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Mix Design and Mechanical Properties of Fly Ash and GGBFS-Synthesized Alkali-Activated Concrete (AAC)

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Abstract: Cement is one of the construction materials widely used around the world in order to develop infrastructure and it is also one of the factors affecting economies. The production of cement consumes a lot of raw materials like limestone, which releases CO₂ into the atmosphere and thus leads to global warming. Many investigations are underway in this area, essentially focusing on the eco-accommodating environment. In the research, an alternative material to cement binder is geopolymer binder, with the same efficiency. This paper presents scanning electron microscope (SEM) and X-ray diffraction (XRD) analysis of factory byproducts (i.e., fly ash and ground granulated blast furnace slag (GGBFS)). The mix design process for the manufacture of alkali-activated geopolymer binders synthesized by fly ash and GGBFS is presented. The mechanical properties (compression, split tensile and flexural strength, bond strength) of geopolymer concrete at different mix proportions and at dissimilar curing conditions were also investigated. Geopolymer concrete synthesized with 30% fly ash and 70% GGBFS has better properties at 14 M of NaOH and cured in an oven for 24 h at 70 °C.

Keywords: SEM; XRD; geopolymer binder; CO₂; fly ash; GGBFS; properties

1. Introduction

Worldwide cement production has grown incredibly in recent years. After fossil fuels and land-use change, it is the third biggest basis of anthropogenic emissions of CO₂ (carbon dioxide) [1]. A future challenge to the construction industry is to use alternative materials to replace cement with industrial by-products [2]. In the year 1972, “Joseph Davidovits” discovered a new class of inorganic material: geopolymer binder/resin. One of the main reasons for the development of geopolymers is to utilize industrial wastes as well as to control the emission of greenhouse gases emitted by cement production. Inorganic materials-based geopolymer concrete are the same as conventional concrete but these inorganic materials should be rich in silica and alumina-like fly ash, silica fume, GGBFS (ground granulated blast furnace slag) with soluble solutions (sodium or potassium) [3,4]. The main components of geopolymer concrete can be generally divided into two categories: alkaline liquids and the source materials. The alkaline activation on these industrial by-products will result in the configuration of geopolymer binders [3]. A full substitution of cement in the geopolymer concrete manufacturing with the byproduct materials reduces CO₂ emissions into the atmosphere [5]. Geopolymer concrete typically shows evidence of outstanding compressive strength, restricted durability and low flexural quality, which may confine its utilization in auxiliary constructions [6]. There are so many factors

affecting the mechanical and durable properties of geopolymer concrete. These are: fineness of fly ash and GGBFS, molarities of NaOH solution, and curing conditions (sunlight and oven) [5,6]. With an increase of molarities of NaOH solution, the mechanical properties are also increased. Generally up to 14 M NaOH solution is economic and it is suggested can improve the durability of geopolymer concrete [7]. Table 1 indicates the density, surface area and distribution of particle sizes of different materials like ordinary Portland cement (OPC), flyash, silica fume and GGBFS.

Table 1. Particle size distribution, Specific surface area and density data of cement and industrial byproducts [8].

Cement and Industrial Byproducts	Density (g/cm ³)	Specific Surface Area (m ² /g)	Particle Size Distribution (mm)
OPC (type I) (I33)	3.12	0.85	1.1–50
Class F flyash (G 05)	2.45	0.7	2.0–40
Granulated blast-furnace slag (G42)	2.9	0.75	0.70–40
Silica fume (G-15)	2.05	18.02	0.1–5.0

In this paper, the chemical composition of cementitious materials (fly ash and GGBFS) and mechanical properties like compressive, split tensile and flexural strengths of geopolymer concrete at dissimilar curing conditions are presented. Alkali-activated binders synthesized with fly ash and GGBFS and their mix proportions at dissimilar NaOH molarities are also explained. The available literature on fly ash GGBFS-synthesized geopolymer concrete was limited to sunlight or ambient or steam-curing conditions. The present study is performed to analyze the mechanical properties of fly ash and GGBFS synthesized geopolymer concrete by placing the specimens in an oven for 24 h and simultaneously curing under sunlight for 3, 7, 14 and 28 days.

