

A
DISSERTATION REPORT
ON
**STRENGTH AND DURABILITY STUDIES OF ALKALI-
ACTIVATED FLY ASH BASED GEOPOLYMER MORTAR**

Submitted in partial fulfillment of the requirement for the degree of

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In

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Submitted by

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CERTIFICATE

This is to certify that the dissertation entitled “**Strength and Durability Studies of Alkali-Activated Fly Ash Based Geopolymer Mortar**” which is being submitted by **Suman Choudhary**, in partial fulfillment for the award of the degree of Master of Technology in the Civil Engineering Department (Disaster Assessment and Mitigation), MNIT Jaipur, is a bonafide work carried out by her under my guidance and supervision.

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ABSTRACT

The objective of present study is to investigate the performance of alkali-activated fly ash based geopolymer mortar. For this purpose, two fold experimental program was conducted. Firstly, the influence of various parameters mix composition and process parameter on the compressive strength of the geopolymer mortar was examined. The mix composition parameters included Sodium hydroxide concentration and aggregate to binder ratio, while process parameter included curing temperature. The compressive strength tests were also accompanied with advanced analytical techniques such as X-ray diffractometry (XRD) and Scanning Electron Microscopy (SEM). Secondly, the performance of the geopolymer mortar against the carbonation was also evaluated. The influence of aggregate to binder ratio and curing temperature on the carbonation of the mortar was investigated. Carbonation is determined in terms of carbonation depth. Further, the carbonation tests were also supported by XRD and SEM testing. It was found the compressive strength test of the geopolymer mortar increase with increase in concentration of NaOH in range of 8M to 14M. Aggregate to binder ratio of 2:1 was found to be optimum for the attaining maximum compressive strength at 28 days. The higher strength attained can be attributed to mineral formed such as zeolite and sodalite, as evident form XRD tests. Further, mortar having higher porosity was found to be more vulnerable to carbonation. The carbonation of the mortar can be due to change in pore solution chemistry which is evident from XRD tests. Hence, an attempt has been made to delve deeper into the understanding of the performance of geopolymer mortar. However, the long term performance of the geopolymer mortar subjected to carbonation could not be performed due to limitation of time, which warrants the further investigation and is beyond the scope of the present study.

Keywords: Geopolymer; mortar; compressive strength; carbonation; micro-structure.

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CHAPTER-1

INTRODUCTION

1.1 General

History of the mankind had witnessed the continuous improvement and development in the construction practices. Over the last century, it was experienced that expenses involved in preparation of mortar and concrete represents the significant portion of total cost of the project. Driven by commercial and industrial demand to economize the construction process, paradigm shift was noticed in the technological advancement for preparation of mortar and concrete. The unavailability of the various important constituents of mortar and concrete such as lime made the many construction projects economically unviable. Hence, various researchers explored the replacement of ordinary Portland cement concrete and mortar by geopolymer concrete and mortar. This study focuses on the various aspects related to geopolymer mortar.

1.2 Conventionally Used Mortar in the Construction

Conventionally used mortar consists of binder material cement, fine aggregate and water. It is mostly used in bridging the gap between masonry or brick blocks. The mortar can be made up of asphalt, cement and mud.

1.3 Need and Advantages of use of Geopolymer Mortar

Ordinary Portland cement and ordinary mortar includes the use of cement as their one of the important components. The manufacturing of cement involves the heating of lime stone at 1450° C in the kiln, which is referred as calcinations process. This process produces the carbon dioxide as a byproduct, while converting calcium carbonate in to calcium oxide. In parallel, coal utilized in heating process in the kiln also makes significant contribution to carbon dioxide emission. Various studies reveal that approximately 1 ton of carbon dioxide is emitted with production of 1 ton cement (*Bosoga et al. 2009*).

According to the literature, entire construction industry in the world requires 2.6 billion Tons of Cement every year. Further, the increase in the demand of cement in upcoming 10 years is estimated about 25% of the present day. As lime stone is the fundamental material for production

of cement, in the wake of growing global demand, natural reserves of lime stone may face probable shortage in next 25 years. (*Bosoga et al. 2009*).

Hence, it can be seen that use of cement poses many challenges to the nature, environment and atmosphere. They have potential to disrupt the eco-system of the planet. International organizations such as United Nations also pushing reforms on policies regarding climate change by placing UNFCCC (United Nations Framework Convention on Climate Change). The COP conference held in Paris is seen as watershed moment in policy regarding climate change, which intends to make it legally binding to reduce carbon footprint of the signatory countries. Hence, it has been imperative and unavoidable for policy makers of country to pay heed to the environmental challenges posed by cement production. It is vital to explore the alternative methodology to replace the monopoly of cement as a binder material in construction industry.

The use of geopolymer has come as a best solution for solving the environmental challenges and reducing carbon footprint of the country. In one hand, geopolymer helps utilizing waste material generated from thermal industry such as fly ash and blast furnace slag and on the other hand, it also replaces use of cement in the preparation of mortar as a binder material.

1.4 Types of Geopolymer and Their Applications

1.4.1 Types of Geopolymer

The geopolymer is defined as solid and stable aluminosilicate matrix formed due to alkali activation of Al/Si sources such as Fly Ash, rice husk ash and Ground granulated blast furnace slag. Types of geopolymer can be varied by using different constituents such as types of fly ash, types of blast furnace slag and various concentrations and ratio of alkali hydroxide and alkali silicates.

Another criterion for classifying the geopolymer is based on types of bonds between various molecules present in the structure of aluminosilicate gel. The structure of alkali aluminosilicate gel is reported to exist as a framework in which molecules of aluminum and silicon are connected in a three-dimensional tetrahedral gel framework (*MacKenzie 2003 and Rees et al. 2007*). These bonds between silicon, oxygen and aluminum are named by various researchers. *Davidovits (1982)* used the name 'sialate' nomenclature to depict the aluminosilicate structures. The bond type (Si-O-Al) was described as a sialate bond, and linkage (Si-O-Si) as a siloxo bond.

