Influence of Al and Si Oxides in Geopolymer Nano-Silica Composite

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Abstract

Geopolymerization is a chemical process in Geopolymer concrete, which involves under the alkaline condition with Si and Al minerals, it brings in a 3D polymeric chain and ring structure brings of Si-O-Al-O-Si bonds. Geopolymer concrete is an innovative material that is characterized by long chains or networks of inorganic molecules. It is a potential alternative to conventional Portland cement concrete for use in construction. In this paper, the examination concerns the utilization of an ideal mix of pozzolanic material such as Ground Granulated Blast furnace Slag (GGBS) with an alkaline solution as the binder. Moreover, Metakaolin which is rich in silica and aluminum is added as an admixture to increase the strength of the Geopolymer concrete. In addition, Nano-silica is utilized to decrease the pores and boosts the strength of geopolymer concrete. The Chemical composition of the materials, mixing proportions and curing methods play an important role in the development of strength and influence the rate of polymerization. 30 mixes were outlined at different extents such as 10%, 20%, 30%, 40% and 50% of Metakaolin and 0.5% and 1% of Nano-silica at ambient curing. It is observed that the result of compressive strength at 30% of Metakaolin and 0.5% of Nano-silica gives high strength. At the same combination, the chemical analysis also revealed an increase in the strength of concrete with this level of Silica and Alumina content.

Keywords— *Geopolymerization, Chemical Analysis, Green Concrete, Sustainable Material, Environmental Friendly*

Introduction

Geopolymers have drawn the attention of the research world, as one of the most advanced and promising alternatives to traditional construction materials, largely due to its primary characteristics like superior strength and durability. Geopolymer technology in the near future is expected to take a huge leap in terms of sustainable use of the construction materials sector. Geopolymerization is considered to be the most feasible technology for the way forward in terms of the reduction of greenhouse gas emissions as well as for waste management solutions [1-2]. The geopolymer is obtained when the reaction takes place between a source of active Si-Al and the alkaline solutions resulting in the formation of Si-O-Al-O polymeric bonds, following which a geopolymer resin is rinsed with a unique filler to form the binder termed as geopolymer binder [3-4]. Geopolymers contains a large number of semi-crystalline to amorphous phases with Si-O-Al linkages forming a 3D inorganic polymer. It is well understood that polymerization is a process of collaborating multiple monomers and creating different sized and shaped macromolecules. Geopolymerization is the process that typically occurs in four different stages namely, destruction, coagulation, followed by condensation and crystallization. In the process of destruction, the bonds Si-O-Si, Al-O-Si and Al-O-Al that are present in the source material are broken which is termed as the dissolution of solid aluminosilicate. The leached products from the primary stage incorporated into the aqueous phase and collaborated to form a coagulated structure. During the stage of condensation, the zeolite precursors are formed resulting from the disintegration of source material's aluminosilicates in the alkaline medium.

In comparison to the conventional ordinary Portland cement (OPC), geopolymers have a vast number of advantages which include higher compressive strength, superior resistance to

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chemicals, heat, and fire, higher thermal resistivity, lower level of shrinkage, significantly lower greenhouse gas emissions, faster curing times and easy and accurate mould ability [5-6]. In one of his research works, Garcia et al., (2008) postulated through his experiments that geopolymer that was fabricated from calcined materials i.e. fly ash, meta-kaolinites and granulated blast furnace slags usually report superior strength compared to that of GPC from uncalcined materials, like stilbites, kaolinites, and albites, etc. This infers that the calcined materials result in a greater degree of Geopolymerization comparatively. For the synthesis of geopolymer, industrial wastes like fly ash, granulated blast furnace slag, zinc, and copper slag can also be used as an aluminosilicate source, as Silica (SiO_2) and Alumina (Al_2O_3) are the primary oxides in the processing of geopolymer. Due to its vast availability, Fly ash is widely considered as a crucial pozzolanic by-product material that is suitable for application in the construction sector worldwide. Additionally, it is mixed in particular compositions along with other calcined materials and result in forming binders with good properties. Hence, many geopolymer researchers investigated its physical properties, micro structural compositions, mechanical properties as well as durability aspects to a great extent [7].