Development of Geopolymers

The improvement and research of geopolymeric materials has been presented since 1930 and the distinctive research papers are accessible in this field; many researchers have investigated the development of geopolymers and Table 2 provides brief information about the authors, recent investigations on geopolymer concrete (GPC) and significance of work.

Table 2. Development of geopolymers and their significance.

Author	Year	Significance
Davidovits [9]	1972	Discovery of Geopolymers
Davidovits [10]	1989	Geopolymeric new materials
Davidovits [11]	1994	Geopolymer cements
Davidovits [12]	2002	Successes and Failures in Geopolymer Applications
P. Duxson et al. [13]	2006	Current state of the art
Peter Duxson et al. [14]	2007	Geopolymer technology in the development of green concrete
Komljenovi M et al. [15]	2010	Microstructural properties
Kong DLY and Sanjayan JG [16]	2010	Structural composites for high temperatures
PetrSazama et al. [17]	2011	Geopolymer based catalysts
Jannie S.J. van Deventer et al. [18]	2011	Geopolymer cement
Davidovits [19]	2013	Foundry Resins (Nano-poly (Silaxilanol))
Zuhua Zhang et al. [20]	2014	Geopolymer foam concrete
Yun Yong Kim et al. [21]	2014	Durability Performance
Sara Dashti [22]	2015	Use of Lake Michigan Dredged Materials
Dr. Konstantin Sobolev et al. [23]	2015	Optimized Concrete Pavement
Naskar and Chakraborty [24]	2016	Nano materials in geopolymer concrete

2. Material and Properties

Fly ash particles are spherical in shape, obtained from a mid-burning of powdered coal. The chemical composition of fly ash contains silica, alumina and minor measured oxides viz: Fe, Na, Ca, Mg and K. The synthetic constituents in class F fly ash are silica and alumina, both accounts for over 90% of chemical compounds present in fly ash, which was obtained by XRF (X-ray fluorescence)

analysis [7,25,26]. The flyash was obtained by National Thermal Power Corporation, Vijayawada, Andhra Pradesh, India and its specific gravity value is mentioned in Table 3. GGBFS was acquired from JSW cements, Vijayawada, A.P. and India. Figure 1a,b depict the X-ray diffraction (XRD) analysis of fly ash and GGBFS samples, respectively.

Table 3. Specific gravity and density of different materials.

Materials	Specific Gravity
Coarse aggregate	2.71
Fine Aggregate	2.48
Fly ash	2.1
Ground Granulated Blast Furnace Slag	2.22
Density of Sodium Silicates	1.53
Sodium Hydroxide	1
Water	1
Super Plasticizer	2.12

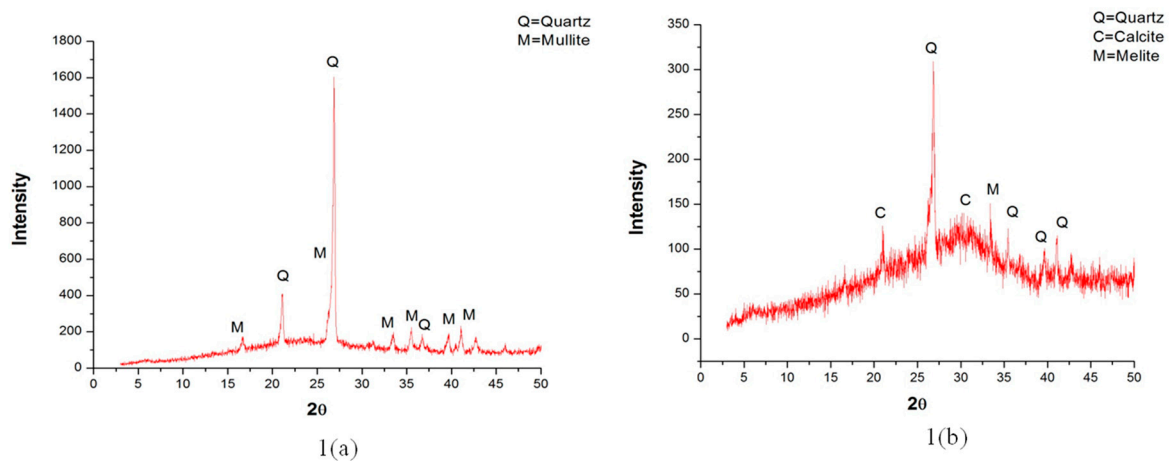


Figure 1. (a) X-ray diffraction (XRD) of flyash inorganic sample, (b) XRD of ground granulated blast furnace slag (GGBFS).