Further, this could be used to classify the geopolymers according to the values of Si/Al ratio such as for 1.0 a poly (sialate), 2.0 a poly (sialate-siloxo), and 3.0 a poly (sialate-disiloxo). However, a universal pictorial representation of the aluminosilicate is not agreed upon by researchers.

1.4.2 Applications of Geopolymer

Geopolymer is a binder material which upon adding with fine aggregate attains high compressive strength and good thermal properties within a short span of time. This provides the immense opportunities for their application as construction materials. Given its vast range of engineering properties, it can be used in various applications which are as follows.

a) Rapid construction: Given the shorter duration of curing, it can be used in where rapid construction is required. However, geopolymer requires the heating, which restricts the broader range of constructions where heating of structure is not viable option.

b) Pre-cast structures: As geopolymer mortar sets very quickly when subjected to heating, it opens the opportunity of its usage in precast construct industries. The geopolymer mortar can be used to fabricate panels by attaching the small block units such as bricks, masonry and concrete block. It can also be used to construct of precast wall-panels, joining brick blocks etc.

c) Isolation of low level and intermediate level nuclear waste: Various properties of geopolymers such as flash-set and set-inhibition; radiolytic hydrogen formation, fire resistance, freeze-thaw behavior provides an edge over conventionally used concrete. However, it was found that aluminosilicate gel has pore water within its pores, which makes the process more complex at the onset of radioactive activity. Hence, more research is needed in order to realize full potential of the geopolymer in isolation or immobilization of low level or intermediate level nuclear waste.

d) Isolation of toxic and hazardous waste: Several hazardous waste are leached out of from man-made activity such as Pb and Cr from mining and Cs and Sr from radioactive waste. These hazardous wastes can be contained with help of geopolymers. Geo chemistry of geopolymer is reported to be ideal for containment of low-charged cations. However, highly charged cations are found to be difficult to contain using geopolymers matrix. However, it is reported that by tailoring the process of preparation of binder material, varying degree of effectiveness can be achieved for geopolymers.

1.5 Various Mechanisms Involved in formation of Geopolymer Mortar

Geopolymer derives its strengths and several properties by polymerization process. The polymerization takes place from one amorphous material (Fly ash/ metakaolin and/or slag) to another amorphous material (geopolymer gel). For proper understanding of the behavior of geopolymer, it is important to understand the various mechanisms involved in the polymerization process.

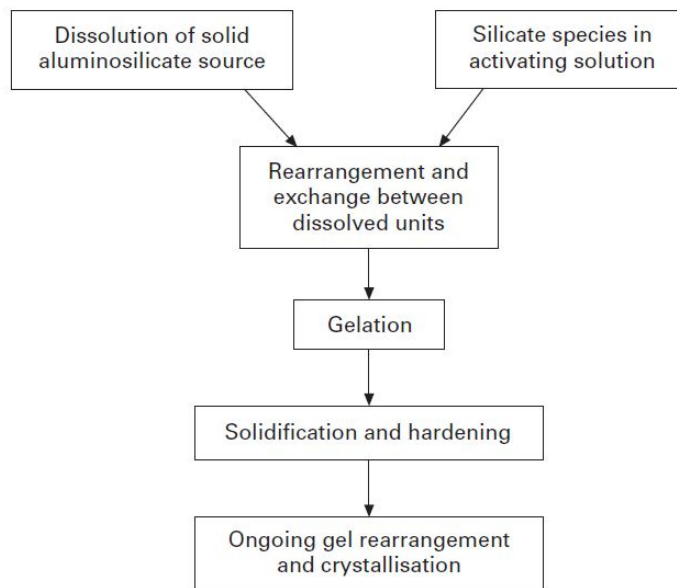


Figure 1.1: Schematic diagram of various steps of Geopolymerization

1.6 Aim and objectives of the Study

The main aim of the present study is to understand and investigate the performance of geopolymer mortar. Further, the performance of the geopolymer mortar subjected to carbonation also deserves through investigation. For this purpose, following objectives were fixed for the study, which are as follows.

- i) To understand the possible mix composition of the geopolymer mortar
- ii) To study the influence of the mix composition on the mechanical strength of geopolymer mortar
- iii) To study the influence of the mix composition on the micro structure of mortar

- iv) To understand the influence of the mix composition parameters on the carbonation of geopolymer mortar

1.7 Methodology Adopted in the Present Study

In order to achieve above stated aim and objectives, extensive literature on the geopolymer mortar was surveyed. First of all, Literature concerning application of geopolymer mortar and its mix design was referred. Further, influence of mix composition on the performance of geopolymer, in particular on the fly ash based geopolymer was focused. Furthermore, influence of the climate change in form of attack of CO₂ on the performance of the geopolymer was investigated. In conclusion, in the present study an attempt has been made to understand the mechanical behavior and microstructure of the fly ash based geopolymer mortar with and without carbonation.

1.8 Organization of the report

This study reports the findings of the various studies on the performance of the geopolymer mortar. For this purpose, whole report is partitioned in various chapters. First chapter present the introduction of the geopolymer mortar. Various sections of the first chapter includes various aspects of the geopolymer mortar such as need and advantages of the geopolymer mortar, types of geopolymer mortar and their applications and various mechanism involved in the preparation of the geopolymer mortar.

Second chapter includes the literature review on the performance of geopolymer mortar. The sections of the second reports the process involved in manufacturing of geopolymer mortar, and mix design proportion used by previous researchers and their effect on the performance of mortar. Furthermore, influence of carbonation on the performance and its microstructure of the geopolymer mortar are also presented.