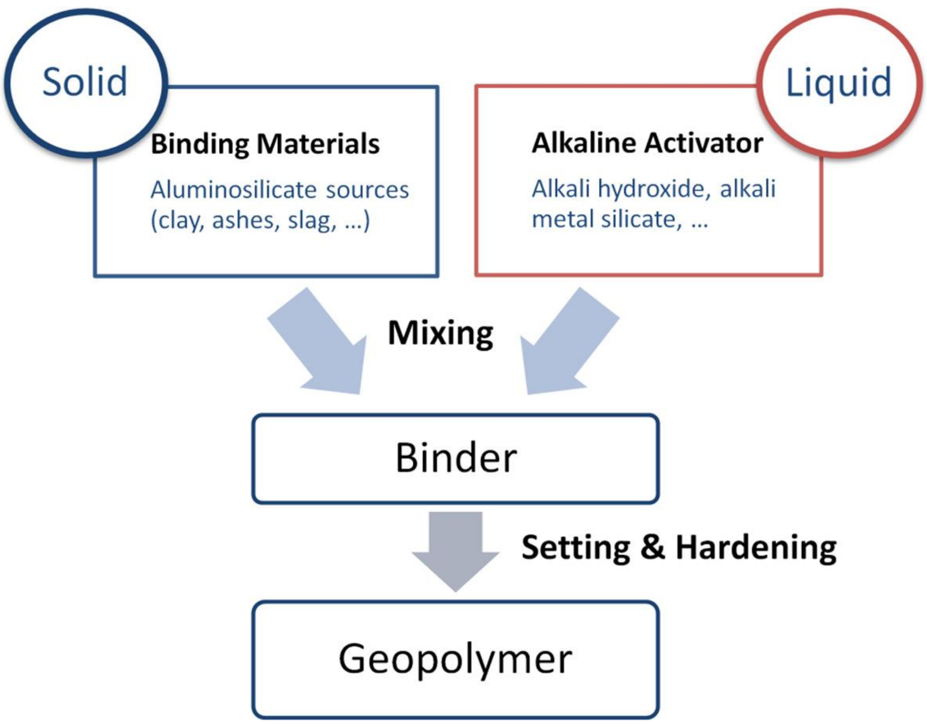

Figure 1: Geopolymer formation schematic (Source: L Y Ming et Al. (2016))

Most of the waste materials like fly ash, mine tailings, and furnace slag contain significant quantities of reactive silica and alumina which can, therefore, be used as source materials for in situ geopolymerization reactions. In earlier studies, many silica-alumina containing source materials including fly ash, building residues, pozzolan, furnace slag, and some pure minerals of Al-Si, and the clays like kaolinite and Metakaolinite have been analyzed. In his research, Joseph Davidovits (2008) used metakaolin and kaolin purely as a source of oxides of alumino-silicate for the production of geopolymers [8]. Metakaolin, as a structure is considered as a de-hydroxylated variant of the kaolinite clay mineral with 4-, 5-, and 6-coordinated Al ions in the

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polyhedron sheet structures of alumina. The advantages of Metakaolin as a valuable admixture include higher specific area, lower porosity, good ability to absorb and ability to form strong coordinative bonds when stimulated. Many researchers have also experimented on the production of geopolymeric products as well as their area of industrial applications by utilizing either metakaolin or kaolin as the primary reactant. Kaolin is widely considered to be the main structure-forming species during the total process of geopolymerization. However, the geopolymers that are manufactured using either kaolin or fly ash have framework structures that originate from the process of condensation of tetrahedral aluminosilicate units with different Al/Si ratios [9, 10].

As studied from the literature review, geopolymers exhibit different properties in compressive strength, shrinkage, resistance to chemicals, and thermal conductivity, depending on the raw material used. The metakaolin which is the aluminosilicate source is regarded as one of the most widely used raw materials mainly because of its higher reactivity, purity as well as conductivity. Regardless of the aluminosilicate source i.e., the alkaline solution is considered to play a key role in the geopolymerization reaction [11, 12]. In one of his works, A Gharzouni et al., (2015) investigated the effect of the alkaline activating solution where it was postulated that the molar ratio of Si/K molar ratio was found to control the type and degree of depolymerization of the silicate solution, and the amount of the siliceous species. Besides, due to the highly reactive nature of the depolymerized species, the oligomers are rapidly formed resulting in significantly superior mechanical properties for the geopolymers. It is also to be noted that during a geopolymerization reaction both the alkaline solution and aluminosilicate source are important parameters [13]. Of late, alkali-activated geopolymers have gained major attention largely due to their potential use in the construction sector as a key raw material, as well as their advantage of the formation of environmentally cementitious binders that replace the conventional ordinary Portland cement (OPC). Hongling Wang et al (2005) studied the mechanical strength properties of the geopolymers and their dependency on the concentration of alkaline solution i.e., NaOH solution. It is observed that compressive strength, apparent density and flexural strength of the geopolymer enhanced with the increase in the concentration of NaOH, which is as a result of the enhanced dissolution of particulates of Metakaolinite.

