

EXPERIMENTAL STUDY OF STRENGTH AND MICROSTRUCTURAL PROPERTIES OF GGBS AND METAKAOLIN BASED GEOPOLYMER CONCRETE

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ABSTRACT

Geopolymer concrete is an emerging material in the field of sustainable construction. This material is alkali-activated aluminosilicate having little environmental impact as industrial by-products are utilized. Geopolymer concrete has previously displayed better performance properties. However, its extensive use in the construction industry seems to be hindered because of multiple factors and particularly due to lack of long term behavioural data and practical

issues like poor workability. Working on resolving this issue, the proposed research aims to study the microstructure of GGBS and metakaolin based geopolymer concrete by varying concentrations of alkaline NaOH solution (8M, 12M and 16M). Keen attention is paid to understand correlation of binding product formation with some of the mechanical properties of the concrete also the effect of use of superplasticizer on geopolymer concrete is studied. The outcomes clearly indicate that the strength increases with the increase in concentration of NaOH solution. This is attributed to significant amount of geopolymerisation product formed due to easy dissolution of Al and Si ions in the mix. The study confirms there is substantial amount of increase in the geopolymerisation product with increase in curing period. Likewise,

use of Naphthalene based superplasticizer has decreased the use of water. Yet, the slump values show no significant change.

KEYWORDS: Geopolymer Concrete, Microstructure, SEM Analysis, Water absorption, workability, Compressive strength, Superplasticizer, Water reducer, GGBS, Metakaolin.

1. INTRODUCTION

Cement, is an incredible manmade material and it is the main component of concrete which is the second most widely used material after water on the planet. A boon that shapes modern manmade structures is now creating concerns of catastrophic climate change due to its excessive CO₂ emissions. CO₂ is a major greenhouse gas which now is increasing at an alarming rate, and construction industry has notoriously wreaked havoc.

There are studies carried out in the industry to understand behaviour and performance of alternate binding materials. Geo-Polymer Concrete (GPC) has significantly displayed better performance than conventional cement concrete numerous times and also possess the ability to add significant value to industrial by-products.^[1] Despite of all the beneficial attributes of geopolymer concrete, great bulk of the industry is inherently conservative, ignorant of the alternatives to cement and reluctant to adopt change due to multiple reasons such as adherence to compositionally based building codes and standards and lack of quantitative data on durability due to relatively recent popularization of research in this field.^[2]

The majority of the studies on geopolymer concrete have focused on compressive strengths and flexural strengths of the systems. Very limited studies have intensively observed the microstructural details due to complexities in analysis of materials formed from different aluminosilicate sources like fly ash or GGBS etc.^[3-5] Various studies have found the different significant parameters which effect the performance of concrete like the ratio of binder to alkali activators, ratio of silica to alumina content in binder materials, curing temperatures, curing time and concentrations of alkali activators etc. The optimum values of these parameters is found but, the long term study of durability still remains a concern.^[6-7]

Conventional concrete consists of matrix, aggregate and interfacial transition zones (ITZ). To improve the concrete performance, the following three aspects should be considered

1. Strengthening the matrix
2. Lowering porosity

3. Toughening the ITZ

Permeability plays a fundamental role in deterioration of concrete due to chemical attacks and durability in general.^[8] Hence, it becomes vital to have a system which has fairly homogeneous microstructure with lower porosity as durability strongly depends upon pore system characteristics.^[9] And the compressive strength of the binder is connected to its composition and degree of reaction.^[4]

The current research aims at understanding the amount of product formed through the microstructure images in GGBS and metakaolin based geopolymer concrete by varying NaOH concentrations to understand some of the strength properties of geopolymer concrete. And also study the effect of use of superplasticizer.

2. EXPERIMENTAL DESIGN

Ground Granulated Blast Furnace Slag (GGBS) conforming to Indian Standard IS 16714-2018 was used in the present experimental investigation. The GGBS, having specific gravity 2.91 and fineness as $381\text{m}^2/\text{kg}$ was purchased under the brand name JSW. The metakaolin was collected from, Gautum Dyes and Chemicals, Chennai, in Tamil Nadu state, India. The metakaolin used was off-white colour in appearance, containing 98% of Meta-Kaolin content. With SiO_2 and Al_2O_3 being 51% and 43% by mass. River sand of downsize 4.75 mm and specific gravity of 2.6 (conforming to Indian Standard IS 383- 1970) was used in concrete. Locally available aggregate, of downsize 20 mm and specific gravity of 2.7. (Conforming to Indian Standard IS 383- 1970) was used as coarse aggregate. The White deliquescent Sodium hydroxide pellets (CAS No: 1310-73-2) procured from Nitin Surgical and Chemicals, Hubballi, Karnataka in India were dissolved in required quantity of water to make 8M, 12M and 16M solution. The pellets (Table 1) were added gradually into water and the solution was constantly stirred with glass rod. Sodium Silicate solution (containing 16.51% Na_2O and 34.80% SiO_2 by mass) was procured from Megha Chemicals, Hubballi, Karnataka in India was used in the concrete mixes. Naphthalene based superplasticizer was used in this study. It was procured under the brand name MasterPel 777 BASF (conforming to IS 2645-2003, IS 9103-1999 and ASTM C494: Types A & D) from Sri Sai Engineering Services in Hubballi, Karnataka in India.