In third chapter, properties of various materials used in study are presented. In fourth chapter, complete experimental program is reported. The experimental program is followed by results and discussion. The experimental program includes, compression testing on the geopolymer mortar, carbonation testing and advanced analytical techniques such as SEM and XRD.

Finally, in last chapter, conclusions based on various aspects focused in the study are summarized.

CHAPTER-2

REVIEW OF LITERATURE

2.1 General

Manufacturing of geopolymer is complex process with combined application of various engineering knowledge. The study of geopolymer should include all the aspects of manufacturing processes involving different engineering background. In order to study contribution of individual component on the performance of geopolymer, it is imperative to focus on influence of each component on the entire product as a whole. Hence, extensive literature review was done in order to delve deeper in to the performance of geopolymer. For this purpose, several research book, article, notes and state-of-art were referred. Researchers adopted several methodologies to understand the behavior of the geopolymer, they are i) theoretical or analytical studies, ii) experimental studies and iii) laboratory scale testing. This chapter reports research conducted by many researchers on manufacturing of geopolymer, mix design of the geopolymer mortar, influence of mix composition on the performance of the geopolymer mortar and its microstructure. Further, the durability of the geopolymer mortar under carbonation and its effect on the microstructure was studied.

2.2 Manufacturing of the geopolymer

The geopolymer consists of two important parts i.e. a) granular Al/Si source and b) Alkali activator. Combination of these two components is used as a binding material in the manufacturing of geopolymer mortar or concrete in place of ordinary Portland cement. The source of Si/Al used in preparation of geopolymer consists both pozzolanic material and hydraulic materials. The pozzolanic material possesses very little or no cementing properties, while hydraulic material demonstrates little binding properties upon interacting with water. Table 2.1 summarizes various types of Al/Si source used by various researchers. It can be noticed that selected material as a Si/Al source encompasses various types of by-products of industry (fly ash, GGBS and tungsten mine waste) or the agricultural waste (rice husk ash and palm oil fuel ash). Use of waste material as an important component in manufacturing of geopolymer will help in reducing carbon foot print of the country as an additional advantage.

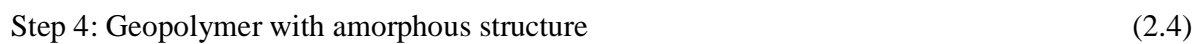
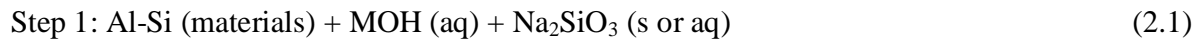
Table 2.1: Summary of various raw materials used in preparation of geopolymer in recent times

S. N. of component	Functionality	Raw materials	Researchers
1	Alkaline activator	NaOH + Na ₂ SiO ₃	Duxson and Provis (2007);
2	Al/Si source	Fly Ash	Rattanasak and Chindapasirt (2009); Temuujin et al. (2010)
		Palm Oil fuel Ash	Islam et al. (2014)
		Rice Husk Ash	Nuruddin et al. (2008) and Nuruddin et al. (2011)
		GGBS (<i>Ground Granulated Blast Furnace Slag</i>)	Islam et al. (2014)
		Calcined clay (<i>Metakaolin</i>)	Rovnaník P. (2010)
		Tungsten mine waste	Torgal et al. (2008)

As the present study is focused on fly ash based geopolymer, investigation on type of fly ash used in the manufacturing of geopolymer also important aspect. The fly ash is by product of the coal based thermal power plants. Fly ash is classified based on the carbon content, which depends the type of coal such as anthracite, bituminous, lignite and peat. ASTM C-618 provides the guidelines for characterization of fly ash. Preparation of geopolymer necessitates the specific type of fly ash which contains high proportion of silica and low content of calcium. High proportion of silica (SiO₂), alumina (Al₂O₃) and iron (Fe₂O₃) is necessary for pozzolanic reaction. Hence, Class “F” fly ash (ASTM C-618) is suitable for preparation of geopolymer.

Similarly, Table 2.1 also reports the various alkaline activators used in preparation of geopolymer. Alkaline activator is mixer of metal hydroxide and sodium silicate. In this mix, strong base is used such as sodium hydroxide or potassium hydroxide. However, due to economic consideration, sodium hydroxide is preferred in place of potassium hydroxide.

In the present study, fly ash based geopolymer is focused. It is vital to understand the process involved in the manufacturing of the fly ash based geopolymer. At the onset of mixing the alkaline solution with the fly ash powder, chemical reactions take place. The formation of cementitious product because of mixing of Si/Al source and alkaline solution is called as geopolymerisation. *Duxson et al. (2007)* studied the geopolymerisation and reported the mechanism involved in the formation of geopolymer. *Duxson et al. (2007)* reported various steps of the geopolymerisation, they are i) dissolution, ii) speciation equilibrium, iii) gelation, iv) reorganization and v) polymerization and hardening. Initial attack of alkali on the fly ash leads to dissolution of fly ash particles at their surface. Upon dissolution of fly ash, reaction products are created which either remain in isolation or stick to the surface of fly ash particles. The whole process creates the complex morphologies which exist simultaneously in the matrix of mortar, they are i) unreacted particles, ii) particles attacked by alkaline solution but incompletely dissolved, iii) reaction products etc. These processes can also be explained with help of eq 2.1 to eq. 2.4. It can be noticed that formation of $[M_a (AlO_2)_a(SiO_2)_b.nMOH.mH_2O].gel]$ depends upon the dissolution of Si/Al source material, which in turn is governed by quality of Si/Al source, concentration of alkaline solution *Xu(2002)*.