Duxson et al. (2007) proposed a model which is shown in Figure 2 for the geopolymerization mechanism. It consists of five different stages namely dissolution, speciation equilibrium, gelation, reorganization, leading to polymerization followed by hardening. He postulated that alkaline activator plays a significant role in the geopolymerization process and affects the mechanical properties of the geopolymer to a great extent. Although significant research works were carried out with different calcined materials and their effect on properties of the GPC mix, the compressive strength estimation and the effect of different compositions of Metakaolin and Nano-silica are very limited. Apart from that, the effect of Metal oxides which play a crucial rule in bonding behavior within the concrete mix has not been studied in detail. In the current study, the effect of different concentrations of Metakaolin and Nano-silica and their effect on the compressive strength of the GPC composite is investigated and validated with the chemical compositions of the samples under study.

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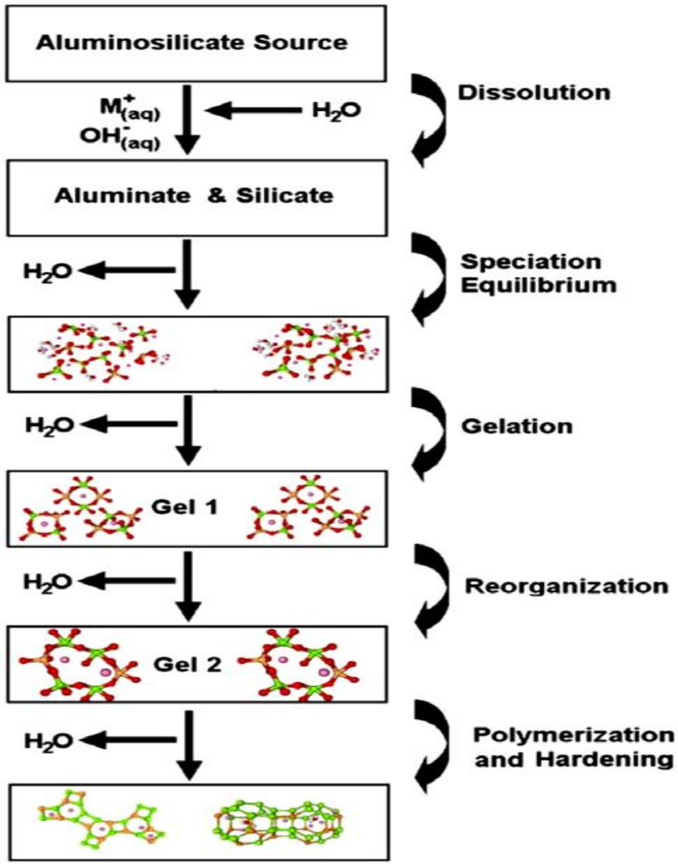

Figure 2: Geopolymerization – Concept (Source: P. Duxson et al. (2007))

Materials and Methods

A. Reagents

i. Ground Granulated Blast Furnace Slag (GGBS)

Ground Granulated blast furnace slag (GGBS) is a byproduct material obtained from the process of manufacturing of iron ore in the blast furnace. GGBS is one of the primary raw materials used to make a cementless binder. GGBS is effectively used as a mineral admixture in modifying the microstructure of geopolymer concrete and also the polymerization process.

GGBS is off-white in color and when mixed with water, can develop its unique hydraulic reaction. GGBS is considered as a key pozzolanic material as 90% of its composition includes the combination of silicates, alumina, and calcium. It also develops very good binding property with alkaline liquids to yield better strength and alkali activation process. GGBS was obtained from JSW Cements, Madurai and used in this research as the main constituent of binding materials in geopolymer concrete. GGBS is half white in color. GGBS has both pozzolanic and cementitious properties, also has greater alumina and silica content which helps to increase the strength of the concrete. The specific gravity of the GGBS is 2.84. The chemical composition of the GGBS is given in Table 1. X-ray fluorescence analysis is performed to understand the elemental composition of the material and presented in Figure 3.