Table 1: Concentration of NaOH.

Concentration of solution	NaOH Pellets
8M	320 g/L
12M	480 g/L
16M	640 g/L

The design mix was prepared by keeping GGBS to Metakaolin ratio as 70:30 and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ solution ratio as 2.5 as constant and varying concentrations of NaOH solution (8M, 12M and 16M). And cubes of size 150mm x 150mm x 150mm for compression testing and determining water absorption were casted both with and without use of superplasticizer (2% of binder). The cubes were tested after 1, 3 and 7 days of ambient curing. SEM analysis of the cubes for microstructure study was conducted. The design mix (Table 2) was prepared by keeping the following constant.

GGBS: Metakaolin ratio was fixed as 70: 30

Water to binder ratio was fixed as 0.23

Alkaline solution to binder ratio was fixed as 0.35

Sodium Silicate to Sodium Hydroxide ratio was fixed as 2.5

Concentrations of NaOH was varied as 8M, 12M, 16 M.

Table 2: Mix Design.

S. No.	Product	Quantity/ m ³	Quantity in kg for 15 cubes
1.	GGBS	285.6	14.56
2.	Metakaolin	122.4	6.25
3.	Fine Aggregates	554	28.25
4.	Coarse Aggregates	1294	65.94
5.		NaOH Solids	
	8M	9.91	0.17
	12M	13.25	0.23
	16M	15.94	0.27
6.	Na₂SiO₃ Solids	52.39	2.67
7.		Total Water	
	8M	80.68	1.37
	12M	77.34	1.31
	16M	74.65	1.26

The coarse aggregate and fine aggregate in saturated surface dry conditions were initially mixed on flat tray for 3-4 minutes. GGBS and Metakaolin was then added to the aggregates and mixed thoroughly. Appropriate dosage of alkali solutions and water was added to increase the workability of concrete and further the mixing was continued for 50-60 seconds to produce

well blended Geopolymer concrete. The fresh concrete is then added into pre-oiled moulds. And slump test was conducted. The concrete was filled with 3 layers following standard compaction procedure, giving 25 blows for each layer using tamping rod. Finally, the concrete moulds were vibrated on vibrating table for 10-15 seconds edges were trimmed and kept on flat ground for setting. After 24 hours the concrete specimens were demoulded and subjected to ambient curing. The demoulded concrete cubes were kept to cure at standard temperature for specified period of days i.e. 1, 3 and 7 days. After the specified curing period the cubes are tested for compression and water absorption.

3. RESULTS

3.1 Compressive strength and water absorption test for GPC without superplasticizer

The cubes casted were of size 150 x 150 x 150 mm³, with surface area of 0.0225 m² without the use of super plasticizer. The mixes with 8M, 12M and 16M concentrations of NaOH are named as M1, M2 and M3 respectively.

Table 3: Compressive strength at day 1.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M1	503.1	514.8	509.7	509.2	22.63
M2	654.1	653.2	658.9	655.4	29.12
M3	736.4	743.5	744.6	741.5	32.95

Table 4: Compressive strength at day 3.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M1	643.9	632.1	611.8	626.26	27.96
M2	973.6	981.4	988.8	980.6	43.58
M3	954.3	963.2	957.4	958.3	42.59

Table 5: Compressive strength at day 7.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M1	821.4	825.3	832.1	826.6	36.72
M2	1001.7	993.1	982.5	992.4	44.11
M3	1071.3	1105.8	1003.9	1060.3	47.12

