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RESEARCH ARTICLE

Effect of Sodium Oxide on Physical and Mechanical properties of Fly-Ash based geopolymer composites

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Abstract

Objective: To analyze the effect of variation of Na_2O content on the compressive strength and water absorption of geopolymer paste and mortar specimens. **Method:** Alkali Activator solution was prepared by mixing sodium silicate solution and sodium hydroxide pellets in appropriate proportions 1 day before its use. The prepared solution was mixed with fly ash (FA) for 5 minutes in Hobart Mixer for geopolymer paste preparation and further sand was mixed to obtain geopolymer mortar. These specimens were placed in a mold and kept in an oven at 48°C for 48 hours. Compressive strength and water absorption tests were conducted on the specimens afterward. In this study, Na_2O with 5%, 6.5% & 8.0% was analyzed for its effect on geopolymer paste and mortar. **Findings:** This research helped in determining the appropriate quantity of NaOH pellets required for geopolymer composite preparation and reducing the cost of construction, as NaOH pellets are more expensive than sodium silicate solution. Maximum compressive strength was achieved by using 8% Na_2O content and water absorption was observed maximum at 6% of Na_2O content. **Novelty:** This study is the first of its kind, which has analyzed the effect of Na_2O content variation on water absorption and compressive strength of geopolymer paste and mortar specimens.

Keywords: Geopolymer composite; compressive strength; water absorption; Sodium Oxide; fly ash

1 Introduction

Fly ash (FA) obtained from biomass and coal combustion in thermal power plants has been extensively used for the preparation of alkali-activated materials and geopolymer[1]. To obtain other binding properties similar to that of the Portland cement (PC), FA is generally mixed with basic alkaline activators such as Na_2SiO_3, CaO, and NaOH[2]. These are a class of mineral binders synthesized by activation of the source materials that could be natural or by-products of the industries, which has a high content of alumina and silica with an alkali metal hydroxide/ silicate solution at moderate temperatures [3]. These binders have been reported to possess better properties than ordinary Portland cement (OPC) based binders [4].

Unlike Portland cement hydration, "interstitial or structural water" has been incorporated into the geopolymer gel production, due to which it has caused a reduction in demand of the mixing water [5]. Also, a certain percentage of free water is required for the product to be homogeneous and workable [6] and this water evaporates at 150°C [7]. Geopolymer concrete production using FA is an alternative to conventional OPC concrete due to high early strength gain, durability, less carbon emission, and less cost involved [8]. With fixed composition (75%wt of wood biomass FA and 25%wt of metakaolin), sodium hydroxide and sodium silicate (1.0 and 1.5 weight ratios), two concentrations of alkali activators, one with and another without moisture and temperature control, five curing methods were applied [9]. Also, micro-pore formation began on the replacement of 100% of POFA in FA based geopolymer when exposed to high temperatures from 300 °C to 500 °C [10].

It was analyzed that mechanical properties were enhanced with alccofine with a simultaneous reduction in GPC transport properties. Without elevated heat curing, compressive strength of 42 MPa was achieved in maximum. [11]. It was told that to gain good mechanical strength, water content is an important parameter in the synthesis of FA based geopolymer. Reduction in water content during geopolymer synthesis generally results in an increment in the compressive strength. Also, compressive strength is affected due to sodium hydroxide concentration in geopolymer, with increasing molarity of the NaOH, the strength generally increases [12], however after a certain point, it affects the strength negatively. The molarity corresponding to NaOH concentration of 6.6 M resulted in geopolymer materials with a 38.5 MPa compressive strength. The authors also stated that for their particular mix composition, the optimum concentration of the NaOH is 6.6M and specimens developed greater or lower NaOH concentration than 6.6M resulted in lower compressive strength [13].

It was further observed that with an increase in NaOH molarity, there is a significant effect on the setting time of geopolymer paste. The decrease in setting times from 66-95 minutes to 37-68 minutes with an increase in FA content from 45% to 75% along with an increase in NaOH molarity from 8M to 12M was found. It was also found that there was a rapid increase in the compressive strength of specimens in the first 24 hours, afterwards, there was a sharp decline in the rate of increase as concluded by the author [14].