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Table 1: GGBS – Chemical composition

SiO_2	5.806 %
CaO	72.435 %
MgO	3.416 %
Fe_2O_3	4.830 %
Al_2O_3	4.510 %
Na_2O	0.001 %
TiO_2	3.557 %
MnO_2	5.446 %

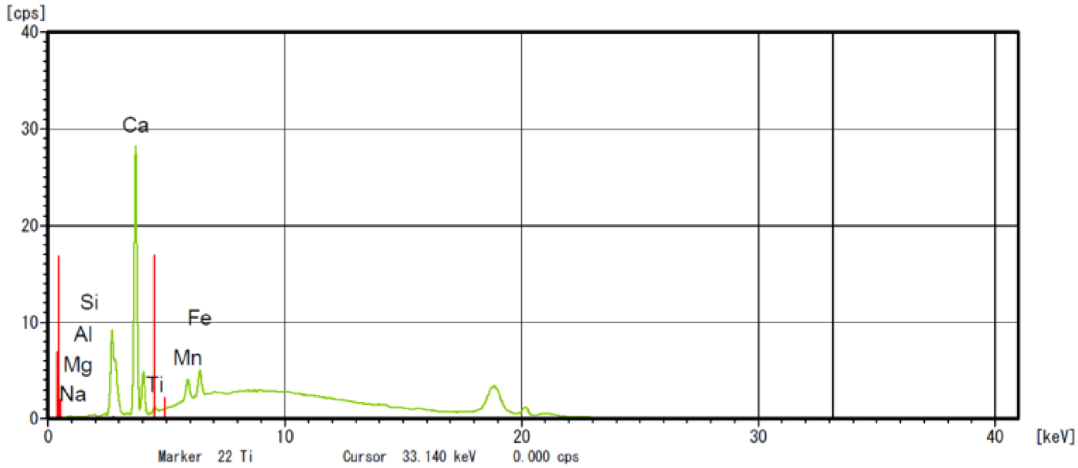

Figure 3: XRF analysis of GGBS

ii. Metakaolin

Metakaolin is a form of refined clay that undergoes the process of calcination under controlled conditions, resulting in the formation of an amorphous aluminosilicate which is reactive in concrete. Metakaolin (MK) is an industrial byproduct matter, is considered as a highly reactive pozzolanic admixture and possesses good potential in the manufacturing concrete-based composites. MK is generally manufactured from a natural geological mineral which generally contains a lot of impurities and inconsistent Si:Al atomic ratios. MK is very fine in physical appearance and is highly reactive which provides creamy and non-sticky and creamy texture for fresh concrete that makes finishing easier. Metakaolin is collected from Astra Chemicals, Chennai. It devours calcium hydroxide which reduces efflorescence and reacts with $Ca(OH)_2$ to form additional cementing components, the material that is mainly responsible for holding the concrete in-place and together. MK is a good source of aluminum (Al_2O_3) and silicate (SiO_2) and generates a greater degree of Geopolymerization as it is highly reactive with the alkaline activators. MK is considered to be an ideal material for geopolymer because it has superior amorphous content and consisting of fine particles which increase its compressive strength. The specific gravity of Metakaolin is 2.6. Chemical composition is given in Table 2 below. In an attempt to understand the elemental analysis and composition of Metakaolin, XRF analysis is performed and the results are interpreted in Figure 4 below.

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Table 2: Metakaolin – Chemical composition

SiO_2	29.312 %
CaO	0.671 %
MgO	10.266 %
Fe_2O_3	3.950 %
Al_2O_3	24.269 %
Na_2O	23.746 %
TiO_2	7.785 %
MnO_2	3.950 %

