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Study of mechanical and microstructural properties of geopolymer concrete with GGBS and Metakaolin*

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Abstract

Geopolymers are a new type of artificial polymer which is developed when an aluminosilicate source is triggered or activated by the application of alkaline hydroxide and silicate solution. They have shown good mechanical properties and shows good resistance to chemicals, lesser shrinkage, no damage to environment and shows formidable durability. Ground granulated blast furnace slag (GGBS) is a good aluminosilicate source as it contains high amounts of alumina and silica which are necessary for the geopolymerisation reaction to take place. In this paper, three variations in terms of weight percentages of GGBS and metakaolin clay have been taken with 80%-20%, 50%-50%, and 20%-80% respectively. A 10M solution of sodium hydroxide with sodium silicate was used as alkaline activator solution. For practical purposes ambient curing of the geopolymer samples has been adopted. The Degree of reaction was evaluated for all the percentage variations of the geopolymer concrete at 7 days of curing. The mechanical properties of geopolymer concrete have been studied by compressive test, split tensile test and four-point loading test. To understand the structural integrity of the casting of the geopolymer specimens, ultrasonic pulse velocity test was performed. The study of micro-structure of geopolymer concrete was carried out by Fourier transform infrared spectroscopy (FTIR) technique.

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Keywords: Geopolymer concrete; GGBS; metakaolin; aluminosilicate, amorphous phases

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Nomenclature

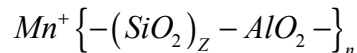
GGBS	Ground granulated blast furnace slag
GPC	Geopolymer concrete
GPCG20M80	Geopolymer concrete with 80% metakaolin and 20% GGBS by weight
GPCG50M50	Geopolymer concrete with 50% metakaolin and 50% GGBS by weight
GPCG80M20	Geopolymer concrete with 20% metakaolin and 80% GGBS by weight

1. Introduction

With the continuous growth of industries in the world, the CO₂ emissions continue to rise. Manufacturing of cement contributes about 5-7% of CO₂ emissions. A lot of studies have been done in the past which examine the greenhouse emissions from concrete and how they affect the atmosphere. The manufacturing of construction materials requires a lot of fuel and so, in turn, consumes a lot of energy [1]. GGBS, Wollastonite, fly ash and other materials have been seen as ancillary materials that can be also used in cement and have been used over the years to develop geopolymerisation reaction and the previous studies have shown that geopolymer concrete has the potential to be an alternative to standard concrete [2]. The amount of hydrothermal stability, resistance to reducing atmosphere and acidic environment, these are arguments in favor of geopolymer concrete. This new concrete shows a lower amount of carbon footprint, low amount of embodied energy and low amount of carbon emission. Davidovitis et al. was the first person to study geopolymer concrete [3]. Formation of geopolymer concrete is a result of aluminosilicate reaction triggered by the alkaline solution. There are numerous sources of aluminosilicate compounds out of which some of them are of natural origin such as metakaolin, wollastonite, and pozzolan or they may be obtained from an artificial source in the form of industrial waste such as fly ash and GGBS, which also have aluminum and silicon compounds. The reaction is initiated by the alkaline attack which further results in dissolution and hydrolysis of aluminum and silicon through alkaline reagent MOH. In these cases, the M can be an alkali or alkaline earth cation (Na⁺, K⁺, etc.). The reaction which occurs can be described as follows [4]:



The above sequence of reactions leads to the condensation process where the aluminosilicate oligomers are formed with three-dimensional amorphous structure after which the material hardening takes place. The chemical formulation obtained from previous research is [5]:



Raw materials greatly affect the chemical reactions which are responsible for the synthesis of geopolymer concrete and so affect its properties. Alteration in the microstructure properties has been observed, along with changes in the physical and chemical properties because of various type of raw materials in use [6]. The alkaline activator solution in use greatly affects the strength and structural aspects of geopolymer concrete.

2. Materials and methods:

Materials: Ground granulated blast slag (GGBS) and Metakaolin from Astra Chemicals, Sodium hydroxide and Sodium silicate from Astra chemicals and VARAPLAST PC 432 superplasticizer from Aakash Specialties have been used in this study.