Relevant studies have also found that initial SiO_2 and Al_2O_3 content have effects on the stability of the amorphous geopolymer phase in the long term concerning crystallization. A gradual transformation from the amorphous phase(s) to crystalline phase(s) in some mixtures was observed when subjected to prolonged curing. In particular, molar ratios of Al_2O_3/ SiO_2 if not equals to 3.8 then an increase in Na_2O content supports the transformation [15].

1.1 The objective of the study

1. Study of physical and mechanical properties of manufactured geopolymer composites, which are FA based with varying composition of sodium oxide in a hardened state.

To accomplish the objective, the experimental work consists of testing a total of six geopolymer mortar and paste specimens prepared by varying alkali content (%Na_2O) in unexposed condition. Compressive strength was determined to study the behavior of FA-based geopolymers. Also, simple yet important tests like bulk density, water absorption were conducted.

1.1.1 Materials used

Fly Ash: Class F FA [16] of low calcium concentration was procured from the Thermal Power Plant of Kolaghat, West Bengal, India, and used as the solid aluminosilicate source material for manufacturing FA-based geopolymer concrete. Before the commencement of the research work, a required quantity of FA was brought in bulk packed in bags from the thermal power plant. The FA was then remixed thoroughly to ensure homogeneity and replaced in sealed plastic containers until its use for the manufacture of a geopolymer. The specific gravity of FA was found to be 2.73. The particle size distribution in percentage by volume passing size is shown in Figure 1. Nearly 75% of the particles of the FA were smaller than 45 micrometers. The specific surface area determined by Blaine's method was 380m2/kg.

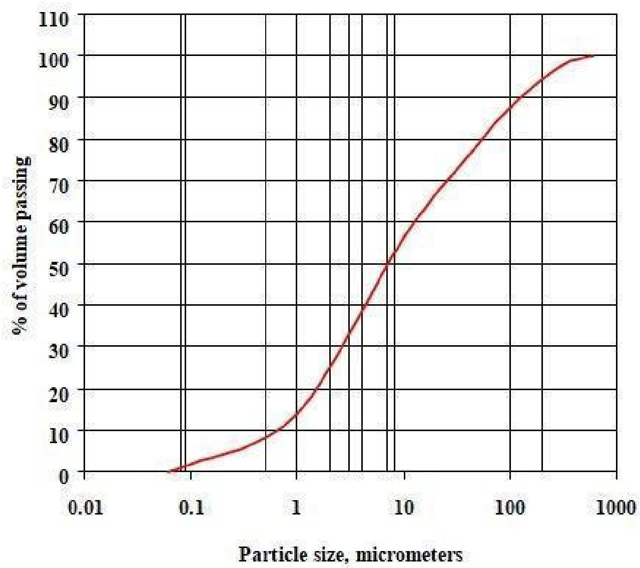

Fig 1. Particle size distribution

Fine Aggregate: Fine aggregate such as well graded river sand which passed through 4.75mm IS sieve was used. The sand was washed for removal of any dirt associated with it, air-dried, sieved, and used in saturated surface dry conditions for making geopolymer mortar specimens. For mortar, locally available sand was used from the nearby river. The fineness modulus and a specific gravity of the sand were 2.50 and 2.65 respectively. Sieve analysis results are shown in Figure 3. Selected sand falls in Zone-II due to its particle size distribution, as per IS: 383-2016 classifications[17]. Figure 2 depicts the percentage of fine aggregate passing through IS sieves of different sizes.