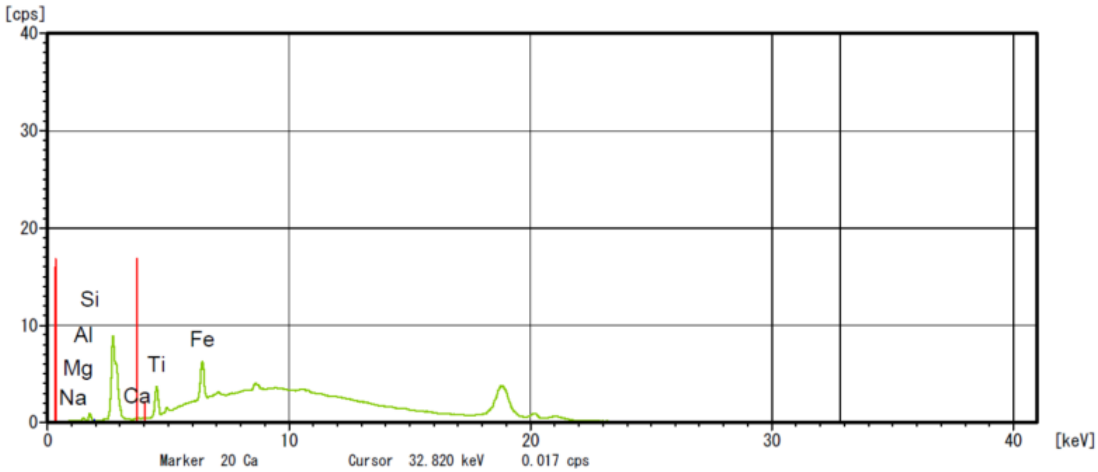

Figure 4: XRF analysis of Metakaolin

iii. Nano-silica (NS)

Nano-silica is the most generally utilized materials in concrete to enhance the performance largely because of its superior surface area, pozzolanic reactivity, as well as the pore-filling effect. Nano-silica addition to concrete results in higher strength. NS used was of particle form with 99.9% SiO_2 and a specific surface area of 201 m^2/g. It was produced by Astra Chemicals and was used as received. The specific gravity of Nano-silica is 2.4 and the particle size is 17 nm. The chemical composition of Nano-silica is given in Table 3 below.

Table 3: Nano-silica - Composition

SiO_2	48.532 %
CaO	6.618 %
MgO	3.847 %
Fe_2O_3	0.151 %
Al_2O_3	1.51 %
Na_2O	39.642 %
TiO_2	0.058 %

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The XRF analysis of the Nano-silica material is carried out and presented in Figure 5 below.

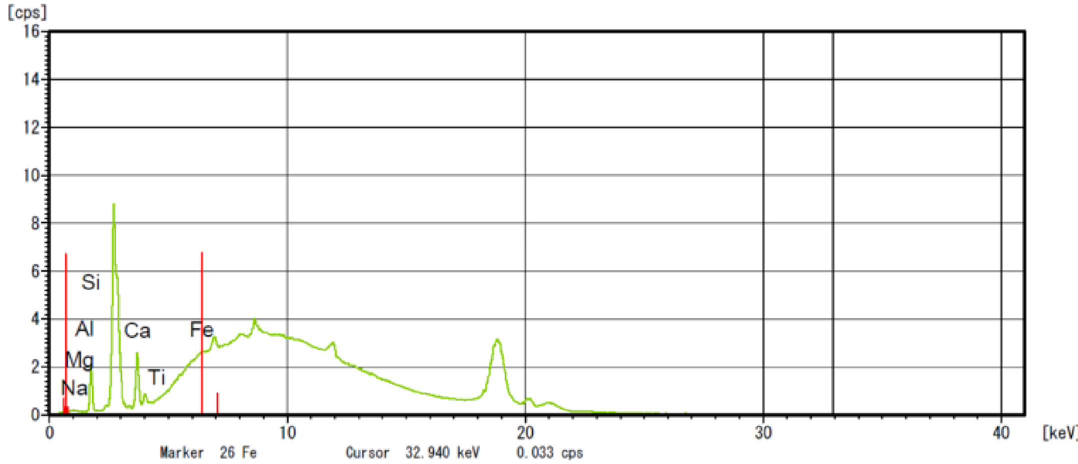

Figure 5: XRF analysis of Nano-Silica

iv. Alkaline Solutions

Commonly used alkaline solutions are sodium and potassium-based. In this study, the sodium-based liquid is used as an alkaline solution. In geopolymer materials, for the geopolymerization process, the primary contribution of ions of silicate is through the alkali activation. The sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are generally used as alkaline solutions in most cases. NaOH in the form of pellets with 97-98 % purity is used where the solid form of pellets are dissolved in water to obtain 12M concentration of the solution. For all the mixes, the ratio of Na_2SiO_3 to NaOH is kept at 2.5. The ratio of alkaline solution to GGBS is kept as 0.40. The mixture of Na_2SiO_3 and NaOH solution is blended together and kept aside for 24 hours for the polymerization process to occur. An enormous amount of heat is liberated during the reaction.

