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Effect of Increasing the Molarity Concentration and Curing Time on Strength and Durability of Geopolymer Mortar

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Abstract
This paper presents the effect of increasing the molarity concentrations and curing time for strength and durability of flyash based geopolymer mortars activated using combined sodium silicate and sodium hydroxide solutions. Keeping NaOH concentration as 8M, 10M and curing time as 24hrs, 48hrs totally nine mixes were prepared. Compressive strength, Split tensile strength and Rapid Chloride Penetration tests were conducted on each of the nine mixes. Results of the experimental study showed that there was an increase in compressive strength and split tensile strength with increase in NaOH concentration and curing time. Compressive strength up to 52.28 MPa was obtained with curing at 75°C for 10 molarity geopolymer mortar.

Keywords: *molarity, compressive strength, split tensile strength, concentration, Rapid chloride penetration.*

INTRODUCTION
The production of Portland cement is an energy-intensive process that releases a very large amount of greenhouse gas in to the atmosphere [1]. The efforts seek to replace Portland cement with other forms of cementitious materials such as geopolymers. A geopolymer or alkali-activated cement is an inorganic, alumino-silicate based material [2]. Class F fly ash is designated in ASTM C 618 and originates from anthracite and bituminous coals. It consists mainly of alumina and silica and has a higher LOI than Class C fly ash. Class F fly ash also has a lower calcium content than Class C fly ash [3]. A favourable research outcome established in last decade is low-calcium fly ash based geopolymer cement and concrete [4-6]. For the geopolymers low-calcium ash is desired as a source material rather than high-calcium fly ash as the existence of high amounts of calcium may restrict with the polymerisation process and modify the microstructure [7]. This paper reports the study of strength and durability of fly ash-based geopolymer mortar by keeping the parameter as varying the concentration of 8M, 10 M NaOH and curing period as 24 hours and 48 hours for silicate-to-hydroxide ratio of 1 and 2.5.

EXPERIMENTAL PROGRAMME
Materials characterization
Ordinary Portland cement of grade 53 according to IS 4031-1988 is used to prepare control specimens. Some of the properties of the cement are given here,
- Specific Gravity = 3.15
- Standard Consistency = 30 %
- Initial Setting time = 35 mins.(IS 1489-2001)
- Final Setting Time = 250 mins. (IS 1489-2001)
- Compressive strength = 56.75 N/mm^2 (28 days)

Geopolymer

Geopolymer is a combination of the following compounds,

- Pozzolan (Flyash)
- Activator solution (Silicates of sodium)
- Alkali powder (Hydroxides Of sodium)
- A high –range water reducing Ligno-Sulphonated normal Super Plasticizer
- Distilled water

Fine aggregate

Locally available river sand having specific gravity of 2.65 was used as fine aggregate for geopolymer mortar and cement mortar mixes.

Fly ash

Fly ash is one of the most widely used by-product materials in the construction field. Class-F Fly ash is produced from anthracite coal used for the replacement of cement in this work was obtained from sterlite industry, Tuticorin. An X-Ray Fluorescence (XRF) analysis is used to determine the chemical composition of the flyash and is shown in Table.1. The silicates and hydroxides of water soluble high alkaline Sodium is utilized in this investigation. The alkali silicate (Na_2SiO_3), with a modulus ratio of 2.15 is purchased from a local supplier in bulk. The alkali hydroxide (NaOH) which is also called lye or caustic soda, in flake form with 97%-98% purity is also purchased from a local supplier in bulk. The hydroxide solution is prepared to a concentration of 8M, 10M using NaOH in flakes and Potable water. In order to improve the workability of stiff and fresh mortar, a high-range water-reducing Ligno-Sulphonated normal Super Plasticizer are added.

Figure 1: *Composition of Geopolymer.*

Table 1: *Chemical Composition of Flyash*

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	TiO_2	SO_3	Na_2O
Mass (%)	65.43	20.67	6.18	1.26	0.82	-	-	Trace	-

Test Variables

Alkaline liquid to fly ash ratio (by weight) was taken as 0.4. While the ratio of sodium silicate solution to sodium hydroxide solution (by weight) in alkaline liquid was taken as 1:1 and 1:2.5.