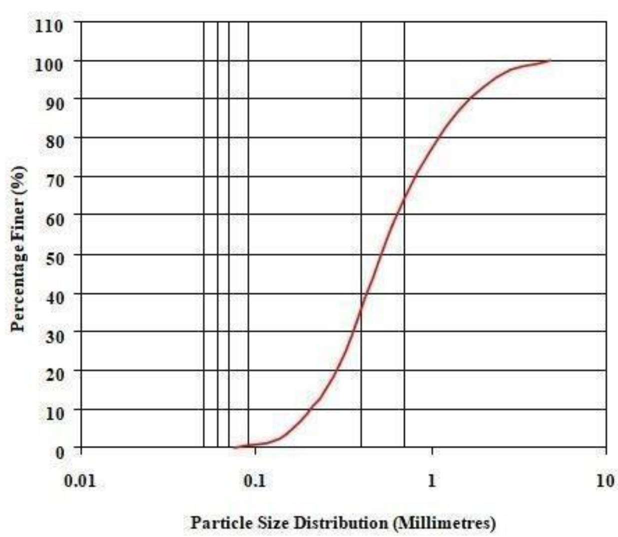

Fig 2. Results of sieve analysis of fine aggregate

Alkali activator: A mixture of Na_2SiO_3 solution, NaOH solids, and water was used as an alkaline activating solution. NaOH solids used were of laboratory grade in the forms of pellets with 98% purity and a specific gravity of 2.15. Sodium silicate (Na_2SiO_3) solution had a chemical composition of Na_2O=8%; water=65.5%; SiO_2=26.5% and bulk density of 1410 kg/m^3. Chemicals were procured from Loba Chemie Ltd, India. To make sodium hydroxide solution of desired Na_2O content, required quantities of water and sodium hydroxide pellets were mixed in the initial stage. Afterward, it was mixed with an appropriate quantity of Na_2SiO_3 solution for the desired content of SiO_2. The mixture of activator solution finally obtained was left for 1 day at ambient temperature before its use in geopolymer mix preparation.

Ground Granulated Blast Furnace Slag (GGBFS): It is a by-product obtained from the cooling of molten iron found in a blast furnace within steam or water, which is afterward dried and grounded into a fine powder and thus finally used in practical applications. It provides excess heat to mix the alkali activator and FA [18]. It also increases the workability and gives a good surface finish. The main constituents present in the blast furnace slag are SiO_2, CaO, MgO, Al_2O_3, and their concentrations are mentioned in Table 1 . It has been found that these compounds are common in most of the cementation substances.

Table 1. Chemical composition and their concentration in GGBFS

Compounds	Concentration
MgO	1% - 18%
Al_2O_3	8% - 24%
SiO_2	28% - 40%
Na_2O	30% - 50%

Water: For geopolymer preparation, drinkable water is found to be suitable. Hence, potable water has been used in this study to cure and mix.

2 Preparation of geopolymer composites

Specimens prepared by mixing only FA with alkali activator solution are called geopolymerpaste. The mixture is usually a homogeneous slurry and dark grey. When fine aggregate, i.e. sand is added to the mixture, the resulting product is termed as geopolymer mortar. Both geopolymer paste and mortar show a cohesive nature in the fresh state. Table 2 shows the materials required and their composition.

Table 2. Materials required & their composition

Materials	Composition
Activator Solution to Fly Ash (Ratio)	0.435 to 0.524 (Paste) & 0.445 to 0.475 (Mortar)
Alkali Content (Sodium Oxide)	5% to 8% by weight of Fly Ash
Silica Content (Silicon dioxide)	6.5% to 13% by weight of Fly Ash
Water to Fly Ash (Ratio)	0.32 (Paste) & 0.33 (Mortar)
Fly Ash to Sand (Ratio)	1.0

The above constituents were used in the manufacture of geopolymer composites, the mixing, and the curing procedure followed after Thakur and Ghosh [19].

The following steps describe the manufacturing processes of geopolymer composites.

1. Preparation of alkaline activator solution by mixing sodium hydroxide pellets, sodium silicate solution, and water in the required quantities to obtain desired alkali and silica content along with predetermined water to FA ratio. It should be prepared at least one day before its use for the manufacturing of geopolymer paste/mortars.
2. In a Hobart mixer, an activating solution and FA is mixed for 5 minutes to obtain a homogeneous slurry.

3. Introduction of fine aggregate (sand) gradually into the slurry and further followed by mixing in the Hobart mixer for another 5 minutes to get geopolymer mortar.
4. Transferring the geopolymer mix (paste or mortar) into steel molds of 50×50×50 mm cubes and vibrating it for 2-3 minutes to remove any entrapped air from the vibrating table.
5. Rest period of 60 minutes before heat curing.
6. Replacing the filled molds into an oven for heat curing at a temperature of 85°C for 48 hours.
7. Storing the geopolymer specimens at a dry place at ambient temperature until testing is to be done.
8. Various tests such as compressive testing, water absorption, etc.