(a) GGBS

(b) Metakaolin

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(c) Nano-Silica

(d) Sodium Hydroxide

(e) Sodium Silicate

Figure 6: Materials used in the Geopolymer Mortar Mix

B. Preparation of materials

i. Mixing and casting Procedure

Figure 7: Hand Mixing of Geopolymer Mortar for M40

According to Lloyd and Rangan's mix design methodologies, the density of GPC is assumed as 2080 kg/m^3. The ratio of the binder to the sand content was kept at 1:3. Consequently, GGBS

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content was determined based on the activator solution to the GGBS ratio. In this work, AAS to GGBS ratio was taken as 0.40. Further, the ratio of Na_2SiO_3 to NaOH was assumed as 2.5 and kept constant for all the mixes. The binder GGBS, MK, and NS are mixed well in dry conditions. Later, the alkali activator solution is added into the mixture slowly. Then, water is added to increase the workability of the mortar. And finally, the superplasticizer of 1.5% of the binder is added to the mix. Mix the mortar for 5 – 7 minutes as shown in Figure 7. Finally, the mortar was cast in 70.6 mm Mortar Cube moulds and then poured in three different phases with accurate compaction as shown in Figure 8.

Figure 8: Casting of Geopolymer Mortar

ii. Mix Proportions

Metakaolin was added in 10%, 20%, 30% and 40% by the weight of the binder and the samples are labeled as M1, M2, M3, and M4 respectively. In addition, nano-silica was also added in 0.5%, and 1% by the weight of the binder to the 30% Metakaolin sample and labeled as M5 and M6 respectively.

iii. Curing Regime

It is generally understood that the curing time and curing method influences the strength of the GPC. Immediately after casting, the specimens are allowed to cure after 24 hours under ambient curing shown in Figure (9). The rate of increase in strength was rapid up to 24 hours of curing time and the strength increases accordingly from 3, 7, 14 to 28 days. In 28 days it attains the strength of M40 grade concrete.

Figure 9: Mortar Cubes after Casting

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C. Experimental Studies

To measure the compressive strength, the compressive testing machine is used and the test is performed in accordance with ASTM: C109 standard as shown in Figure 10. The compressive strength is carried out on the samples up to 28 days and is measured by taking an average of 3 specimens for each sample. For each sample, the compressive strength is calculated as the ratio of maximum load taken by the specimen and the cross-sectional area.

(a) *(b)*

Figure 10 (a), (b): Testing of Mortar Cubes

Results and Discussion

D. Compressive Strength

The compressive strength of cured GPC mortar is the key indicator to assess the performance of source materials since it gives a good description concerning the quality of geopolymerization products. Six different GPC mortar samples are prepared with compositions M1 (10% MK), M2 (20% MK), M3 (30% MK), M4 (40% MK), M5 (30% MK, 0.5% NS), M6 (30% MK, 1% NS), where MK indicates Metakaolin and NS indicates Nano-silica. The Mortar Cubes after Testing is shown in Figure 11.

The development of compressive strength after 28 days from sample preparation for all the 6 different samples are tabulated and represented in Table 4 and Figures 12 (a) and (b). It can be observed that with the increase in the loading concentration of Metakaolin, the compressive strength also increased monotonically. As observed in literature as well, the highly reactive nature of Metakaolin towards alkali activators as well as its higher amorphous phase collaborated by a good degree of geopolymerization due to the presence of good source materials in silica and alumina gives better strength properties. It can also be observed from Figure 12 (a) that, 30% Metakaolin loaded GPC Mortar is found to be the optimum concentration beyond which the addition of MK had a negative effect on the compressive strength.