2.3 Mix design for preparation of geopolymer mortar

Geopolymer mortar is prepared by mixing alkali-activated fly ash (geopolymer binder) and fine sand (fine aggregate) with little amount of water. Well established guidelines are not present for mix design for preparation of geopolymer mortar. In recent years, researchers conducted experimental studies in order to establish the guidelines. Hence, performance based comparative studies on various parameters of geopolymer mix design using experimental program can be referred.

Mix design of geopolymer requires knowledge of two types of parameters; they are a) composition parameters and b) processing parameters. The composition parameters are the arithmetic relations between constituents or the properties of specific constituents present within the final geopolymer paste. The processing parameters depend on the environmental condition or methodology adopted for preparation of final product from the geopolymer mortar. Table 2.1 reports various types of parameters and their typical values adopted by various researchers. The composition parameters includes several arithmetic ratio such as alkali content ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$), silica content ($\text{SiO}_2/\text{Al}_2\text{O}_3$), $\text{Na}_2\text{SiO}_3/\text{NaOH}$, NaOH concentration, Alkali activator-FA ratio, water-geopolymer solid ratio, sand-fly ash (FA) ratio and sand-binder ratio. Similarly, processing parameters are curing time, curing temperature, rest period and type of mixing.

Adoption of typical values of different ratios depends upon the area of application of geopolymer mortar. Values of these ratios may vary from case study to case study. However, in general application, the literature provides guidelines to assume typical values of various parameters.

It can be seen that alkali content which is referred as ratio of Na_2O to Al_2O_3 ($\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$) is varied from 0.46 to 0.62 as explained by *Thakur and Ghosh (2009)*. Similarly, *Thakur and Ghosh (2009)* varied other parameters such as silica content (3.7 to 4.3), alkali activator to FA ratio (0.4 to 0.6), water to geopolymer solid ratio (0.157 to 0.366) and sand-FA ratio (0.5 to 3.0). It was attempted to study the influence of these parameters on performance of geopolymer mortar. *Rattanasak and Chindaprasirt (2009)* examined the influence of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ and concentration of NaOH on the performance of geopolymer. Range of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ and concentration of NaOH was adopted 0.5 to 2.0 and 5 to 15 respectively. *Temuujiin et al. (2010)* studied the effect of sand-binder ratio on the engineering properties of geopolymer mortar. Sand-binder ratio was varied from 10% to 50% in the study and influence of increase in sand content on the compressive strength was presented.

Further, processing parameters were studied found to be important in the mix design of geopolymer mortar and its performance. Final compressive strength and durability is governed by curing time, curing temperature and type of mixing (*Thakur and Ghosh 2009; Rovnanik 2010; Rattanasak and Chindaprasirt 2009*). *Thakur and Ghosh (2009)* and *Rovnanik (2010)* examined the influence of curing temperature on the compressive strength of geopolymer mortar. For this purpose, curing temperature was varied from 45°C to 120°C. Similarly, curing time was

varied from 6 to 72 hours and its effect was studied. *Rattanasak and Chindapasirt (2009)* examined the type of mixing of geopolymer during the preparation i.e. i) separate mixing and ii) normal mixing. It was found that separate mixing performed better than normal mixing. Finally it can be seen that literature review provides the range of values for various parameters. Adoption of any value needs trial and error method and engineering judgment in experimental studies.

Table 2.2: Typical variation in parameters of mix design of geopolymer mortar as adopted in literature

S.N.	Mix design parameter	Properties	Researchers	Values
1	Composition parameter	Alkali Content	Thakur and Ghosh (2009)	0.46 to 0.62 (0.62)
		Silica Content	Thakur and Ghosh (2009)	3.7 to 4.3 (4.0)
		Na ₂ SiO ₃ /NaOH	Rattanasak and Chindapasirt (2009)	0.5, 1, 1.5 and 2.0 (1)
		NaOH concentration (M)	Rattanasak and Chindapasirt (2009)	5, 10 and 15 (10)
		Alkali activator-FA ratio	Thakur and Ghosh (2009)	0.4 to 0.6
		Water –geopolymer solid ratio	Thakur and Ghosh (2009)	0.157 to 0.366 (0.3)
		Sand-FA ratio	Thakur and Ghosh (2009)	0.5 to 3.0 (1.5)
		Sand –Binder ratio (%)	Temuujin et al. (2010)	10 to 50(50)
2	Processing parameters	Curing temperature (°C)	Thakur and Ghosh (2009)	45 to 120 (85°)
			Rovnanik (2010)	10, 20, 40, 60, 80
		Curing time (hrs)	Thakur and Ghosh (2009)	6 to 72(48)
		Type of mixing	Rattanasak and Chindapasirt (2009)	Separate mixing
Values in parenthesis are concluded as optimum or value corresponding to maximum output.				

2.3.1 Influence of Mix Design Parameters on the Engineering Properties of Geopolymer Mortar

As explained in previous section, various mix design parameters govern the performance of geopolymer mortar. By virtue of being multi-phase mixer, evaluation of its performance is complex process. Researchers studied the influence of particular parameter on the performance of geopolymer by keeping other parameters constant. In this section, influence of some of the parameters on the behavior of geopolymer mortar is explained.

Composition Parameters

Composition parameters are defined as ratio, arithmetic relations or the properties of various constituents of the geopolymer mortar. In this section, influence of these parameters on engineering properties such as compressive strength, flexural strength of the final product is discussed.

Alkali Content: *Thakur and Ghosh (2009)* reported the variation in compressive strength (3 days, 7 days and 28 days) of the geopolymer mortar with different alkali content. It was noted that compressive strength increases with increase in alkali content within the geopolymer mortar. This was attributed to increased aluminosilicate gel due to increased alkali content.