The ratio of 0.32 of water to FA was maintained for the preparation of the paste and 0.33 for the mortar specimens. It was found to give sufficient workability after several trial mixes. Mortar and paste having Na_2O content varying from 5 - 8% were prepared as shown in Table 3 .

Table 3. Mix proportion of Geopolymer mortars & paste for studying the effect of Na_2O content

Geopolymer Mix Details				Geopolymer Mix Proportion				
ID	Na_2O % In Activator (a)*	% SiO_2 In Activator (b)*	Water / Fly Ash Ratio	Fly Ash (gm)	Sand (gm)	NaOH Pellets (gm)	Na_2SiO_3 Solution (gm)	Water Added (gm)
M1	5.0	6.5	0.33	1000	1000	39	245	161
M2	6.5	6.5	0.33	1000	1000	58	245	156
M3	8.0	6.5	0.33	1000	1000	78	245	152
P1	5.0	6.5	0.32	1000	—	39	245	151
P2	6.5	6.5	0.32	1000	—	58	245	146
P3	8.0	6.5	0.32	1000	—	78	245	142

(a)* and (b)* by weight of fly ash

2.1 Test conducted on the specimen

2.1.1 Study on Physical and Mechanical properties

The first part of the experimental investigation was to manufacture FA-based geopolymer composites of different compositions varying in alkali content (%Na_2O) of the activator solution. Specimens were prepared by activating dry FA with activator solutions, which were a mixture of NaOH solids, Na_2SiO_3 solutions, and water. For studying the effect of alkali content on the physical and mechanical properties of resulting geopolymer composites, Na_2Ocontent in the activator solution varied from 5% to 8.5% of the weight of FA. Variations in Na_2Ocontent were controlled by mixing required quantities of NaOH solids, water, and Na_2SiO_3solutions to make activator solutions. After 28 days from the manufacture, physical and mechanical properties such as water absorption and compressive tests were studied and inter-related.

2.1.2 Compressive strength

Relevant details related to the determination of the compressive strength of geopolymer paste and mortar are shown in Table 4.

Table 4. Details relevant to compressive strength

Age of Geopolymer at Test Date	28 Days
No. Of Specimen	03
Compression Testing Machine Capacity	2000 kN (Digital CTM)
Test Carried as per	ASTM C-109-02 [15]

2.1.3 Water absorption

It was measured first by completely drying the specimen to a constant mass, after that submerging the specimen in water, and then measuring the increase in weight as a percentage of dry mass. The procedure followed for the determination of water absorption of geopolymer specimens was following ASTM C-642 [20]. 28 days old specimens were dried at 105°C for 24 hours and immersed in water after measuring its weights. The specimens were taken out from submergence and surface water was wiped out after this it was weighed immediately in saturated surface dry (SSD) condition to find out the increment in the weight of the composite.

3 Results and Discussion

3.1 Effect of Sodium Oxide on compressive strength

Figure 3 depicts the effect of sodium oxide concentration on the 28 days compressive strength of the geopolymer composites. It is evident from the figure that the compressive strength increases with an increase in Na_2O content for both the geopolymer paste and mortars. For paste specimen P1 having Na_2O of 5%, the compressive strength measured is 19.74 MPa, while for specimen M1, the strength is 22.42 MPa. A significant increase in compressive strength is recorded when the Na_2O content is increased to 6.5%. P2 and M2 having a Na_2O concentration of 6.5% showed a compressive strength of 31.13 MPa and 37.84 MPa respectively, i.e., an increase of 57.70% for paste and 68.77% for mortar specimens. Details of compressive strength variation of geopolymer composites due to Na_2O content variation are shown in Figure 3**.**