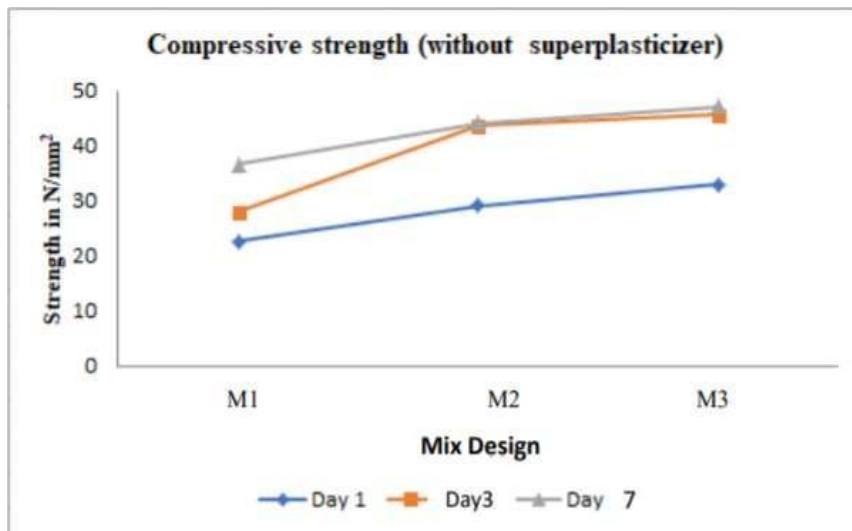


Figure 1: Compressive strength for mixes M1, M2 and M3.

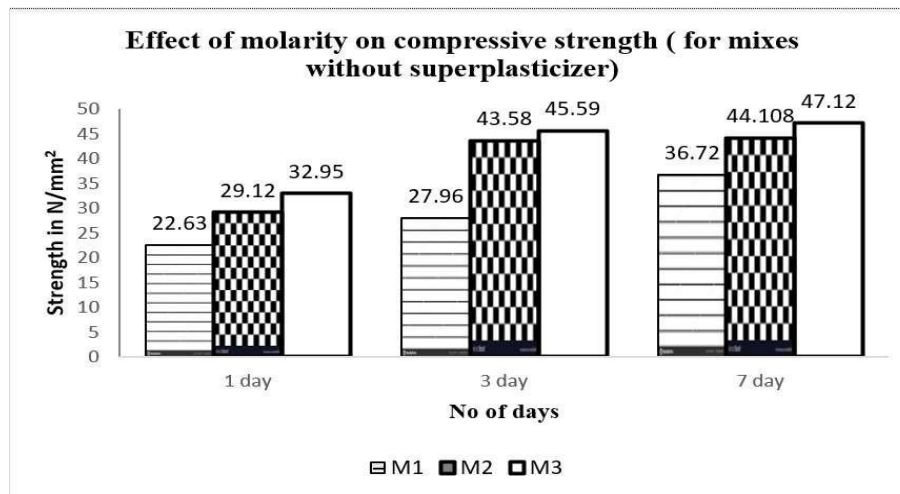


Figure 2: Effect of Molarity of NaOH on compressive strength.

Table 6: Water absorption for M1, M2 and M3.

No. of days	Wet weight Ww(kg)	Dry weight Wd(kg)	Absorbed water Ww-Wd(kg)	Percentage absorption ((Ww-Wd)/Wd)*100 %
M1				
1 day	8.340	7.900	0.440	5.27
3 day	8.300	7.800	0.420	5.06
7 day	8.370	7.940	0.430	5.13
M2				
1 day	8.125	7.775	0.35	4.31
3 day	8.210	7.890	0.32	3.90
7 day	8.175	7.875	0.30	3.60
M3				
1 day	8.205	7.885	0.320	3.90
3 day	8.400	8.120	0.280	3.33
7 day	8.315	8.045	0.270	3.24