Scanning electron microscope (SEM) analysis was conducted on fly ash and GGBFS samples to determine the particle size distribution. Figure 2a,b show the microscopic images of fly ash and GGBFS, respectively. The SEM images of fly ash particles depict the shape as spherical and size ranging from 14 to 23 μm , while the shape of GGBFS particles tetrahedron and size of particles range from 2 to 6 μm . Geopolymers are generally formed by the synthesis of fly ash by alkali activators [27].

Alkali-activation of alumino-silicate materials like GGBFS and fly ash is a complex process. In the present paper, fly ash was synthesized by alkaline solutions viz, sodium silicate and sodium hydroxide. The alkaline solution was prepared by mixing of NaOH pellets with water (depending on molarities) for 8 to 10 min and then mixed with sodium silicate solution. The NaOH and Na_2SiO_3 ratio was maintained constant at 2.5. Locally available river sand containing zone-II as per IS 383-2016, specific gravity was 2.48 (Table 3), fineness modulus was 2.20. Coarse aggregates of maximum size 20 mm were used and the fineness modulus and specific gravity were 6.97 and 2.71 respectively. Consumable water with pH value 7.11 as utilized for the geopolymer concrete preparation. In the mix of geopolymer concrete, super plasticizer was used to improve the workability of concrete by adding at different percentages. In the present work, sulphonated naphthalene based super plasticizer was used to increase the workability of GPC.

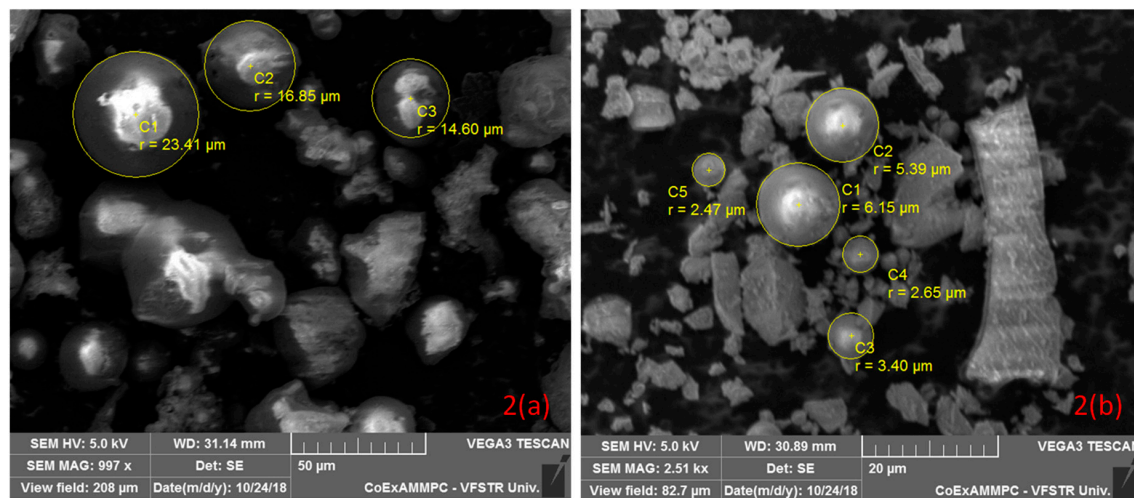


Figure 2. (a) Scanning electron microscope (SEM) image of fly ash at 50 μm , (b) SEM image of GGBFS at 20 μm .

3. Mix Proportions

Standards

According to Davidovits [12], the ranges of material ratios for geopolymer concrete mixes are indicated in Table 4. The alkaline liquid to binder ratio was suggested as 0.3–0.45, and sodium silicate to sodium hydroxide solution as 2.0–2.5. Total content of aggregate in the mass of geopolymer concrete is 65–85%, and the content of fine aggregate in total aggregate volume is chosen as 30%. The range of super plasticizer is taken as 1.5–4% by mass of binder content. The extra water content if needed can be added in the range of 0.02–0.06% by mass of cementitious material.

Table 4. Standards of materials used in geopolymer concrete mixes.