Preparation of Geopolymer Mortar

Preparation of solution

Separate solutions of NaOH and Na_2SiO_3 of required concentrations were prepared and mixed together 24 hours prior to casting

Mixing

Fine aggregate and fly ash was first mixed in a Hobart mixer for five minutes followed by the activator solution and further mixed for another 5 minutes. The geopolymer mortar mix was then transferred into 70.6 mm cube moulds and vibrated on a vibrating table for 2 minutes. Specimens were cured along with the moulds in a heat curing chamber for a period of 24 hours and 48 hours at 75 ^{o}C and allowed to cool inside the chamber before being removed to room temperature until tested.

Curing

Specimens were cured along with the moulds in a heat curing chamber (fig.2) for a period of 24 hours and 48 hours at 75 ^{o}C and allowed to cool inside the chamber before being removed to room temperature until tested

Figure 2: *Heat curing Chamber.*

Test Specimens

Figure 3: *Mortar mould.*

Cube specimens of size 70.6 mm by 70.6 mm by 70.6 mm (fig.3.) for measuring Compressive strength, Cylinder specimens of 60 mm diameter by 120 mm height for Split tensile strength and disc specimen of size 95 mm nominal diameter and 50mm thick were cast in our study.

Testing

Compressive strength

The Cubes were tested for cube compression testing at the age of 28 days by Compression testing machine (fig.4). For each mix three cube specimens were tested for cube compression testing and the average value have been recorded and shown in the tables 2 and 3.

Figure 4: *Compressive test in UTM.*

Table 2*: Compressive and split tensile strength curing at 24 hours and 48 hours for $Na_2sio_3/NaOH$ =2.5*

Specimen	**compressive strength (Mpa)**		**Split tensile strength (Mpa)**	
	24 hrs	**48hrs**	**24 hrs**	**48 hrs**
Cement mortar	35.32		2.65	
8M Geopolymer mortar	35.6	38.82	2.75	2.89
10M Geopolymer mortars	43.50	46.90	3.30	3.53

Table 3: *Compressive and split tensile strength curing at 24 and 48 hours for $Na_2sio_3/NaOH$ =1*

Specimen	**compressive strength (Mpa)**		**Split tensile strength (Mpa)**	
	24 hrs	**48 hrs**	**24 hrs**	**48 hrs**
Cement mortar	35.32		2.65	
8M Geopolymer mortar	41.50	45.80	2.98	3.21
10M Geopolymer mortars	48.90	52.28	3.56	3.89

Split tensile strength test

Split tensile strength was used to determine the tensile strength of mortar. Cylindrical specimens of size 60 mm diameter and 120 mm height (fig. 5) were used in this test. Tensile strength test was carried out in a 2000 KN capacity of the compression testing machine (fig. 6) in which the specimens are placed in such a way that its axis was horizontal to the platens of the testing machine. The load was applied uniformly at a constant rate until failure by splitting along the vertical diameter took place. The failure load was recorded and the splitting tensile strength was computed.

Figure 5: *Cylinder Specimens.*

Figure 6: *Split Tensile Test in CTM.*

Rapid Chloride Penetration Test (ASTM: C1202-10)

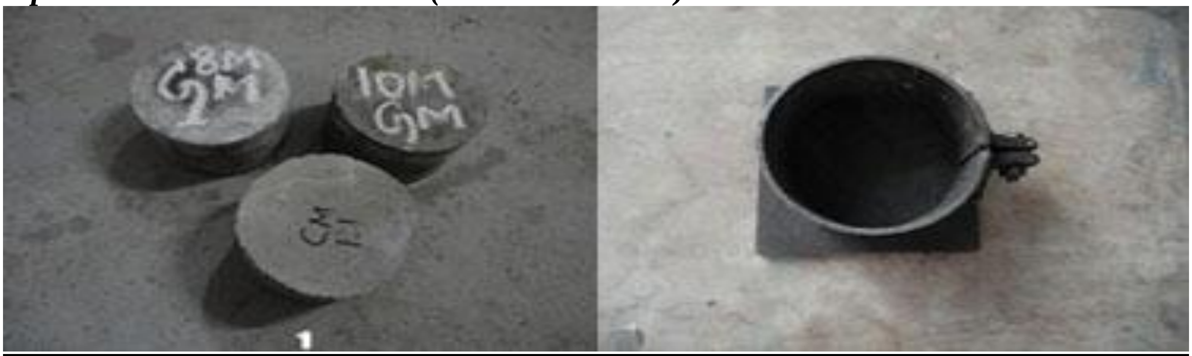

Figure 7: *RCPT Test on mortar disc specimen.*

Disc specimens of size 95 mm nominal diameter and 50mm thick (Fig. 7) were cast for cement and geopolymer mortar. The test setup is called Rapid Chloride penetration test (RCPT). Disc specimen is assembled between the cathode compartment which is filled with 3% Nacl solution and anode compartment is filled with 0.3 normality NaOH solutions. Mortar specimens were subject to RCPT by impressing a 60 V between cathode and anode (fig. 8) as per the procedure given in ASTM C 1202.

