percentage

Table 10: Approximate sand contents per cubic metre of concrete for grades up to M35grade

Nominal size of coarse aggregate	Sand as percentage of total aggregate by absolute volume
10mm	40
20mm	35
Change in condition	Sand content in %
For sand conforming to Zone II	0%
For decrease in sand content	-2.5%
Total	-2.5%

From above graph decrease in sand content = 2.5%

Total aggregate by absolute volume =  $(40 - 2.5) = 37.5\%$

#### Estimation of Air Content

Table 11: Approximate air content

Nominal maximum size of aggregate in mm	Entrapped air as percentage of volume of concrete
10	3%
20	2%

#### 4.3 Determination of aggregate content

From table, for the specified maximum size of aggregate of 10 mm, the amount of entrapped air in the wet concrete is 3 %. Taking this into account and applying the following equations:

$$V = \left[ \frac{SO}{S_{so}} + \frac{S}{S_s} + \frac{F}{S_F} + \frac{1}{P} \frac{F_a}{SF_a} \right] \times \frac{1}{1000} \text{ and}$$

$$V = \left[ \frac{SO}{S_{so}} + \frac{S}{S_s} + \frac{F}{S_F} + \frac{1}{1-P} \frac{C_a}{SC_a} \right] \times \frac{1}{1000}$$

Where,

- $V$  = Absolute volume of fresh concrete, which is equal to gross volume minus the volume of entrapped air.
- $S$  = Sodium Silicate Solution (kg) per  $m^3$  of concrete.
- $SO$  = Sodium Hydroxide Solution (kg) per  $m^3$  of concrete
- $F$  = Weight of Flyash (kg) per of  $m^3$  of concrete
- $S_F$  = Specific gravity of Flyash
- $P$  = Ratio of fine aggregate to total aggregate by absolute volume

$F_a, C_a$  = Total masses of fine aggregate and coarse aggregate (kg) per  $m^3$  of concrete respectively

$SF_a, SC_a$  = Specific gravity of saturated surface dry fine aggregate and coarse aggregate respectively.

$S_s$  = Specific gravity of Sodium Silicate solution.

$S_{so}$  = Specific gravity of Sodium hydroxide solution.

On substitution, in the above equation

**Fine aggregate content:**

$$0.97 = \{(95.86/1.47) + (239.64/1.6) + (550.0/2.3) + (1/(0.36))(F_a/2.6)\} \times (1/1000)$$

$$F_a = 600 \text{ kg} / m^3$$

**Coarse aggregate content:**

$$0.97 = \{(95.86/1.47) + (239.64/1.6) + (550.0/2.3) + (1/(1-0.36))(C_a/2.6)\} \times (1/1000)$$

$$C_a = 838.3 \text{ kg} / m^3$$

Table 12: Mix proportion

Sodium silicate	Sodium hydroxide solution	Extra water	Flyash	Fine aggregate	Coarse aggregate
239.64 kg/m <sup>3</sup>	95.86 kg/m <sup>3</sup>	11 kg/m <sup>3</sup>	550 kg/m <sup>3</sup>	600 kg/m <sup>3</sup>	838.3 kg/m <sup>3</sup>
	0.61	0.03	1	1.04	1.56

## 5. CONCLUSION

- With the generic information available on geopolymers, a rigorous trial-and-error method was adopted to develop a process of manufacturing fly ash-based geopolymer concrete following the technology currently used to manufacture Ordinary Portland Cement concrete. After some failures in the beginning, the trial-and-error method yielded successful results with regard to manufacture of low-calcium (ASTM Class F) fly ash-based geopolymer concrete.
- Geopolymer concrete is an excellent alternative solution to the CO<sub>2</sub> producing port land cement concrete.
- Low-calcium fly ash-based geopolymer concrete has excellent compressive strength within a day and is suitable for structural applications [10].
- The price of fly ash-based geopolymer concrete is estimated to be about 10 to 30 percent cheaper than that of Portland cement concrete.
- In this study it is observed that Compressive strength results obtained for M-sand was nearly equal when compared to control concrete.

- Tensile strength of the river sand is high when compared to the M- Sand.
- Due to presence of sodium silicate as a sticky gel form in nature And also flyash is finer element it recommended to have 20% higher than of Mix design values.
- Due to presence of sodium silicate as a gel form it take delay in setting at ambient temperature. In order to overcome it recommended that heat curing is necessary, which also increase the compressive Strength. The temperature to be maintained is 60°C for about 24hours to 48hours.

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