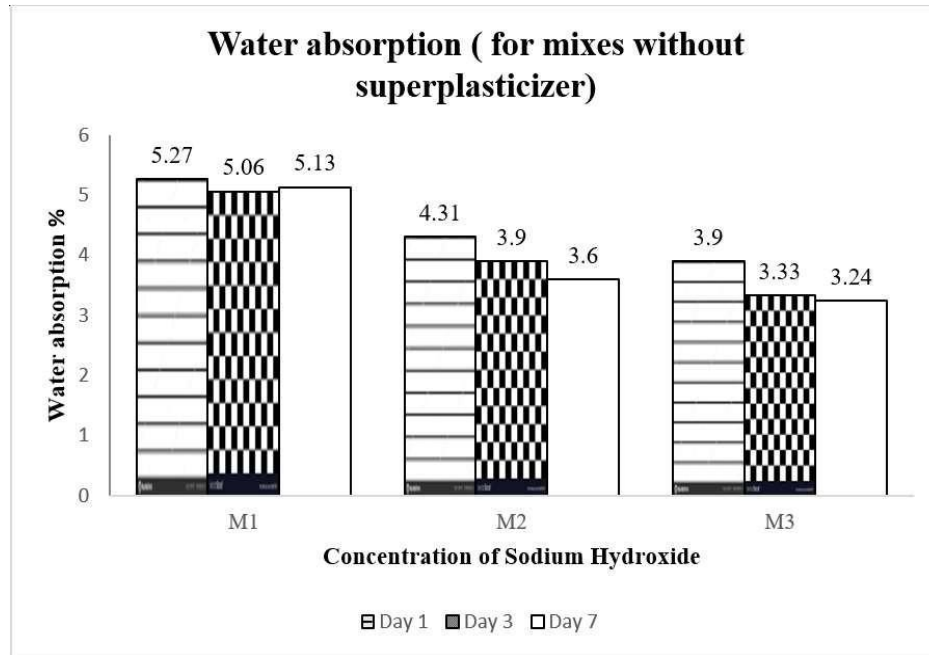


Figure 3: Water absorption for mixes M1, M2 and M3.

At 1 day the compression strength (Table 3) of the concrete will increase with increase in the concentration of the NaOH solution. The increase in the graph shows the improving compressive strength. Further at 3 days the compressive strength (Table 4) again increases showing the action of additional binding product formed over the curing period. At 7 days (Table 5) the concrete have achieved maximum strength, showing maximum strength gain at 16M NaOH solution (as shown in Figure 1 and 2).

Water absorption for day 1 is decreased for about 26 % from 8M to 16 M and 34% for day 3 and 36% for day 7 (as shown in Table 6 and Figure 3).

3.2 Slump, compressive strength and water absorption test for GPC with superplasticizer

The cubes casted were of size $150 \times 150 \times 150 \text{ mm}^3$, with surface area of 0.0225 m^2 with the use of superplasticizer (2% of binder). The mixes with 8M, 12M and 16M concentrations of NaOH are named as M4, M5 and M6 respectively. The slump test results for mixes M1, M2 and M3 are shown in Table 7.

Table 7: Slump test results.

Mix	Slump Values (mm)
M4	26
M5	24
M6	23

Table 8: Compressive strength at day 1.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M4	558.1	580.5	582.2	573.6	25.49
M5	668.2	693.1	699.4	686.9	30.52
M6	772.5	782.9	794.5	783.3	34.81

Table 9: Compressive strength at day 3.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M4	648.6	604.9	579.9	610.9	27.21
M5	997.5	935.0	1081.0	1004.5	44.60
M6	963.1	987.2	953.4	967.9	45.01

Table 10: Compressive strength at day 7.

Mix	Load at failure (W)kN				Compressive strength N/mm ²
	S1	S2	S3	Average	
M4	887.9	875.6	841.0	868.16	38.58
M5	981.2	1086.0	1144.0	1053.73	47.57
M6	955.5	919.9	1502.4	1125.93	50.00

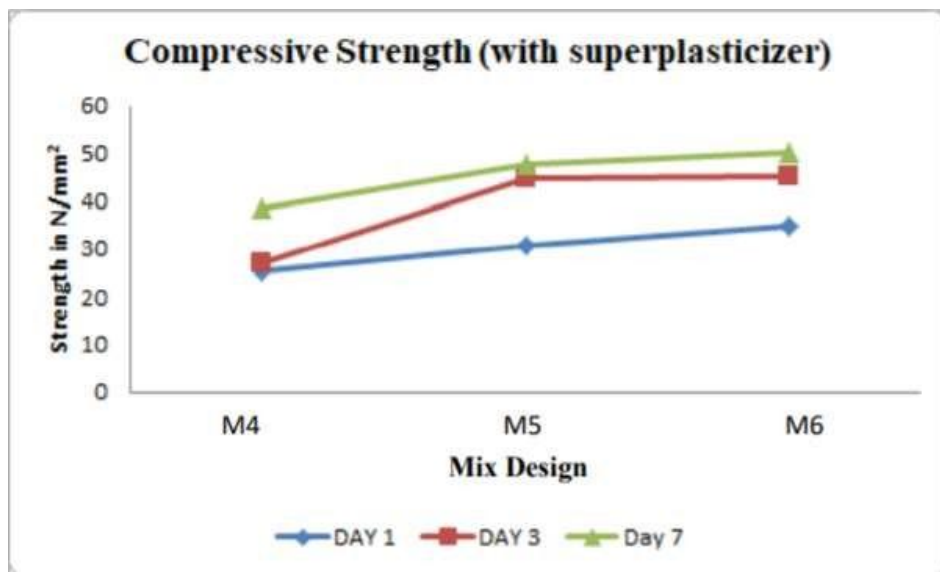


Figure 4: Compressive strength for mixes M4, M5 and M6.