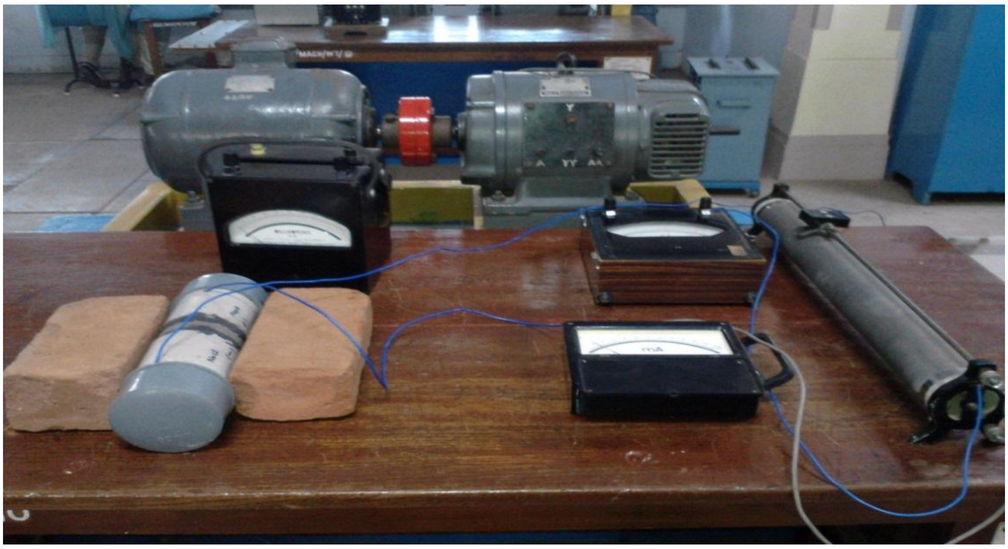

Figure 8: *Experimental setup of Rapid Chloride Penetration test.*

From the current values, the chloride permeability is calculated in terms of coulombs at the end of 6 hours by using the following equation (1)

$Q=900((I_0+I_{360}) + 2(I_{30}+I_{60}+..+I_{330}))$ (1)

Where Q=charge passed (coulombs)

I_0=current (amperes) immediately after voltage is applied, I_t=current (amperes) attime't' after voltage is applied

RESULTS AND DISCUSSIONS

Concentration of Sodium Hydroxide Solution

Mortar mixes were prepared to study the effects of concentration of sodium hydroxide solution on the Compressive strength and Split tensile strength of Geopolymer mortar. It was observed that the strength increased when the concentration of sodium hydroxide increases from 8M to 10M. It was observed that alkaline concentration is proportionate to the Compressive and Split tensile strength of Geopolymer mortar (Fig. 9, 10, 11, 12).

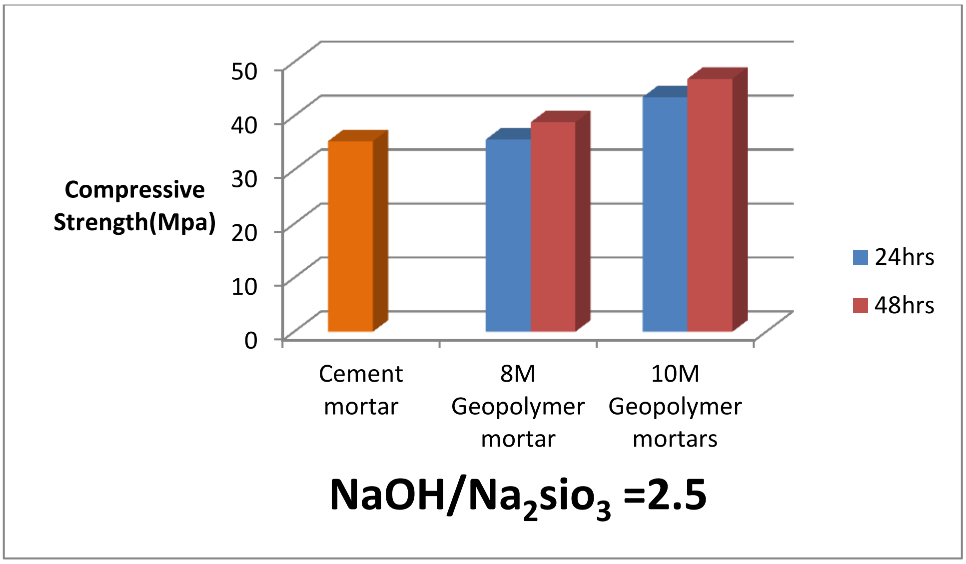

Figure 9: *Variation of silicates-to-hydroxide ratio=2.5 on the compressive strength of geopolymer mortar for 8M and 10M Solution and curing periods.*