Method: Preparation of alkaline activator solution was done by taking the ratio of sodium silicate to sodium hydroxide as 2.5 and a 10 M solution of each of them has been used in the above study. Preparation of NaOH was done by use of regular water and then it was brought back to room temperature by cooling, after the preparation of sodium hydroxide it was mixed with sodium silicate to obtain uniformity. This combined solution was then used as an activator in the concrete. The mix design used for Geopolymer concrete has been derived from previous research and is provided in the following table [7]:

Table 1. Mix design of the geopolymer concrete.

Components (in Kg/m ³)	GPCG20M80	GPCG50M50	GPCG80M20
GGBS+Metakaolin	400	400	400
Course aggregate (20mm and 10mm)	1312	1312	1312
Fine aggregate	724	724	724
Sodium Hydroxide solution	37	37	37
Sodium silicate solution	93	93	93
Superplasticizer	2.3	2.3	2.3

After the concrete mix was prepared, they were poured into cube moulds of dimension 100mm x 100mm x 100mm in order to perform the compressive test for all the specimens, for evaluation of mechanical properties. In order to get an idea of the structural integrity of casting of the geopolymer specimens, ultrasonic pulse velocity test was performed on the cubes of each of the percentage variations of GGBS and metakaolin. The casting of cylinders of 100mm x 200mm was done for split tensile test and casting of beams of 100mm x 100mm x 500 mm was done for evaluation of flexural aspects of geopolymer specimens. All the geopolymer specimens prepared were kept for ambient curing for practical purposes. For all the cube specimens the comprehensive strength was evaluated at curing period of 7 days and then again at 28 days of curing. Similarly, the split tensile test was performed at 7 days of curing period and 28 days of curing period respectively. After 7 days of being cured the same samples that were used for evaluation of compressive strength were used for calculation of degree of reaction. For evaluating the degree of reaction, a 2M solution of HCL was used. In this solution 3grams of the crushed samples obtained from various percentage variations in the GPC was immersed and then stirred for 20 minutes, this was done with the temperature maintained at 60°C using a water bath. The samples were filtered and then washed with distilled water and acetone. This was done 4 times to completely wash off the HCL from the original sample. For the next 2 hours drying of the samples was done at 70 °C. After drying the samples were immersed in a 3% solution of Na₂CO₃ solution and stirred for 20 minutes at 80°C [11]. Again, the drying of the samples was done for another 2hrs at the temperature of 70°C. Loss of ignition was determined using muffle furnace technique for non-volatile samples. The evaluation of s degree of reaction was done by calculating the mass difference as follows [5].

$$\text{Degree of reaction(\%)} = (m_{\text{sample}} - [m_{\text{residue}}(1 + LOI)]) / m_{\text{sample}} \times 100 \quad (3)$$

The microstructure of Geopolymer concrete was investigated by the use of FTIR spectroscopy. The use of FTIR spectroscopy is for the study of bond structure in the geopolymer samples.

3. Results

3.1 Compressive Test

The test has been used to get an idea of the potential strength of the various specimens of the geopolymer concrete he above test is performed by applying the load at right angles to any side of cubes of dimension 100mm x 100mm x 100mm. The outcome of the compressive test on various mixes is given in Table2.

Table2. Tabulation for the compressive test of GPC

Sample ID	Compressive strength (N/mm ²)	
	7 Days	28Days
GPCG20M80	19.2	27.6
GPCG50M50	24.3	32.5
GPCG80M20	37.4	49.2