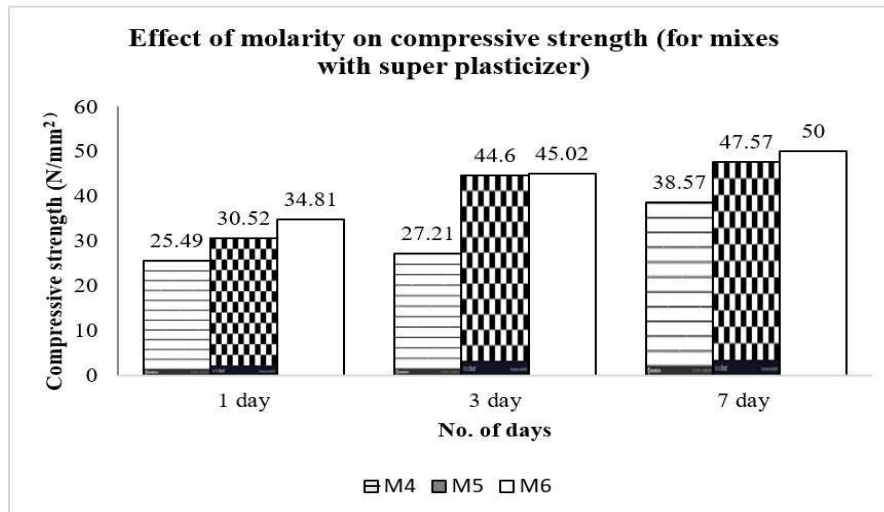


Figure 5: Effect of Molarity of NaOH on compressive strength.

Table 11: Water absorption for M4, M5 and M6.

No. of days	Wet weight Ww(kg)	Dry weight Wd(kg)	Absorbed water Ww-Wd(kg)	Percentage absorption $((Ww-Wd)/Wd)*100\%$
M4				
1 day	8.250	7.830	0.420	5.36
3 day	8.350	7.950	0.400	5.09
7 day	8.235	7.855	0.450	4.83
M5				
1 day	8.300	7.950	0.350	4.20
3 day	8.225	7.935	0.290	3.50
7 day	8.315	8.040	0.275	3.30
M6				
1 day	8.325	8.055	0.270	3.24
3 day	8.400	8.135	0.265	3.15
7 day	8.415	8.140	0.275	3.26

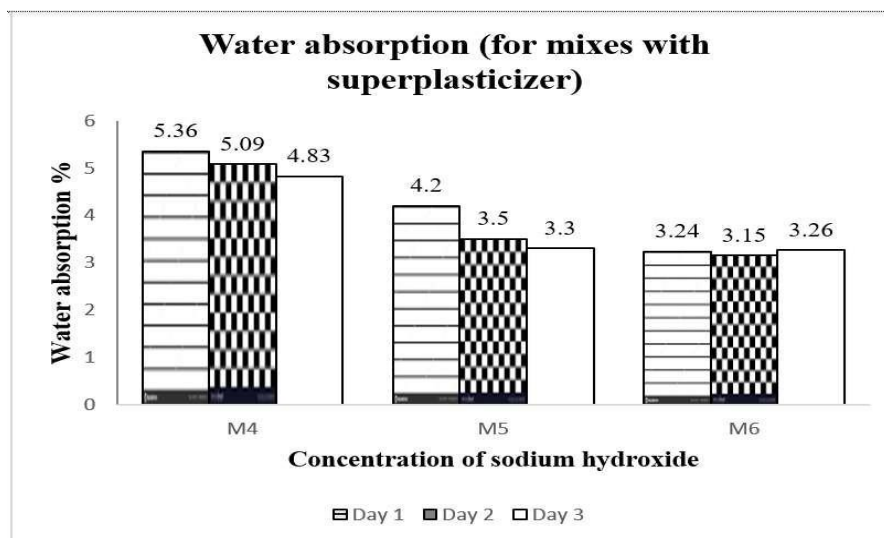


Figure 6: Water absorption for mixes M4, M5 and M6.

At 1 day the compression strength (Table 8) of the concrete will increase with increase in the concentration of the NaOH solution. The linear increase in the graph shows the improving compressive strength. Further at 3 days the compressive strength (Table 9) again increases showing the action of additional binding product formed over the curing period. At 7 days (Table 10) the concrete have achieved maximum strength, showing maximum strength gain at 16M NaOH solution (as shown in Figure 4 and 5).