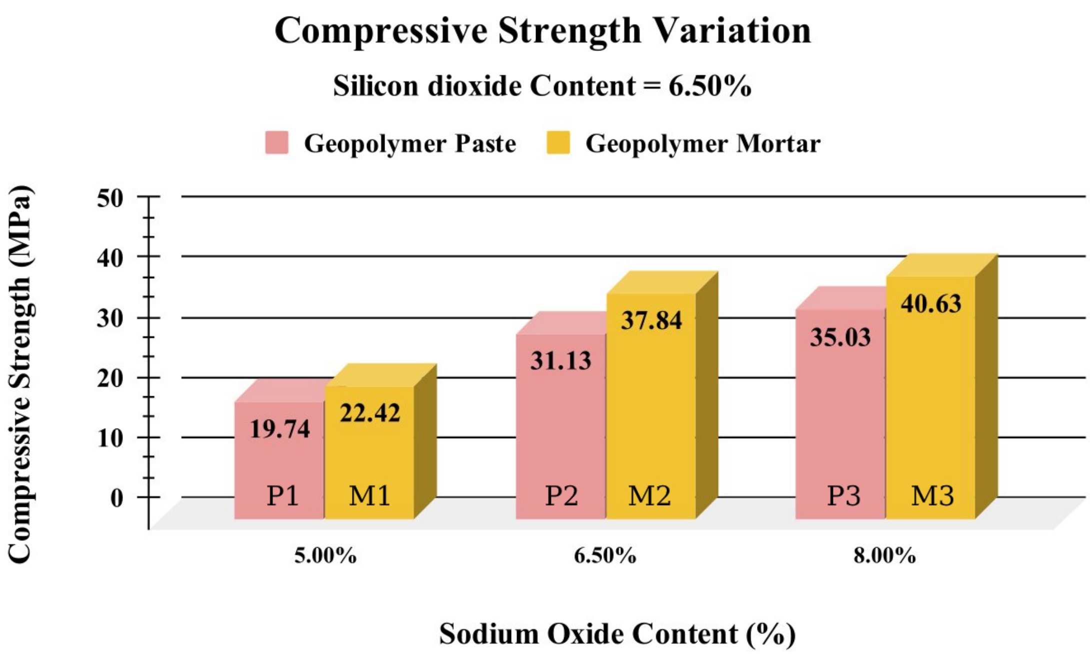

Fig 3. Compressive strength variation subjected to variation in Sodium Oxide content

However, as the Na_2O content is further increased up to 8%, an increase in compressive strength is found to be very little. P3 and M3 having 8% Na_2O have a compressive strength of 35.03 MPa and 40.63 MPa, which are only 12.52 % and 7.37% higher than the strength measured for paste and mortar specimens of 6.5% Na_2O. There is an increment in the compressive strength with the increment in sodium oxidecontent which is due to the higher reactivity of FA in a stronger alkaline solution. It can be noted here that a highly alkaline solution is needed for complete polymerization.

Hence, with a higher content of alkali in the activator solution, better dissolution of the FA takes place thereby forming more geopolymer gel accompanied by lesser pores, which result in higher compressive strength. But, the rate of increase in compressive strength is significantly higher in the lower ranges of Na_2O content also. Therefore, when Na_2O is increased from 5% to 6.5%, a remarkable rise in compressive strength has been observed. However, the change in strength increase is not significant when specimens having 6.5% Na_2O content is compared to specimens of 8% Na_2O content. This indicates that most of the dissolution of FA is possible even at 6.5% Na_2O which causes a highly improved microstructure than the specimen having 5% Na_2O. While improvements in the microstructure are noticed after increasing Na_2Ocontent from 6.5% to 8% and it was not significant. This is the reason for little improvement in compressive strength.

3.2 Effect of Na_2O content on Water absorption

Test Conducted on 28 days, the effect of Na_2O content on water absorption for geopolymer paste and mortar specimens is shown in Figure 4 . It can be noticed that water absorption goes on decreasing with increasing Na_2O content from 5% to 8%. For specimens P1 and M1, both having 5% Na_2O, water absorption values are found to be 22.85% and 10.97% respectively, which shows a remarkably high water absorption in paste specimens. P2 and M2 with higher Na_2O content of 6.5% yielded water absorption of 20.28% and 9.57%, respectively. Further, an increase in the Na_2O content up to 8% (P3 and M3) resulted in a further decrease in water absorption to 15.13% and 6.24%. In the case of paste specimens, water absorption was found relatively higher than the mortar counterparts for the same Na_2O content. It may be noted here that the porosity has been observed higher with lesser Na_2O content and this has caused greater water absorption in these specimens. Details of water absorption variation of geopolymer composite due to sodium oxide variation are shown in Figure 4**.**