Fig1. Compressive strength test

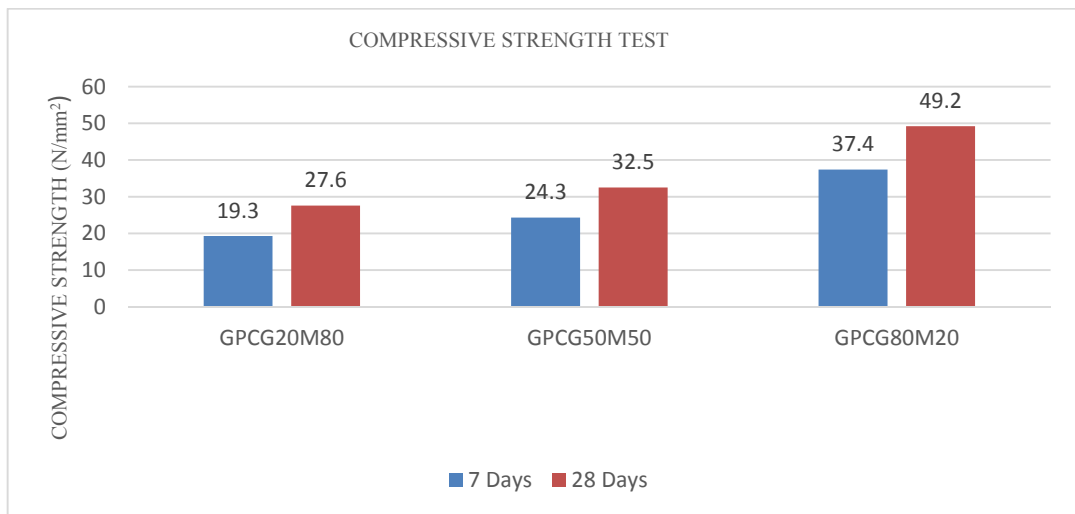


Fig2. Graphical representation of the compressive strength test

It can be inferred from the table that the geopolymer specimens of GPCG8020 show the highest amount of increase in the compressive strength value as it increases by 35.3%. The increment in compressive strength was observed with the increase in GGBS percentage in the geopolymer mix which is consistent with the fact that slag-based GPC provides with better early age strength [8].

3.2 Split Tensile Test

The above test has been used to evaluate the vulnerability of geopolymer concrete to tensile cracking. The above test was performed by application of radial load on cylinder specimens of dimensions 100mm x 200mm. The outcome of the split tensile test is furnished in Table3.

Table3. Tabulation for the split tensile test

Sample ID	Split tensile strength (N/mm ²)	
	7 Days	28 Days
GPCG20M80	2.13	2.51
GPCG50M50	2.31	2.78
GPCG80M20	3.11	3.91