Materials	Range
Alkaline liquids/Binder	0.3–0.45
Sodium silicate/sodium hydroxide	2.0–2.5
Water/Binder	0.16–0.24
Total aggregate in mass of concrete	65–85%
Fine aggregate content in total aggregate	30%
Added water content	0.02–0.06% of mass of cementitious material
Super Plasticizers	1.5–4% of mass of cementitious material

The different materials used in the geopolymer concrete mix design are fly ash, GGBFS, sodium silicate, sodium hydroxide, fine aggregates, coarse aggregates, water and super plasticizer. The mix proportions of all these materials are carried with the base of unit weight of plain concrete (2400 kg/m^3). The proportions of coarse and fine aggregates used were 70% and 30% respectively. The ratio of alkaline liquid to cementitious materials was taken as 0.35 and ratio of sodium silicate and sodium hydroxide was 2.5. Super plasticizer of 4% was used to improve the workability of fresh geopolymer concrete. The mix design of different materials is presented in Table 5 for 6 M NaOH. In a similar way, the design mixes were prepared for 8 M, 10 M, 12 M and 14 M.

The quantity and percentages of different materials for the geopolymer concrete mix is listed in Table 5 but to prepare the unit weight different materials for 1000 kg, corrections are required for coarse and fine aggregates because the absolute weight becomes 1038.82 kg. The corrections are done for unit weight of fine and coarse aggregates; we take coarse aggregate in total mass of aggregate as 1176 kg/m^3 but after correction it was 1102.36 kg/m^3 . Fine aggregate in total mass of aggregate was 504 kg/m^3 , and after correction it was 475.12 kg/m^3 . These values are tabulated in Table 6.

Table 5. Mix design of different materials per cubic meter (6 M NaOH).

Materials used	Quantity
Unit weight of concrete	2400 kg/m ³
Percentage of aggregate in total mass of concrete	70%
Aggregate content in total mass of concrete	1680 kg/m ³
Percentage of fine aggregate in total mass of aggregate	30%
Fine aggregate in total mass of aggregate	504 kg/m ³
Coarse aggregate in total mass of aggregate	1176 kg/m ³
Cementitious materials used	
Ratio of alkaline liquid to cementitious material	0.35
Mass of Cementitious material and alkaline liquid	720 kg/m ³
Mass of Cementitious material	533 kg/m ³
Mass of alkaline liquid	187 kg/m ³
Fly ash (30%)	159.9 kg/m ³
GGBFS (70%)	373.1 kg/m ³
Alkaline liquids required	
Ratio of sodium silicate and sodium hydroxide	2.5
Mass of sodium hydroxide	53 kg/m ³
Mass of sodium silicates	134 kg/m ³
Water required in Sodium silicate	
Na ₂ O	15.30%
SiO ₂	33.69%
H ₂ O	51.01%
Water content in sodium silicate	69 kg
solids content in sodium silicate	65 kg
Water required in sodium hydroxide	
Molarity ratio	6
Mass of NaOH solids	240 gm
NaOH	24%
H ₂ O	76%
Solid content in sodium hydroxide	13 kg
Water content in sodium hydroxide	40 kg
Total water content	109 kg
Total solid content	611 kg
Water to cementitious material ratio	0.178
Super plasticizer requirement	
Super plasticizer	4%
Mass of super plasticizers	24.44 kg/m ³
Percentage of extra water content	1%
Extra water content	5.33 kg/m ³

Table 6. Absolute unit weights and corrected unit weight of the materials.

Material	Absolute Weight of Material	Material	Absolute Weight of Material
Coarse Aggregate (CA)	433.94 kg	Standard weight	1000 kg
Fine Aggregate (FA)	203.22 kg	Difference	38.82
Fly ash	76.14 kg	Coarse Aggregate Difference	27.17
GGBFS	168.06 kg	Fine Aggregate Difference	11.64
Sodium Silicates	87.58 kg	Coarse Aggregate corrected	406.77
Sodium Hydroxide	53 kg	Fine Aggregate corrected	191.58
Water	5.33 kg	Unit weight corrected CA	1102.36 kg/m ³
Super Plasticizers	11.52 kg	Unit weight corrected FA	475.12 kg/m ³
Absolute Weight	1038.82 kg	Water to cementitious material ratio	0.178