Silica Content: *Thakur and Ghosh (2009)* investigated the influence of variation in silica content (3.7 to 4.3) on the compressive strength (3 days, 7 days and 28 days) of the geopolymer specimens.. It was observed that compressive strength increased up to optimum value of silica content. After exhibiting maximum compressive strength at optimum silica content, increases in silica content caused decrease in compressive strength of the geopolymer mortar. It was reported that increase in silica content beyond certain value, hinders the polymerization process within geopolymer mortar. The hindrance in polymerization causes the decrease in compressive strength.

Na₂SiO₃/NaOH: The influence of water glass to NaOH ratio on the mechanical strength of the geopolymer mortar as investigated by *Rattanasak and Chindaprasirt (2009)*. The ratio (water glass to NaOH) was varied from 0.5 to 2 while preparation of geopolymer mortar. It was observed that this ratio plays important role only at the lower concentration of NaOH.

NaOH concentration: The effects of variation in concentration of NaOH on the compressive strength of the geopolymer mortar was explained by *Rattanasak and Chindaprasirt (2009)*. It was noticed that for lower value of water glass to NaOH ratio, increase in NaOH concentration helped in augmenting in compressive strength. Geopolymer mortar prepared with water glass to NaOH ratio equal to one and 15 M NaOH exhibited compressive strength around 70Mpa.

Water –geopolymer solid ratio: Addition of water in the geopolymer helps in increased dissolution of Si and Al source, which in turn helps in polymerization as explained by *Thakur and Ghosh (2009)*. It was reported that compressive strength increases with increase in water to solid ratio up to certain limit. Beyond the optimum value of water solid ratio, compressive strength starts declining. The decrease in mechanical strength is due to super saturation of the paste and delay in gel formation.

Sand-FA ratio: *Thakur and Ghosh (2009)* demonstrated the effect of variation in sand-FA ratio (0.5 to 3.0) on the compressive strength (3 days, 7 days and 28 days) of the geopolymer mortar. It was concluded that compressive strength decreases drastically with increase in sand-FA ratio beyond the value of 1.5.

Sand –Binder ratio (%): *Temuujin et al. (2010)* described the influence of increase in aggregate content on the compressive strength of geopolymer mortar. The aggregate content was varied from 10% to 50% for preparation of the geopolymer mortar. It concluded that there is little or no effect of the change in aggregate content up to the limit of 50%.

Processing parameters

The Processing parameters are dependent on the procedure adopted in the preparation of the geopolymer mortar. This section summarizes the impact of the various parameters on the performance of the geopolymer mortar.

Curing temperature: *Thakur and Ghosh (2009)* studied the effect of curing temperature on the mechanical strength of the geopolymer mortar. For this purpose, compressive strength of geopolymer specimen having identical composition and cured at different temperatures (45°C to 120°C) was evaluated. It was concluded that maximum compressive strength was achieved at curing temperature of 85°C.

Curing time: *Thakur and Ghosh (2009)* investigated the effect of curing time on the mechanical strength of the geopolymer mortar. For this purpose, compressive strength of geopolymer specimen having identical composition and thermally cured for different time durations (5 to 72 hours) was evaluated. It was reported that Maximum compressive strength of 40.8Mpa was obtained with 48 hours of thermal curing

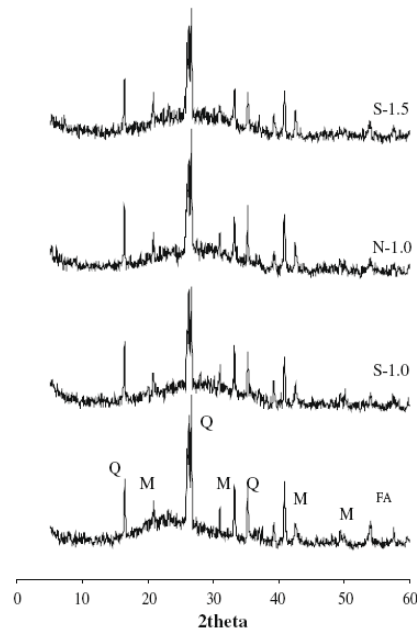
2.3.2 Influence of Mix Design Parameters on the Microstructure of Geopolymer Mortar

Mechanical strength and durability of the geopolymer mortar is essentially governed by the type of geopolymer gel, microstructure of the final product. Study of the microstructure of the geopolymer mortar gives opportunity to understand the mechanism and influence of the particular parameter on the performance of the mortar. Various researchers used advanced analytical techniques such as SEM (Scanning Electron Microscope) and XRD (X-Ray diffraction) techniques for delving deeper into and understanding the microstructure of the geopolymer.

Rattanasak and Chindaprasirt (2009) investigated the effect of leaching of fly ash particle with different concentration of NaOH using SEM technique. It was reported that surface of fly ash particle reacted with NaOH at the onset of leaching. Influence of leaching was higher in case of higher concentration of the sodium hydroxide. It was also reported that overall diameter of the fly ash particle also reduced after leaching of the fly ash.

Rattanasak and Chindaprasirt (2009) also studied the micro structure of the geopolymer paste.. It was noticed that formation of gel takes place around the fly ash particle. It was also observed that size of formed gel varies from 1 μ m to 20 μ m, hence it can be characterized as a colloid.

Rattanasak and Chindaprasirt (2009) studied the microstructure of the geopolymer mortar using XRD. Fig. 2.1 presents the XRD profile of the fly ash and various geopolymer specimens. Geopolymer and fly ash exhibited the identical patterns; hence, it can be concluded that geopolymerization did not change the amorphous and crystal properties of fly ash. Due to geopolymerization amorphous silica peak is shifted from 23° for original fly ash to 30° for fly ash. This indicated that presence of the highly disordered silicate glass phase in the geopolymer.