Water absorption for day 1 is decreased for about 39 % from 8M to 16 M and 38% for day 3 and 32% for day 7 (as shown in Table 11 and Figure 6).

4. DISCUSSION

4.1 Effect of concentration of NaOH solution on compressive strength and absorption of Geopolymer concrete

The compressive strength, of the concrete mixtures clearly shows that with increasing concentration of NaOH solution the strength of the mixes also increases. There is around 22% increase in strength from M1 to M2 concrete and also around 11% increase in strength from M2 to M3 concrete (for day 1). This shows that the highly alkaline solutions react well with aluminosilicate sources. The greater slag percentage in combination with siliceous metakaolin reacts adequately to contribute to the strength gain of the concrete.^[4] However, the increasing concentration makes the mix more viscous leading to poor compaction and also void formation resulting in hindered strength gain and lower workability. The M2 concrete mix apparently turns out to be optimum for use as it possesses desired workability and also attains desired compressive strength. The water absorption decreases with the substantial increase in concentration of NaOH. This decrease in the water absorption confirms the density of the concrete matrix being increased, due to substantial increase in geopolymerisation product. Also the reason for increase in the compressive strength of the concrete can be explained by it. The trend of increasing strength and decreasing water absorption in mixes containing superplasticizer further summarizes the coherence of experimental study.

4.2 Effect of curing period on compressive strength and water absorption of Geopolymer concrete

As the curing days increase the strength gain is evident. There is visible strength gain between 1 and 3 days showing that most early strength is achieved within 3 days of casting. This shows that the aluminosilicates and alkali activators readily react with each other to form Si-O-Al-O

bond framework which contributes to the binding property of the material. There is no significant strength gain in the concrete after 3 days of ambient curing which emphasizes that most of the geopolymerisation process is completed during the initial days of ambient curing.

4.3 Effect of superplasticizer on workability with increasing concentrations of Sodium hydroxide

Chindraprasirt et al. (2007) investigated the workability and strength of fly ash based geopolymer concrete. The experimental results showed that the slump value of GPC is in the range of 115-135 mm and depends on the ratio of Sodium silicate and Sodium hydroxide and the concentrations of NaOH. If the levels of alkali soluble calcium are low, slump loss of GPC produced in case of low calcium fly ash or slag is observed to be equal or less than that of OPC concrete. In this research with increasing concentrations of Sodium hydroxide, it is evident that strength increases while workability decreases. Rangan and Hardjito (2005) observed that the workability can be increased with the use of naphthalene based superplasticizer. The recommended quantity of liquid naphthalene SP is around 2-4% of the mass of the binder. In the research study carried out by Mithanthaya I.R. et al.(2019) the use of naphthalene-based super plasticizer improves the fresh and hardened behaviour of GPC. Various researchers have suggested that the cohesiveness and slump ability of GPC increases with increase of $\text{SiO}_2/\text{Na}_2\text{O}$ ratio in the sodium silicate solution. Accordingly, in this study, the mix that is obtained at high concentrations is a very dry mix thus reducing the workability. Addition of Naphthalene based Superplasticizer (BASF) to the mix reduced the water content upto 2 litres to obtain a workable mix in comparison with the concrete without superplasticizer.

4.4 Microstructure study (SEM analysis)

The images (Figures 7-15) illustrate the SEM determined microstructure characteristics of synthesized geopolymer concrete having NaOH concentrations of 8M, 12M and 16M each at 1, 3 and 7 days.

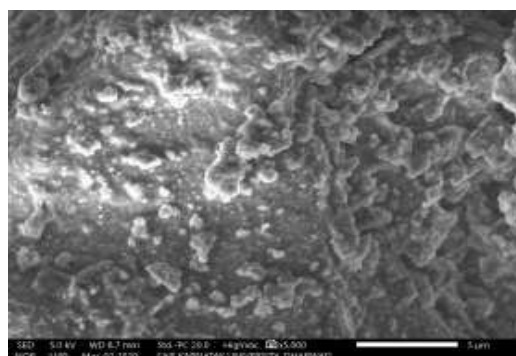


Figure 7: SEM of mix M1 at day 1.