The quantity of materials was prepared with the help of unit weight of those materials (corrected values are used for fine and coarse aggregates). The material quantities were prepared for 25 cubes, 12 cylinders and 4 prisms in each mix. Depending on these quantities of materials, specimens were prepared for testing purposes. All the mix design values were prepared in a Microsoft Office Excel worksheet, and these values are shown in Table 7. By doing these values in the worksheet, it will help to prepare the quantities for different molarities of NaOH, different percentages of cementitious materials (fly ash and GGBFS ratios).

$$\text{Material quantity} = \text{Unit weight} \times \text{Dimensions of mould} \times \text{No of moulds}$$

Table 7. Mix proportions weight of all materials for 25 cubes, 12 cylinders and 4 prisms.

Materials (kg)	M1 (6M NaOH, 30% GGBS & 70%FA)	M2 (8M NaOH, 40% GGBS & 60%FA)	M3 (10M NaOH, 50% GGBS & 50%FA)	M4 (12M NaOH, 60% GGBS & 40%FA)	M5 (14M NaOH, 70% GGBS & 30%FA)
Coarse aggregate	132.26	132.26	132.26	132.26	132.26
Fine aggregate	57.01	57.01	57.01	57.01	57.01
Flyash	44.87	38.46	32.05	25.64	19.23
GGBFS	19.23	25.64	32.05	38.46	44.87
Mass of sodium silicate	7.81	7.81	7.81	7.81	7.81
Total water content in sodium silicate	8.29	8.29	8.29	8.29	8.29
Total sodium silicate	16.11	16.11	16.11	16.11	16.11
Mass of sodium hydroxide	1.56	2.04	2.64	3.12	3.6
Total water content in sodium hydroxide	4.81	4.33	3.72	3.24	2.76

4. Geopolymer Binders Synthesized by Fly Ash and Ground Granulated Blast Furnace Slag (GGBFS)

Flyash and GGBFS synthesized geopolymer mortar is prepared similar to Portland cement mortar by replacing the cement with fly ash and GGBFS and water with alkaline activator solution. The word “Geopolymer” portrays a group of mineral fasteners that have a polymeric silicon-oxygen-aluminum system structure, like that found in zeolites, however without the crystal structure [28]. The geopolymerization procedure includes a significantly rapid response in the very basic form on Si-Al minerals with the intention of outcome a three-dimensional polymeric chain and ring structure comprising of Si-O-Al-O bonds. Geopolymers are regularly combined by blending industrial byproducts containing alumino-silicate and the basic arrangements. Source materials utilized are kaolinite, clays, zeolite, flyash, silica fume, slag, POFA (palm oil fuel ash), rice-husk powder, red mud and some other [29]. The majority of widely recognized antacid fluid utilized in geopolymerization is a blend of NaOH/KOH and Na₂SiO₃. At the point when any of the above source materials for instance fly ash (FA) in strong frame are blended by means of antacid arrangements of suitable fixation and Na₂SiO₃, geopolymers are shaped amid the polymerization response of fly ash [30]. Dissimilar molarities of NaOH solution (Figure 3a), fly ash percentage (Figure 3b) and GGBFS percentages (Figure 3c) for different geopolymer concrete mixes are shown in Figure 3.

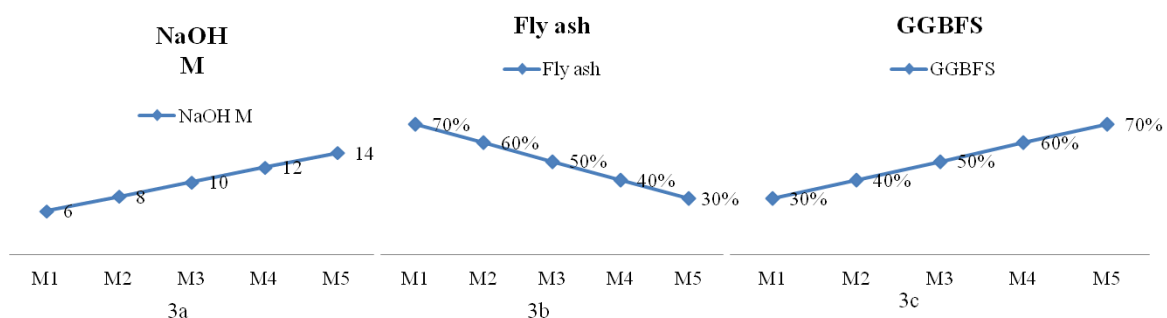


Figure 3. Dissimilar molarities of NaOH solution (a), fly ash percentage (b), and GGBFS percentages (c) for different geopolymer concrete mix.