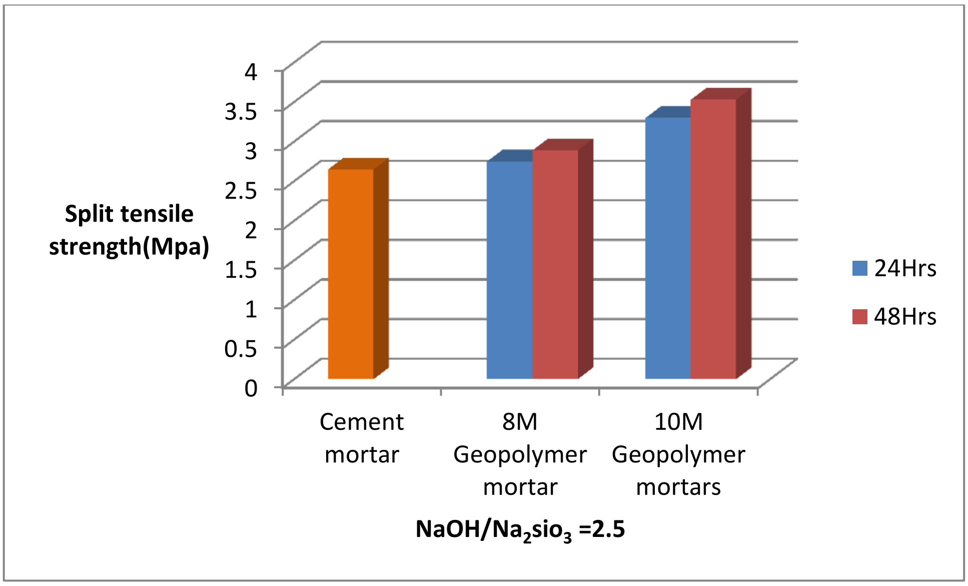

Figure 10: *Variation of silicates-to-hydroxide ratio=2.5 on the compressive strength of geopolymer mortar for 8M and 10M Solution and curing periods.*

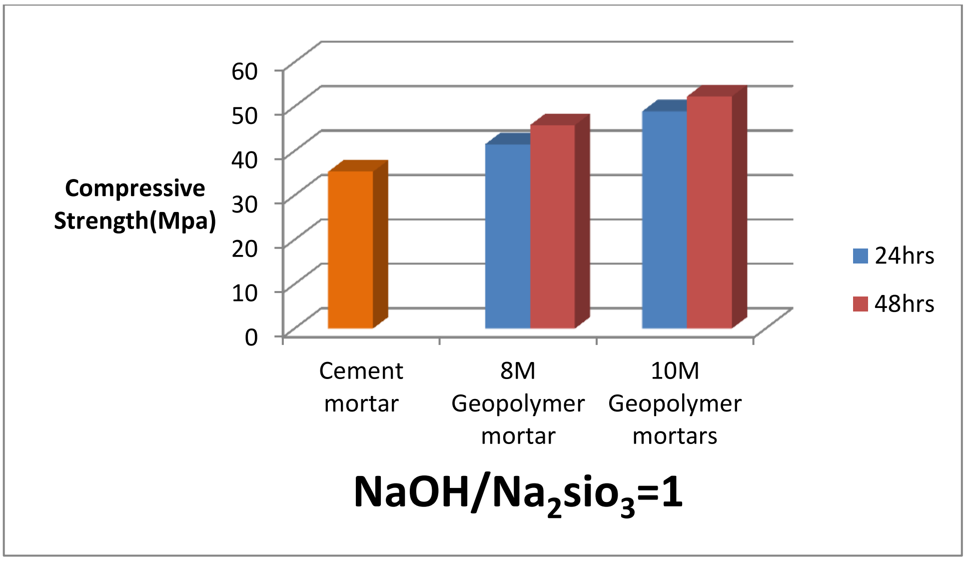

Figure 11: *Variation of silicates-to-hydroxide ratio=1 on the compressive strength of geopolymer mortar for 8M and 10M Solution and curing periods.*