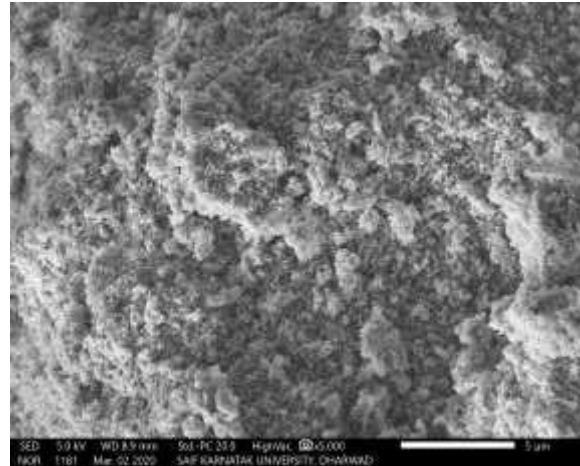


Figure 8: SEM of mix M1 at day 3

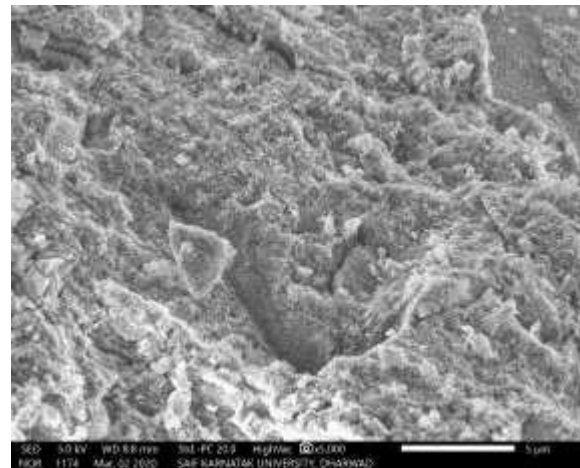


Figure 9. SEM of mix M1 at day 7.

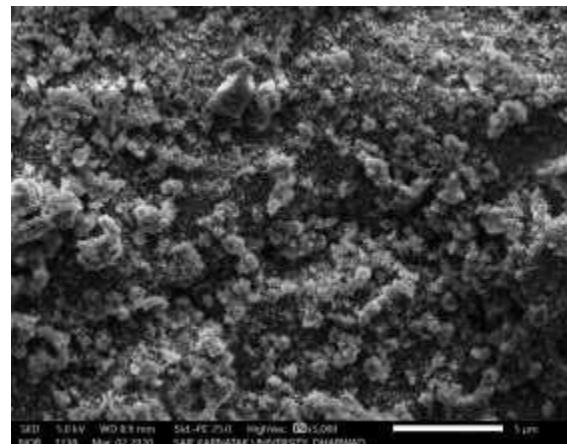


Figure 10: SEM of mix M2 at day 1.

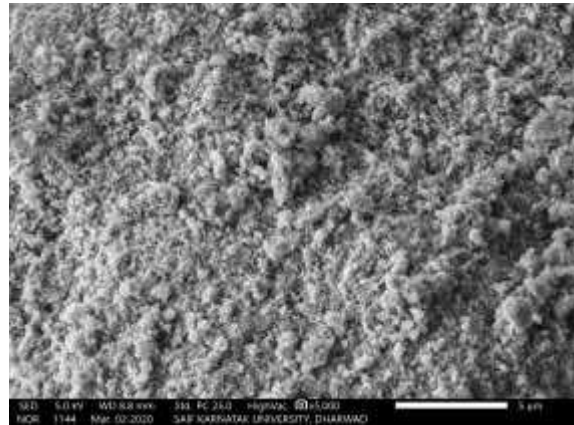


Figure 11: SEM of mix M2 at day 3.

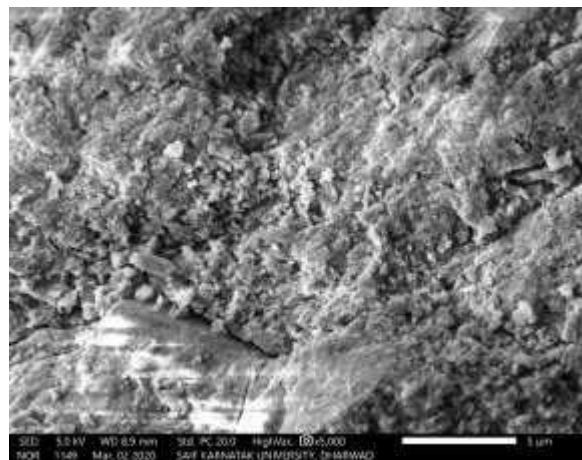


Figure 12: SEM of mix M2 at day 7.