The execution, including quality and strength of geopolymer concrete, are fundamentally impacted by synthesis of material mix, substance composite of a binder, salt activator, water substance, and relieving condition. The fixation and measurements of soluble base activator influence the rate of polymerization and decide the last quality of solidified geopolymer. Also, the activator molarity arrangement would influence the water content too. Hydroxyl particles (water) in activator arrangement add for the freedom of Si and Al from the geopolymer cover. All in all, geopolymerization increment with restoring time and relieving temperature (up to 90 °C) [31]. The higher modulus of elasticity is accomplished utilizing warm restoration because of a higher level of geopolymerization [32]. Amid the restoring procedure, the geopolymer solid encounters the geopolymerization procedure [33]. The type of alkaline solution plays a major role in the process of polymerization. Reactions occur at a high rate when the alkaline solution contains soluble silicate, either sodium/potassium silicate, compared to the use of only alkaline hydroxides [34].

5. Engineering Properties of Geopolymer Concrete

Higher NaOH concentrations or molarities is more powerful in dissolving fly ash particles and can result in a superior geopolymerization flyash particle. This is not only in geopolymerization but also affects the mechanical properties of geopolymer concrete. The engineering properties were calculated at different curing temperatures, dissimilar concentrations of NaOH solution and at different material (fly ash and GGBFS) proportions. All the tests were carried in different equipment like the compressive testing machine and Flexural testing machine after, the completion of curing time of 24 h in an oven at 70 °C and sunlight curing for 3, 7, 14 and 28 days, respectively the entire testing was carried according to IS 516:2013. The testing of geopolymer concrete specimens (cube, cylinder and prisms) is shown in Figure 4.



Figure 4. (a) Geopolymer concrete cylindrical specimens casting, (b) casting of cube specimens, (c) cube and cylindrical specimens, (d) cylindrical specimens for oven curing, (e) testing of cube for compressive strength, (f) Failure of specimens after testing.

5.1. Compressive Strength

To determine the compressive strength of geopolymer concrete, 150 × 150 × 150 mm cubical moulds were used and the specimens were placed for one day (24 h) in oven at a constant temperature of 70 °C, simultaneously the specimens were further cured at sunlight for 3, 7, 14 and 28 days respectively. The effect of dissimilar molarities of NaOH solutions and different cementitious material proportions on compressive strength of GPC is shown in Figure 5.

$$\text{Compressive Strength} = \frac{\text{Maximum failure load (N)}}{\text{Area of the specimen (mm}^2\text{)}} \quad (1)$$

The results depict the influence of molarity of NaOH on improving the strength of concrete. It can be obtained that the increase in molarity from 6 to 14, improved strength by 35% (average) irrespective of curing condition. It can also be noticed that the age of curing significantly affects the strength characteristics of GPC. Out of all mixes, M5 i.e., GPC with 14 M NaOH performed better, and this was oven cured for 24 h at 70 °C followed by sunlight cured for 28 days. The results necessitate the requirement of age of curing in improving compressive strength of GPC.