**Figure 2.1: XRD profiles of fly ash and geopolymer pastes: Q = quartz, M = mullite
(Source, Rattanasak and Chindaprasirt 2009)**

Thakur and Ghosh (2009) studied the influence of silica content on the microstructure of the geopolymer mortar. The larger fly ash particles were almost dissolved or their size was reduced and subsequently attained the denser and finer microstructure. It hints towards a higher degree of dissolution which, resulted in increasing the compressive strength of mortar from 26.73 Mpa to 35.20 Mpa.

2.4 Influence of Acid Attack on the Performance of Geopolymer Mortar

In the wake of climate change, acid rains are the reality in the urbanized cities of the world. As geopolymer mortar is proposed to replace the OPC mortar, it is essential to examine the performance of geopolymer mortar when subjected to acid attack. Researchers studied the influence of acid attack on the engineering properties of geopolymer mortar by using the nitric acid and sulfuric acid (*Thokchom et al. 2009* and *Thokchom et al. 2011*). Influence of the acid attack on the performance of geopolymer mortar was found to have similar pattern for both acids viz. nitric acid and sulfuric acid. In this study, aftereffects of the nitric acid on the fly ash based geopolymer mortar are focused and discussed.

2.4.1 Effect of Acid Attack on Mechanical properties

As geopolymer possesses the alkali content within its structure, its performance is expected to be affected by acid attack. In this study, literature regarding the change in mechanical properties at the onset of acid attack is reviewed. Mechanical properties such as visual appearance, weight loss and compressive strength were considered.

Visual Appearance: *Thokchom et al. (2011)* studied the influence of acid attack on the visual appearance of the geopolymer specimen. It was concluded that it is difficult to capture aftereffects of the acid attack from the naked eye. It was suggested to use the SEM in order to study the change in micro structure of the geopolymer at the onset of acid attack. However, for the preliminary investigation, phenolphthalein solution can be used to demarcate the dealkalised surface.

Weight loss: The geopolymer mortar shows the reduction in weight when exposed to acid. *Thokchom et al, (2011)* examined the influence of alkali content on the change in weight of geopolymer mortar. It was reported that change in weight was very less due to acid attack. However, geopolymer having lowest alkali content exhibited highest loss of weight.

Compressive strength: Attack of acid substance causes chemical changes in the geopolymer matrix, which results in to reduction in compressive strength. *Thokchom et al. (2011)* investigated the effect of duration of exposure to the acid substance on the mechanical strength of the geopolymer. It was concluded that compressive strength decreases with increase in duration of exposure. Geopolymer prepared with higher alkali content (GM3) showed least reduction in compressive strength in geopolymer mortar specimen.

2.4.2 Effect of Acid Attack on Micro structure of the Geopolymer Mortar

Thokchom et al. (2011) examined the effect of acid attack on the micro structure of the mortar. For this purpose, traces of unexposed and exposed surface were examined and compared with help of SEM-EDX technique. Micrographs of the geopolymer mortar were captured and elemental composition was evaluated. It was reported that microstructure deteriorated with acid attack. Further, calcium content in the traces of surface diminished with exposure to acidic substance.

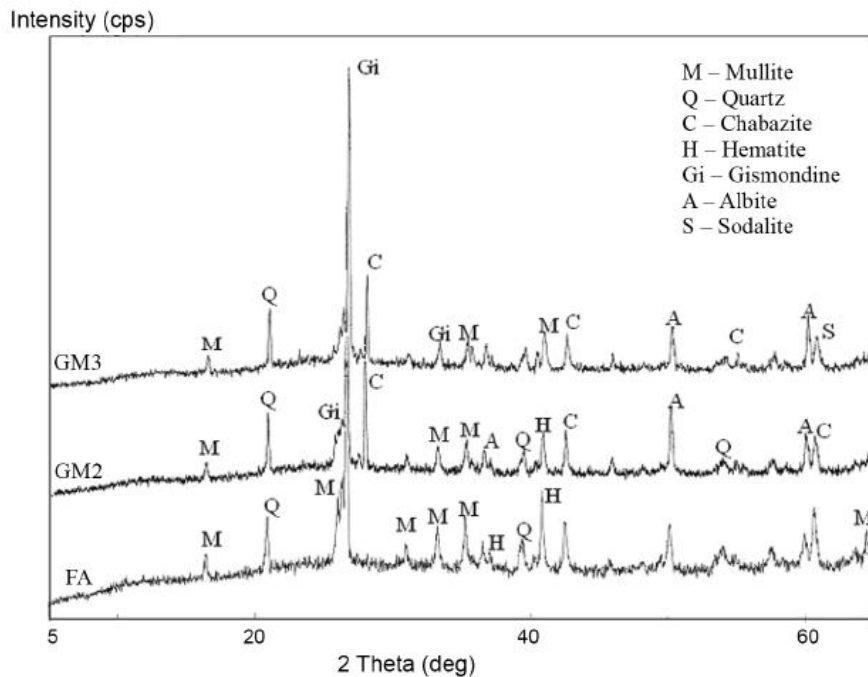


Fig. 2.2: XRD spectra of geopolymer mortar specimens after 18 weeks in nitric acid
(Source, Thokchom et al. 2011)

Similarly, *Thokchom et al. (2011)* also investigated change in mineralogical composition in geopolymer mortar in the wake of acid attack using XRD technique. Fig. 2.2 depicts the XRD spectra of the fly ash and geopolymer mortars. It was concluded that there was very less change in the phase of fly ash. However, due to acid attack, traces of grismondine were found to be prominent in both types of geopolymers.