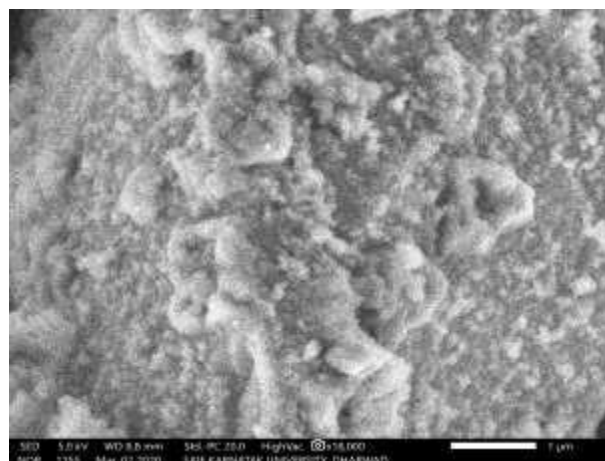


Figure 13: SEM of mix M3 at day 1.

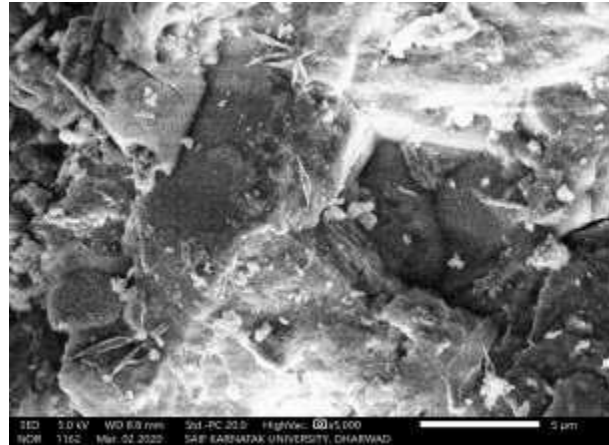


Figure 14: SEM of mix M3 at day 3

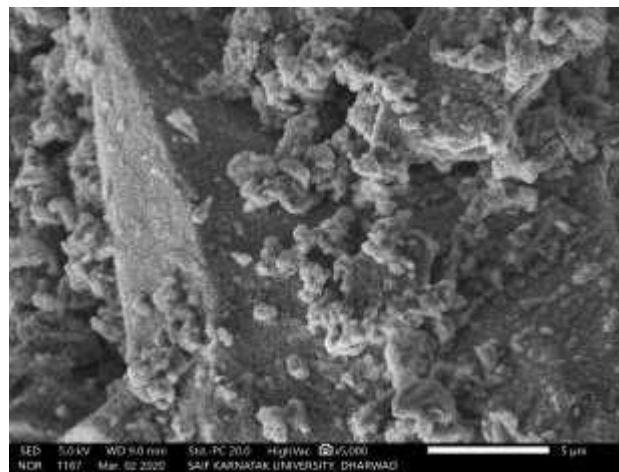


Figure 15: SEM of mix M3 at day 7.

The SEM images of Mixes M1, M2 and M3 all at day 7 demonstrate that with increasing concentration of NaOH, the dissolution of Al and Si ions happens readily to form SiO_4 and AlO_4 tetrahedral units and cross linked polymer framework that increases the amount of dense amorphous geopolymerisation product which in turn enhances homogeneity and results in increased strength and lower water absorption. Hence, mix M3 formed using 16M NaOH shows higher strength and lower water absorption as compared to mixes M1 and M2.

The SEM images of Mixes M1 at day 1, 3 and 7 illustrates that with increasing curing period the microstructure shows significant amount of dense amorphous geopolymerisation product with fewer voids. Hence, the increasing homogeneity causes high compressive strength and lower absorption over extended curing period.

5. CONCLUSIONS

- The increasing concentration of NaOH helps in easy dissolution of Al & Si ions which

increases the rate of geopolymerisation process due to readily available free ions which help in product formation in turn contributing to the increase in compressive strength.

- Water absorption obtained for GPC for 16 M is 3 to 4 % and appropriate for good working conditions of a concrete.
- With increasing curing period and concentration of NaOH, the geopolymerisation product becomes dense and amorphous filling voids and increasing homogeneity which attributes to strength gain and lower water absorption property.
- Use of naphthalene based super plasticizer, indeed reduced the use of water but, no significant change in slump values are observed.
- Geopolymer concrete could potentially replace OPC, with efficient supply chain for raw materials and a supply network. However, it would take time to place geopolymer concrete as saleable commodity in global market.

6. Scope for further studies

Study can be extended in the future with different ratios of various mineral admixtures and also use of other water reducers. And Comparative cost analysis for conventional cement concrete and geopolymer concrete can be done. The other mechanical and permeation parameters like flexure, tensile strength, permeability test should be studied for more optimised results. And other microstructure studies like Mercury Intrusion Porosimetry (MIP) to determine the pore size, volumes and connectivity to understand the long term behavioural and durability properties can be conducted.

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