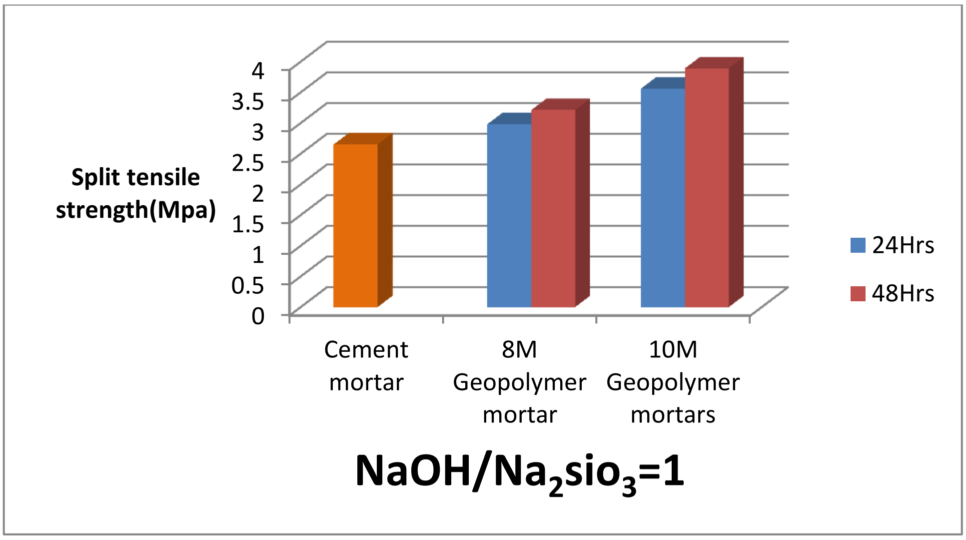

Figure 12: *Variation of silicates-to-hydroxide ratio=1 on the compressive strength of geopolymer mortar for 8M and 10M Solution and curing periods.*

Curing time

Tables 4, 5 and Fig. 9, 10, 11, 12 reveals that prolonged curing time improve the polymerisation process resulting in higher compressive strength and Split tensile strength. The increasing temperature favours the dissolution of reactive species, that of the fly ash, in the same degree. Increase in curing period beyond 48 hrs. Decrease the strength, as it breaks the granular structure of geopolymer mixture results in dehydration and excessive shrinkage due to contraction of gel, without transforming to a more semi-crystalline form.

Table 4: *Charge Passed through RCPT as per ASTM 1202*

Charge passed(coulombs)	**Chloride ion Penetrability**
>4000	High
2000-4000	Moderate
1000-2000	Low
100-1000	Very Low
<100	Negligible

Table 5: *Ion Transfer in Control Specimen, 8 molarity geopolymer specimen, 10 molarity geopolymer specimen*

Time (second)	Cement mortar	8M	10M
0	0.006	0.005	0.004
1800	0.010	0.009	0.008
3600	0.014	0.013	0.012
5400	0.018	0.017	0.016
7200	0.021	0.021	0.016
9000	0.026	0.025	0.020
10800	0.030	0.029	0.024
12600	0.034	0.033	0.028
14400	0.038	0.037	0.032
16200	0.041	0.041	0.036
18000	0.044	0.045	0.040
19800	0.048	0.049	0.044
21600	0.051	0.053	0.048

Silicate and hydroxide ratio

The ratio of sodium silicate to sodium hydroxide plays an important role in the compressive strength development (Fig. 11). By the increase in concentration of alkali or decrease in added silicate, increase in compressive strength is expected (9).

This is because excess sodium silicate hinders water evaporation and structure formation (10). Thus both the compressive and tensile strength is decreased as the silicate –to-hydroxide ratio increased (fig. 9 and 10).

Rapid Chloride Penetration Test (ASTM: C1202-10)

The RCPT values from cast specimens of geopolymer at 28 days fall in the range of 600 to 626 coulombs – indicative of very low penetrability. Conventional mortar on the other hand has values of 634 coulombs which is slightly higher than geopolymer mortars (table 6). The more permeable is the mortar, the more chloride ions will migrate through the specimen, and a higher current will be measured during 6 hours. The total charge or Coulombs passed across the specimen is determined by the area under the curve of current versus time (fig. 13).