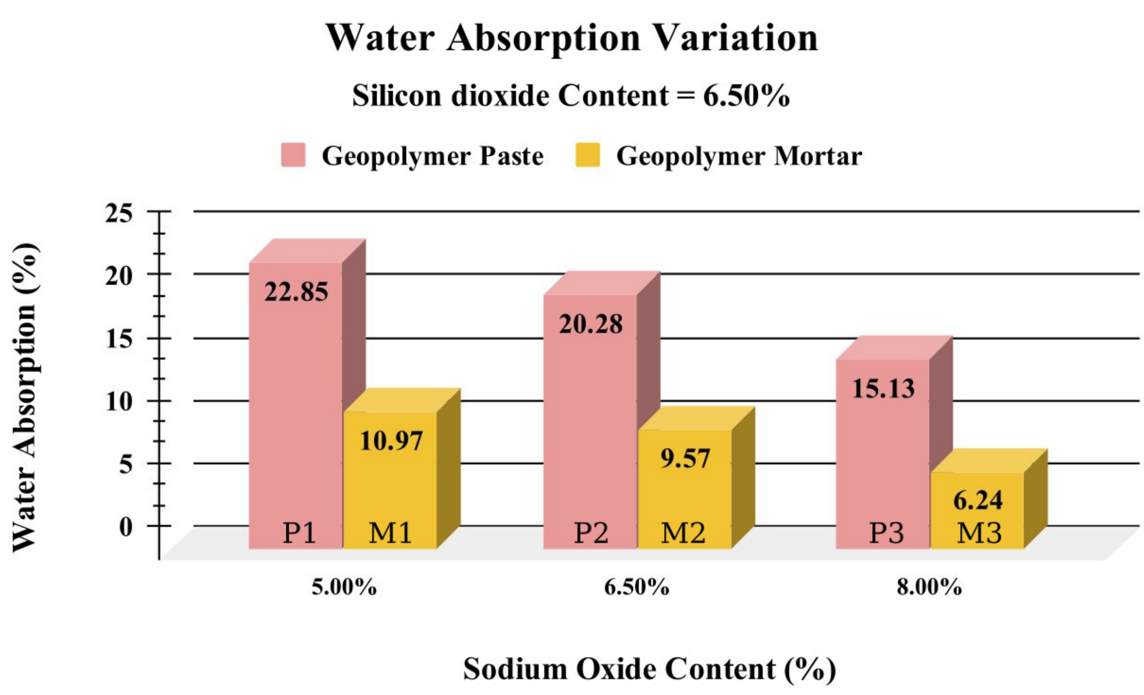

Fig 4. Water absorption variation subjected to variation in Sodium Oxide content

4 Conclusion

This Study presents the experimental investigations on the influence of sodium oxide content on the compressive strength and water absorption of geopolymer paste and mortar. Tests for compressive strength and water absorption were carried out on various samples of geopolymer paste and mortar specimens. The study concludes the following observations noticed during experiments.

1. The compressive strength of the geopolymer composites increases with a high concentration of NaOH in the alkaline liquid, however after a certain level of concentration it shows a negative effect.
2. Geopolymer mortar specimens are more porous and stronger in comparison to geopolymer paste
3. Water absorption was seen maximum at 5% of Na_2O content
4. The trend of water absorption was the opposite of the compressive strength trend
5. Maximum compressive strength can be achieved by using 8% Na_2O content

Future scope of the research

To understand the effect of NaOH concentration on the properties of geopolymers, more investigations are required, specifically on the microstructural part. Electron microscopy, X-ray diffraction analysis, and other advanced analytical techniques are encouraged to have a better understanding in the future.

Data Availability: All experimental data used for analysis is present within the manuscript.