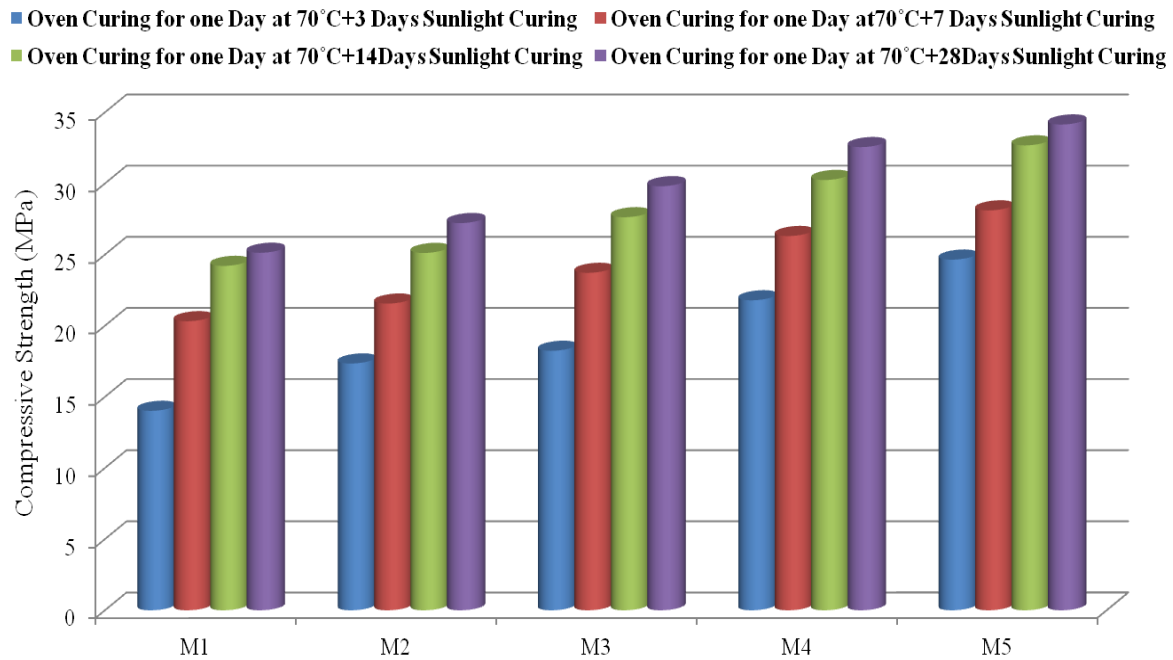


Figure 5. Compressive strength of geopolymer concrete at different mix proportions.

5.2. Split Tensile Strength

To calculate the split tensile strength of geopolymer concrete, cylindrical moulds (150 × 300 mm) are used and the specimens have one day of oven curing at a constant temperature of 70 °C then sunlight cured for 3, 7, 14 and 28 days respectively. The split tensile strength of geopolymer concrete at these curing conditions is mentioned in the Figure 6. The geopolymer concrete mix consists of different molarities of NaOH (6, 8, 10, 12 and 14), out of all these proportions the 14 M mix (M5) achieved higher tensile strength values. The Equation (2) is used to calculate the split tensile strength of concrete.

$$\text{Split Tensile Strength (MPa)} = \frac{2P}{\pi DL} \quad (2)$$

where D and L are diameter and length of cylinder specimen.

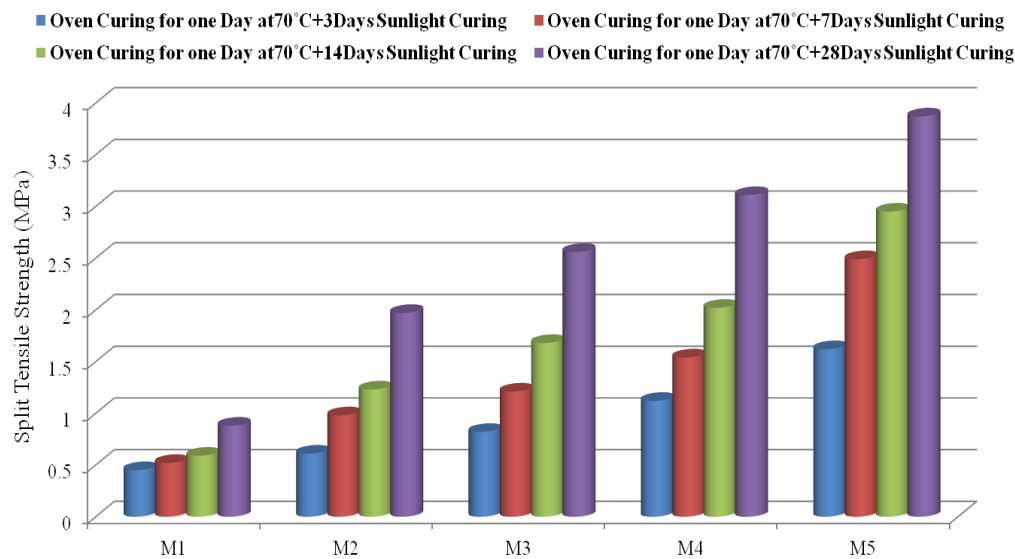


Figure 6. Split tensile strength of geopolymer concrete at different mix proportions.