Table 6: *Results for Rapid chloride ion Penetration test*

Specimen	Charge Passed in Coulombs	As per ASTM C 1202 Chloride penetrating rate
Cement mortar	634.00	Very low
8 M Geopolymer Mortar	626.40	Very low
10 M Geopolymer Mortar	604.80	Very low

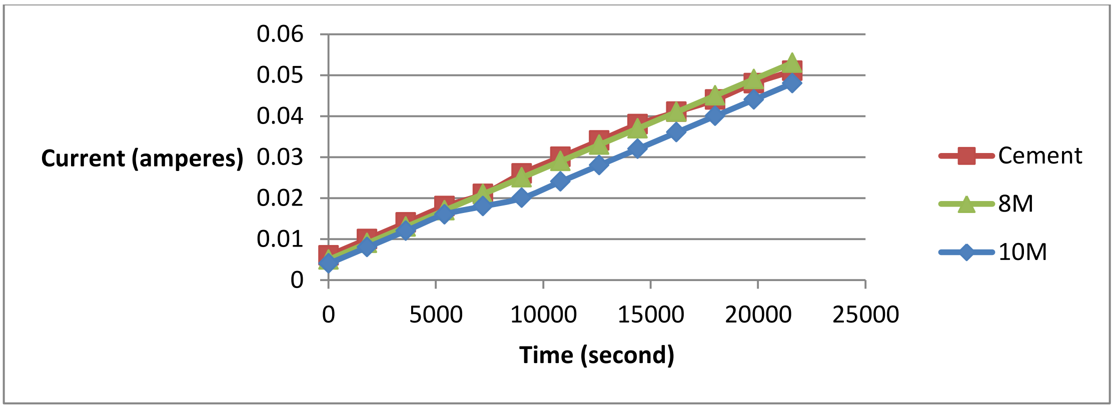

Figure 13: *Current Vs Time curve for 8M and 10 M geopolmer mortar.*

Because of its finer pore structure and chemical stability, geopolymer mortar is substantially more resistant to chloride diffusion. Thus, it reduces the penetration of chlorides and protects embedded steel from corrosion. As per ASTM C1202 (8), the value obtained for cement mortar and Geopolymer mortar are graded under the category very low. As such it is indicating lesser permeability for both the mortars.

CONCLUSIONS

Following conclusions were drawn from the study on geopolymer mortar:

- Geopolymer mortar is eco-friendly and has the potential to reduce the greenhouse gas emission by replacing cement in many applications such as precast units.
- Compressive strength increases with increase in concentration of NaOH from 8M to 10M and curing time from 24 hrs to 48 hrs.
- Higher concentration of sodium hydroxide solution results in a higher compressive and split tensile strength of geopolymer mortar.
- As the ratio of sodium silicate to sodium hydroxide by mass increases, both the compressive strength and split tensile strength of geopolymer mortar decreases.
- Split tensile strength increases with increase in concentration of NaOH from 8M to 10M. Increase in split tensile strength was also observed with increase in curing time
- For silicate-to-hydroxide ratio equal to 2.5,the percentage increase in compressive strength compared to control specimen for 24 hours curing 8M, 10M Geopolymer mortars are 0.78%,23.15% and for 48 hours curing 8M, 10M Geopolymer mortars are 9.9%,32.78% respectively.
- For silicate-to-hydroxide ratio equal to 2.5,the percentage increase in Split tensile strength compared to control specimen for 24 hours curing 8M, 10M Geopolymer mortars are 3.9%,24.52% and for 48 hours curing 8M, 10M Geopolymer mortars are 9.05%,32.78% respectively.
- For silicate-to-hydroxide ratio equal to 1,the percentage increase in compressive strength compared to control specimen for 24 hours curing 8M, 10M Geopolymer mortars are 17.49%,38.84.% and for 48 hours curing 8M, 10M Geopolymer mortars are 29.67%,48.01 respectively.
- For silicate-to-hydroxide ratio equal to 1,the percentage increase in Split tensile strength compared to control specimen for 24 hours curing 8M, 10M Geopolymer mortars are 12.45%,34.33% and for 48 hours curing 8M, 10M Geopolymer mortars are 21.13%,46.79% respectively
- The charge passed for 8M and 10M Geopolymer mortar has showed slightly lesser value than cement mortar specimen
- As per ASTM C1202, the value obtained for Geopolymer and Cement mortar is graded under the category LOW.As such it is indicating lesser chloride penetrate to the entire specimen.

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