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Figure 11: Mortar Cubes after Testing

Table 4: Compressive strength of GPC Mortar mix samples with MK and NS

Sample composition of GPC Mortar	**Compressive strength (N/mm^2)**
M1 (10% MK)	41.94
M2 (20% MK)	47.91
M3 (30% MK)	52.5
M4 (40% MK)	45.69
M5 (30% MK, 0.5% NS)	56.95
M6 (30% MK, 1% NS)	55.49

As it is proved that 30% MK loaded GPC Mortar mix is the optimum concentration level which gives the best compressive strength properties, this is taken as a reference for further study. Different concentrations of Nano-silica (0.5% and 1%) are added to 30% MK GPC Mortar samples to study the effect of Nano-silica on the strength properties. It is observed from Figure 12 (b) that the 0.5% concentration of Nano-silica addition gave superior properties compared to 1% concentration of Nano-silica addition in the 30% MK loaded GPC Mix mortar. This further increase in compressive strength is attributed to the superior surface area for better reactivity and interlocking of bonds, and pole filling ability of the nano materials. Further addition of Nano-silica has a negative effect on the sample.

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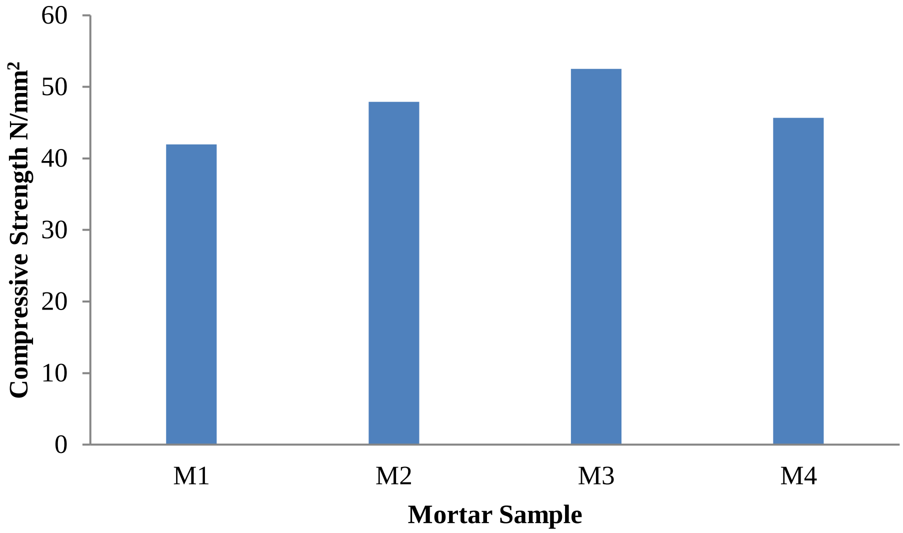

Figure 12 (a): Compressive strength of 4 different mix proportions of Metakaolin GPC Mortar samples (10%, 20%, 30%, 40%)

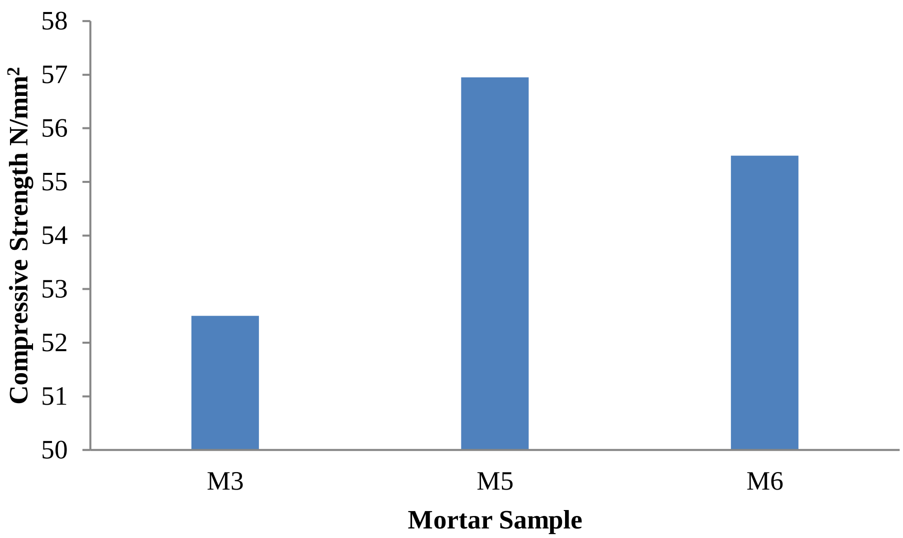

Figure 12 (b): Compressive strength of different mix proportions of GPC Mortar samples with and without Nano-silica for 30% Metakaolin GPC mix