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References

1) Mustakim SM. Improvement in Fresh, Mechanical and Microstructural Properties of Fly Ash- Blast Furnace Slag Based Geopolymer Concrete By Addition of Nano and Micro Silica. *Silicon*. 2020.
2) Das SK. Fresh, Strength and Microstructure Properties of Geopolymer Concrete Incorporating Lime and Silica Fume as Replacement of Fly Ash. *J Build Eng*. 2020;p. 101780–101780.
3) Das SK. Characterization and utilization of rice husk ash (RHA) in fly ash - Blast furnace slag based geopolymer concrete for sustainable future. *Mater Today Proc*. 2020.
4) Das SK, Singh SK, Mishra J, Mustakim SM. Effect of Rice Husk Ash and Silica Fume as Strength-Enhancing Materials on Properties of Modern Concrete-A Comprehensive Review. *Lecture Notes in Civil Engineering*. 2020.
5) Kuenzel C, Vandeperre LJ, Donatello S, Boccaccini AR, Cheeseman C. Ambient Temperature Drying Shrinkage and Cracking in Metakaolin-Based Geopolymers. *Journal of the American Ceramic Society*. 2012;95(10):3270–3277. Available from: https://dx.doi.org/10.1111/j.1551-2916.2012.05380.x. doi:10.1111/j.1551-2916.2012.05380.x.
6) Mishra J, Das SK, Krishna RS, Nanda B, Patro SK, Mustakim SM. Synthesis and characterization of a new class of geopolymer binder utilizing ferrochrome ash (FCA) for sustainable industrial waste management. *Materials Today: Proceedings*. 2020. Available from: https://dx.doi.org/10.1016/j.matpr.2020.02.832. doi:10.1016/j.matpr.2020.02.832.
7) Aliabdo AA, Elmoaty AEMA, Salem HA. Effect of water addition, plasticizer and alkaline solution constitution on fly ash based geopolymer concrete performance. *Construction and Building Materials*. 2016;121:694–703. Available from: https://dx.doi.org/10.1016/j.conbuildmat.2016.06.062. doi:10.1016/j.conbuildmat.2016.06.062.
8) Singh NB. 2018.
9) Rossi AD, Simão L, Ribeiro MJ, Hotza D, Moreira RFPM. Study of cure conditions effect on the properties of wood biomass fly ash geopolymers. *Journal of Materials Research and Technology*. 2020;9(4):7518–7528. Available from: https://dx.doi.org/10.1016/j.jmrt.2020.05.047. doi:10.1016/j.jmrt.2020.05.047.
10) Ranjbar N, Mehrauli M, Alengaram UJ, Metselaar HSC, Jumaat MZ. Compressive strength and microstructural analysis of fly ash/palm oil fuel ash-based geopolymer mortar under elevated temperatures. *Constr Build Mater*. 2014.
11) Bhushan JB. Suitability of Ambient-Cured Alccofine added Low-Calcium Fly Ash-based Geopolymer Concrete. *Indian J Sci Technol*. 2017.

12) Das SMMSK, Mishra J. An Overview of Current Research Trends in Geopolymer Concrete. *Int Res J Eng Technol*. 2018;05(11):376–381.
13) Panias D, Giannopoulou IP, Perraki T. Effect of synthesis parameters on the mechanical properties of fly ash-based geopolymers. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*. 2007;301(1-3):246–254. Available from: https://dx.doi.org/10.1016/j.colsurfa.2006.12.064. doi:10.1016/j.colsurfa.2006.12.064.
14) Wattimena OK, Antoni, Hardjito D. 2017.
15) P, Silva D, Sagoe-Crenstil K. The role of Al2O3, SiO2 and Na 2O on the amorphous → crystalline phase transformation in geopolymer systems. *J Aust Ceram Soc*. 2009.
16) 618 AC. Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use. *Annu B ASTM Stand*. 2010.
17) Coarse and fine aggregate for concrete - Specification. *Bur Indian Standards*. 2016;383.
18) Rangan BV. Geopolymer concrete for environmental protection. *Indian Concr J*. 2014.
19) Thakur RN, Ghosh S. Effect of mix composition on compressive strength and microstructure of fly ash-based geopolymer composites. *J Eng Appl Sci*. 2009.
20) Standard test method for density, absorption, and voids in hardened concrete. *ASTM C642-13*. 2013.