Fig3. Split tensile test

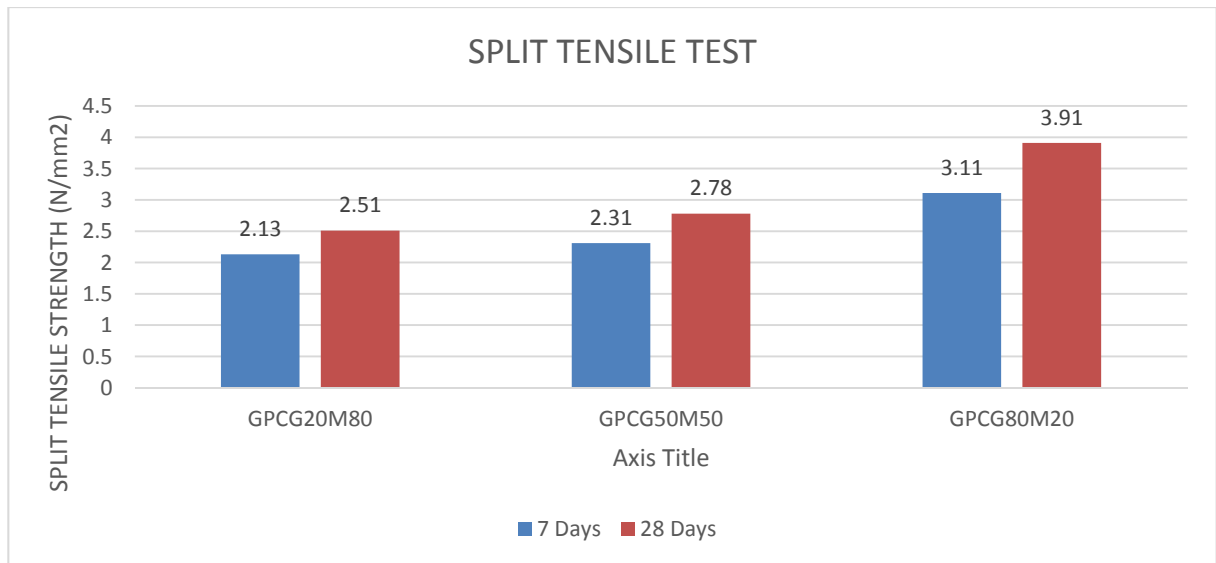


Fig4. Graphical representation of split tensile test

It can be inferred from the table that with the increase in the percentage of GGBS in the geopolymer mix the value of split tensile strength increased. The highest value is obtained for the specimens of GPCG80M20. This can be attributed to denser interfacial zone developed between the aggregates and geopolymer paste [9].

3.3 Ultrasonic pulse velocity test

The above test was performed to evaluate the continuity of casting of various proportions of geopolymer concrete. For the above test cubes of 100mm x 100mm x 100mm were used after a curing period of 28 days. The outcome of the ultrasonic pulse velocity test is given in Table4.

Table4. Tabulation for Ultrasonic pulse velocity test

Sample ID	Width of structure (mm)	Time taken for wave propagation (micro seconds)	Ultrasonic pulse velocity test (Km/s)
GPCG20M80	100	29.06	3.44
GPCG50M50	100	29.2	3.42
GPCG80M20	100	28.4	3.52



Fig5. Ultrasonic pulse velocity test

From the results of Ultrasonic pulse velocity, it can be inferred that there is no significant effect of changes in the proportion of the geopolymer regarding the continuity aspects and relative quality assessment of casting of geopolymer concrete specimens.

3.4 Flexure Analysis

Four-point loading of the 100mm x100mm x 500mm unreinforced beams was performed to get an idea of the ultimate load of the beams. Then the beams were set up with the same parameters in the UTM machine with a 1000KN capacity



Fig6. Four- point loading setup



Fig7. UTM setup for load vs deflection analysis

The results of the flexure analysis highlight's the brittle nature of geopolymer concrete. Load vs deflection curve of the various percentage variation of the geopolymer concrete is given below:

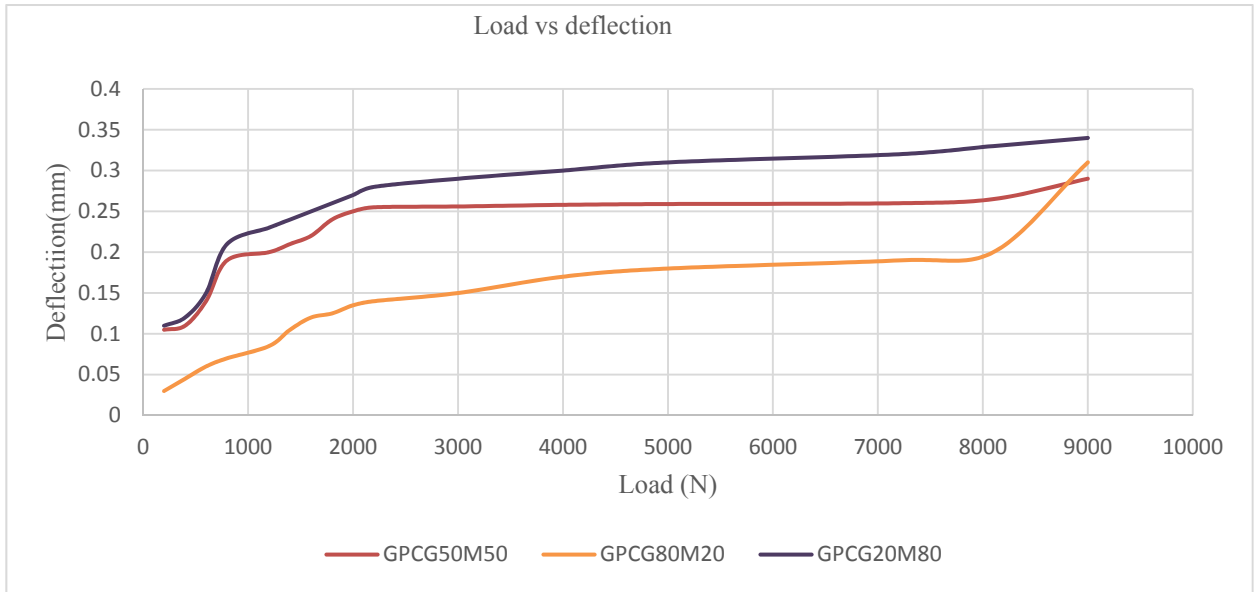


Fig8. Load vs deflection curves of various percentages of GPC

It can be inferred from the results that GPCG20M80 specimens which have higher amounts of metakaolin show greater values of deflection than the other percentages at comparatively lesser loads. With greater amounts of GGBS in the geopolymer specimens were able to resist higher amount of loading. The GPCG80M20 specimen's curve is consistent with the nature of geopolymer as it was observed a linear evolution of load which was then followed by fracture [10].

3.6 Degree of reaction

The degree of reaction of the percentage variations in the geopolymer is shown in figure9.

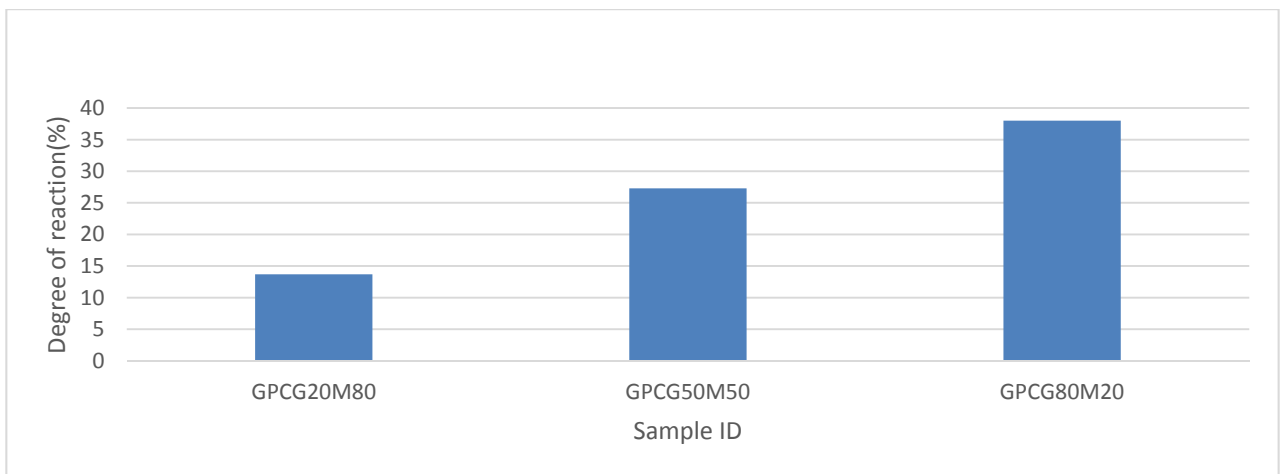


Fig9. Degree of reaction values of the percentage variation of the GPC

The degree of reaction values obtained is 13.7, 27.3 and 38 for GPCG20M80, GPCG50M50, and GPCG80M20 respectively. The GPCG80M20 specimens gave the highest amount of the degree of reaction. But the degree of

reaction obtained for GPCG20M80 was much lower in comparison to the other percentage variations. With the increase in the percentage of the GGBS in the geopolymer concrete, the degree of reaction increased drastically.