E. Analysis of Chemical Composition on development of Strength

Generally, the rate of polymerization is affected by parameters which include curing temperature, moisture absorption, alkali concentration, Silica to alumina ratio, pH, and solid content as well as the type of activators that were used. It is understood from previous works that the comparisons of the compositions of major oxides present in the samples give a basic indication of the performance or properties of the sample. The chemical compositions of oxides of all the 6 different GPC Mortar mix samples are tabulated in Table 5 below. It is observed that M3 samples i.e., 30% MK loaded GPC mix has a superior composition of silica, alumina as well

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as the key parameter SiO_2/Al_2O_3 ratio among M1, M2, M3 and M4 which is in accordance with the compressive strength data presented in Figure 12 (a).

Table 5: Chemical oxide compositions of all mix proportions

Chemical Components	Mortar Designations					
	M1	**M2**	**M3**	**M4**	**M5**	**M6**
CaO	28.258	20.956	15.536	26.065	13.855	23.476
SiO_2	5.087	5.525	11.569	6.767	11.785	10.439
Al_2O_3	2.281	2.928	4.608	3.862	4.65	4.269
MgO	3.372	3.173	2.917	3.913	3.192	3.37
Fe_2O_3	2.631	2.687	2.574	2.412	2.074	2.382
Na_2O	0.48	0.24	0.22	0.3	0.2	0.29
K_2O	3.099	3.678	1.243	2.451	1.008	1.142
SiO_2/ Al_2O_3	2.23	1.89	2.51	1.75	2.53	2.45

Similarly, it can be seen that Silica to Alumina ratio is observed to be 2.53 for M5 which is loaded with 0.5% Nano-silica compared to the value of 2.51 for a similar sample without nano-silica ie., 30% MK GPC mix. It indicates the effect of nano-silica where a higher concentration of silica participated in the reaction enhancing the strength which can be observed in Figure 12 (b) as well. It is to note that Calcium oxide also plays a critical role in the polymerization process in controlling the strength as well as soundness. Excess CaO as seen in the M1 sample (28.25%) makes the sample unsound and causes expansion as well as disintegration. Hence M1, M4 samples which have excess CaO contents (28.25% and 26.06% respectively) are observed to be having lesser compressive strength comparatively. Similarly higher concentrations of K_2O and Na_2O lead to failure of concrete structures as they are considered to be harmful ingredients that are present in cement. It can be validated from Table 5 that M1 and M2 possess higher values of Na_2O and K_2O respectively and coincidentally they are having lesser strength values. Also, among other materials, MgO is expected to produce cracks within the concrete resulted from the disintegration of bonds within the structure. Hence, the M4 sample which has the highest MgO composition is found to be having a very less compressive strength of all other samples at a value of 45.69 N/mm^2.

Conclusion

The advantages of geopolymers being superior strength properties as well as their reduced effect on the environment resulting from the reduction of greenhouse gas emissions. The key raw materials of the geopolymer mix which include GGBS, Metakaolin and Nano-silica are added to alkali activator solution and GPC Mortar samples were fabricated along with the addition of minor amounts of plasticizer. To understand the effect of Metakaolin and nano-silica concentration on the mechanical properties and performance of the GPC mix, different concentrations of MK and NS were prepared and tested. It is observed that with the increase in the concentration of Metakaolin, the compressive strength of the GPC mix increased to 30% loading beyond which it had a negative effect. The increase is attributed to the highly reactive nature of Metakaolin towards alkali activators as well as its higher amorphous state, collaborated by a good degree of geopolymerization. Geopolymerization is directly related to the presence of silica and alumina in the mix and is validated by the XRF analysis and chemical composition details provided. The sample mix which has higher Silica to Alumina ratio has higher strength and coincidentally the same sample has lower values of Na_2O, K_2O, and MgO indicating that mix concrete didn't have any cracks. The optimum loading of 30% MK GPC mix is added with 0.5% and 1% Nano-silica to understand the effect of NS on the strength of the GPC mix. It is observed that the GPC mix containing 30% MK and 0.5% NS has superior compressive strength compared to that of a 1% NS mixed sample. An increase in the strength is attributed to the superior surface area of NS which provides more reactive sites for better bonding and also their ability to fill up pores assists the strength properties.

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