5.3. Flexural Strength

The flexural strength of geopolymer concrete by the use of simple beam/prisms with third point loading, this procedure is commonly referred to as modulus of rupture. To determine the flexural strength of geopolymer concrete, prism moulds (100 × 100 × 500 mm) were used and the specimens underwent one day oven curing at a constant temperature of 70 °C simultaneously sunlight curing for 14 and 28 days respectively. The flexural strength of geopolymer concrete at these curing conditions is mentioned in Figure 7. The geopolymer concrete mix consists of different molarities of NaOH (6, 8, 10, 12 and 14 M), and out of all these proportions the 14 M mix (M5) was gained higher flexural strength values at 24 h in the oven (70 °C) + 28 days sunlight curing. In total, 4 beam specimens were prepared in each mix and cured for 24 h in an oven plus 3, 7, 14 and 28 days in sunlight.

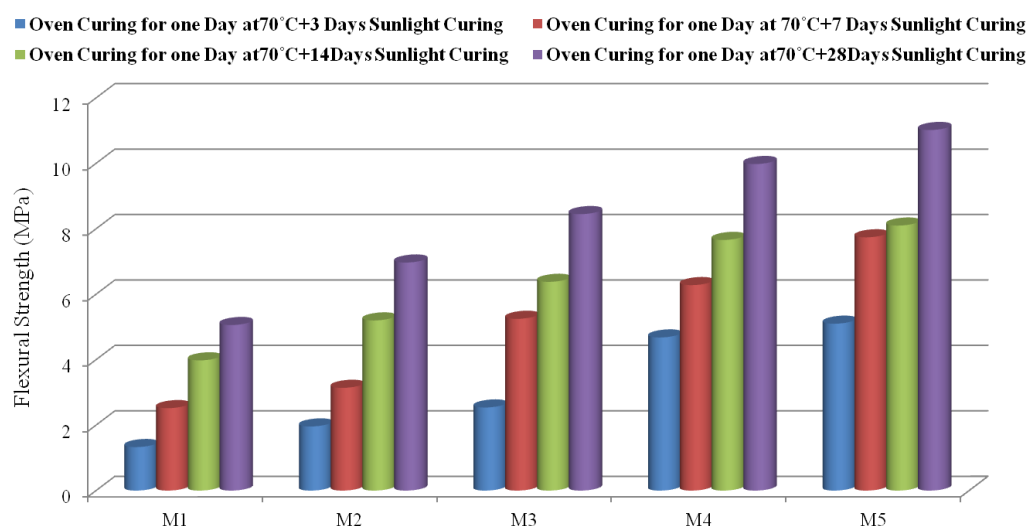


Figure 7. Flexural strength of geopolymer concrete at different mix proportions.

6. Conclusions

The present research paper explains the mechanical characteristics of fly ash and GGBFS-synthesized geopolymer concrete with dissimilar molarities of NaOH. The conclusions obtained from the study are:

1. The GPC specimens were cast with inorganic materials (fly ash and GGBFS) and cured at a constant temperature of 70 °C for one day then further cured at sunlight for 3, 7, 14 and 28 days. In these curing conditions, the compressive strength of GPC was higher at 70 °C oven curing for 24 h as well as 28 days of sunlight curing, that is 34.15 MPa (for mix M5).
2. The maximum split tensile strength was 3.87 MPa obtained at 14 M of NaOH solution and at 70 °C oven curing for 24 h as well as 28 days of sunlight curing. Similarly, the maximum flexural strength value is 11.02 (for mix M5) at the curing of 70 °C in the oven and 28 days of sunlight.
3. The increase of molarity of NaOH solution from 8–14 M, the compressive strength, splitting tensile strength and flexural strength of GPC were increased by 33%, 26% and 42.5% respectively.
4. Oven curing at higher temperatures was not undertaken according to previous research, and it is better to consider some fiber reinforcement in the geopolymer mix for future studies to improve additional properties; the scope is there for durability analysis at different environmental conditions.
5. Geopolymer concrete one of the eco-friendly alternatives to ordinary Portland cement concrete because it utilizes less raw material. For this reason, geopolymer concrete is a strong material that provides sustainable development for infrastructural needs.

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