3.5 FTIR Spectroscopy

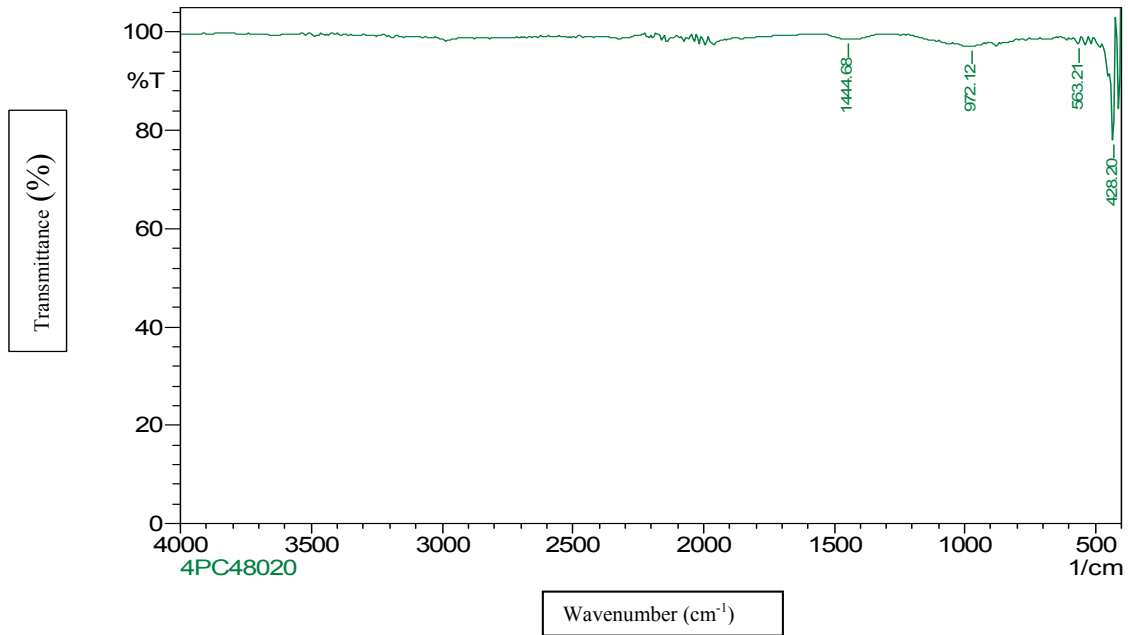


Fig10. FTIR spectrum of the sample GPCG80M20 at 28 days

Peaks at around 972 cm^{-1} indicate that there is some degree of geopolymerisation taken place, as it indicates the presence of stretching vibrations of Al-O and Si-O [11]. The height of the peaks is slightly less considering that samples had been placed in ambient curing. The 1448 cm^{-1} indicates the presence of the slight amount of carbonation as it represents the reaction of atmospheric carbon dioxide with the geopolymer matrix [12]. Beyond 2000 cm^{-1} minor peaks indicate the presence of some amount of hydration. The 563.21 cm^{-1} are due to the out of plane bending of the Si-O bonds and also shows the presence of octahedrally configured aluminium. The 428.2 cm^{-1} peak is of most interest as the presence of peak indicates the presence of bending within the plane of Al-O along with Si-O which is largely contributed by the Aluminium, considering the high $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratio of the specimens of GPCG80M20 due to a large amount of GGBS used in the casting [11]. Minor peaks near to 1000 cm^{-1} indicate the presence of some asymmetric stretching of the Al-O and Si-O bonds [13]. From the FTIR spectroscopy, it can be inferred that the peaks at around 1000 cm^{-1} indicate the degree of geopolymerisation taken place, as they provide an idea of the aluminium incorporation and amount of polysilication taken place.

4. Discussion

This study concentrated on the effect of changes in the percentage variation of aluminosilicate sources in the geopolymer concrete. It was observed that with the increase in the percentage of GGBS in the geopolymer concrete there was a significant improvement in the compressive strength and split tensile strength. This can be attributed to the formation of adequate reaction products in case of greater slag percentage. Low values of the degree of reaction were obtained for all percentage variations in the GPC. This can be attributed to the presence of crystalline phases in the geopolymer matrix, which reduces the effect of amorphous groups present inside it. FTIR spectroscopy indicated the presence of characteristic bonds of geopolymer concrete. It can be seen from the load-deflection analysis that geopolymer concrete shows a brittle nature compared to regular concrete, because of a higher amount of crosslinking in GPC. After the evaluation of the mechanical, chemical and microstructure properties of geopolymer

concrete, and keeping the aspects of environment-friendly production, sustainability in mind, GPC can be recommended for construction works.

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