2.5 Carbonation of the geopolymer mortar

In the wake of climate change, concrete and mortar were found to be vulnerable to loss in strength at the onset of the carbonation. The carbonation process is defined as reaction of concrete or mortar to the atmospheric carbon dioxide in the presence of humidity. The atmospheric carbon dioxide comes into contact to the porous concrete/mortar matrix due its permeation phenomenon. In the presence of humidity, CO_2 is converted into carbonic acid (weak acid). In conventional mortar, the carbonic acid reacts with $\text{Ca}(\text{OH})_2$ and C-S-H gel (alkaline medium) and produces Calcium Carbonate (CaCO_3). The formation of Calcium Carbonate (CaCO_3) leads to the deterioration of mortar.

On the other hand, geopolymer is made up of complex compounds $[M_z(\text{AlO}_2)_x(\text{SiO}_2)_y.n\text{MOH}.m\text{H}_2\text{O}]$ (aluminosilicate gel). Upon interacting with the Carbonic acid (H_2CO_3), which is product of atmospheric (CO_2) and humidity (H_2O), geopolymer gel is converted into sodium carbonate hydrate (Law 2015). The formation of sodium carbonate hydrate makes the mortar vulnerable to deterioration.

Due to carbonation of the mortar/concrete, the corrosion protection of reinforcing steel may be destroyed. Reinforcement corrosion in the concrete/mortar structures is most critical to determine the service life of the structure. Hence, it is important to estimate the time when the carbonation front reaches to the reinforcement.

Carbonation of the mortar is evaluated by two methods. They are: i) Carbonation in natural environment and ii) accelerated carbonation in laboratory. In first method, multiple testing specimens are molded and cured up to 28 days. After sufficient curing, the specimens of mortar are exposed to natural environment, which may contain the natural content of carbon oxides in conjunction with rains. The exposure time may vary from short duration of few days to long term duration of few months or years. As this method is very close to real time situation, it tends to produce more realistic results. However, this type of testing is time and labor consuming, which makes it economically less feasible.

On the other hand, accelerated carbonation method is adopted when the required time and resources are not available for the long term experiments. In the accelerated carbonation, controlled environment in terms of temperature and humidity is created. Further, mortar specimen is subjected to carbon dioxide exposure. The increased concentration of the CO_2 is adopted in comparison to natural concentration of CO_2 in atmosphere. The increased concentration of CO_2 helps in its accelerated permeation into pores of mortar, which consequently helps increasing in carbonation process in the mortar. Fig. 2.3 shows the schematic diagram of accelerated carbonation chamber. The chamber provides the controlled environment for humidity and carbon dioxide.

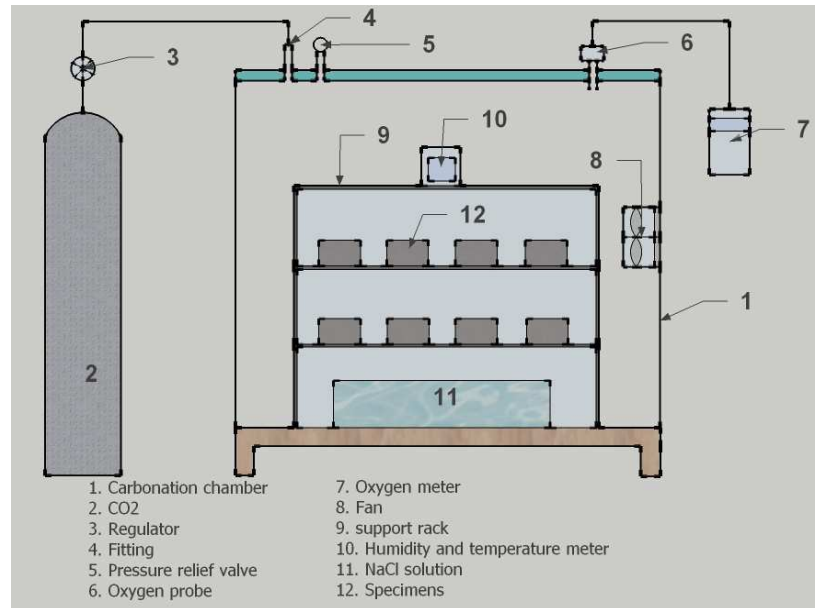


Figure 2.3: Schematic diagram of accelerated carbonation chamber (Adam 2009)

2.5.1 Quantification of carbonation of the geopolymer mortar

The rate and amount of carbonation decides the durability of the geopolymer mortar. Several researchers attempted to quantify the rate and amount of carbonation using experimental set-ups. As we know, the diffusion of CO₂ leads to change in pH of the mortar, the evaluation of pH can be used to evaluate the vulnerability of mortar when subjected to carbonation. Due to attack of weak acid (carbonic acid, H₂CO₃) on the alkaline medium, pH of the geopolymer reduces to 10 approximately. The effects of carbonation are identified by sprinkling phenolphthalein on the carbonated surface of geopolymer and monitoring change in color.

After the carbonation of specimens, they are split and cleaned. The depths of carbonation were obtained by spraying on a newly split surface of mortar specimen with mixer of 1% of phenolphthalein and the solution of 70% ethyl alcohol. The phenolphthalein solution is colorless and used as an acid–base indicator. When the value of pH is found to be higher than the nine, color of the solution changes into purple. Hence, no change in color indicates towards, carbonated surface and purple color indicates towards non-carbonated surface.

Extent of carbonation is measured in terms of carbonation depth as given below (RILEM 1988).

$$X = C(t)^{1/2} \quad (2.5)$$

where X is the carbonation depth(mm), C is the carbonation coefficient (mm/month^{0.5}) and t is the exposure period of accelerated carbonation(months). The formulation in Eq. 2.28 had been agreed upon by numerous researchers (Chang et al. 2004; Houst and Wittmann 2002; Sulapha et al. 2003). For analyzing the effect of carbonation, advance analytical techniques such as FESEM-EDX and XRD can be employed (Law 2015). Law 2015 reported that final pH (10-10.5) of the carbonated geopolymer was higher as compared to pH of the ordinary concrete (approximately equal to 9).

The amount of carbonation is governed by various factors such as duration of carbonation, mix composition of the mortar, process parameters involved in the preparation of the mortar. Unlike the conventional ordinary Portland mortar, the carbonation of geopolymer mortar is governed by change in pore solution chemistry of the mortar. In the view to investigate the mechanism behind the formation of various products in the mortar during the carbonation, Bernal et al. (2012) used the XRD and correlated with thermodynamics of the chemical reactions. Further, Bernal et al. (2013) studied the influence of carbonation on the micro structure of the mortar with help of advanced analytical techniques such as XRD and nuclear magnetic resonance spectroscopy.

2.6 Summary of literature and its Critical Appraisal

In this study, an attempt has been made to understand the behavior of the geopolymer mortar, in particular fly ash based geopolymer mortar with help of literature review. Based on findings from literature review, following summary can be reported.

- i) Various wastes by products of the industry can be used for manufacturing of the geopolymer mortar such as fly ash, rice husk fuel ash and GGBS etc.
- ii) Strength of fly ash based geopolymer is governed by its mix compositions such as alumina content, silica content, concentration of alkali activator etc.
- iii) Strength of fly ash based geopolymer is found to be influenced by processing parameters such as curing period and curing temperature.
- iv) Amorphous phase of the fly ash is not altered significantly during geopolymerisation.

v) Durability is affected significantly by carbonation which is evident by SEM micrographs.

However it was found that the research is primarily focused on the geopolymer concrete. The influence of the mix composition on the performance of the geopolymer mortar was found to be very limited. Further, the influence of the carbonation of the geopolymer mortar was also not studied in details. The lack of availability for the guidelines for the preparation of geopolymer mortar and its performance formed the motivation behind the present study.

CHAPTER-3

CHARACTERIZATION OF MATERIALS USED IN THE PRESENT STUDY

3.1 General

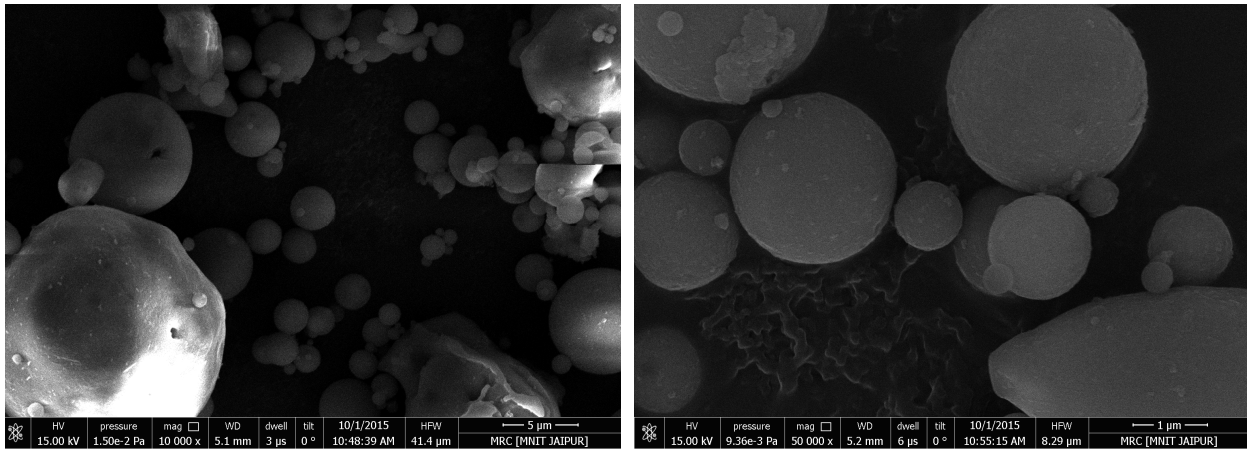
Geopolymer mix is prepared by blending the various components such as Fly ash, fine sand, Sodium Hydroxide (NaOH) and Sodium Silicate (Na_2SiO_3) in different stages. Further, strength properties of geopolymer are attained by the polymerization process. The geo-polymerization process takes place during curing period under elevated temperatures. The strength of final product of the geopolymer is also influenced by physical properties of its constituents. In order to understand the behaviour of geopolymer mortar, it is vital to have in-depth understanding of its components. For this purpose, properties of individual constituent of geopolymer mortar were investigated. In this study, four components were used for the preparation of geopolymer mortar, they are i) Fly ash, ii) Sand, iii) Sodium Hydroxide (NaOH) and iii) Sodium Silicate (Na_2SiO_3). Summary of the properties of these materials are reported below.

3.2 Fly ash

In this study, fly ash used in the present study was procured from Dirk India Limited Nasik. Based on proportion of silica (SiO_2), alumina (Al_2O_3) and iron (Fe_2O_3), the fly ash is classified as class F as per ASTM C-618 standards. Further, physical properties such as fineness, lime reactivity, maximum moisture content and autoclave expansion were obtained. Table 3.1 summarizes the physical properties of the fly ash.

Table 3.1: Physical properties of Fly ash used in the present study (*Dirk India Limited*)

S. N.	Properties	Unit	Standard values (IS-3812)	Experimental Values (Pozzocrete 63)
1	Fineness – Specific Surface by Blaine’s Permeability Method (Min.)	m^2/kg	320	428
2	ROS # 350 (45 MIC) Max.	%	34	6.92
3	Lime Reactivity (Minimum)	N/mm^2	4.5	6.60
4	Moisture Content (Max.)	%	2	0.23
5	Autoclave Expansion (Max.)	%	0.8	0.024



a) Fly ash

b) Fly ash (Closer view)

Figure 3.1: Micrographs obtained from Scanning Electron Microscope (SEM) Fly ash

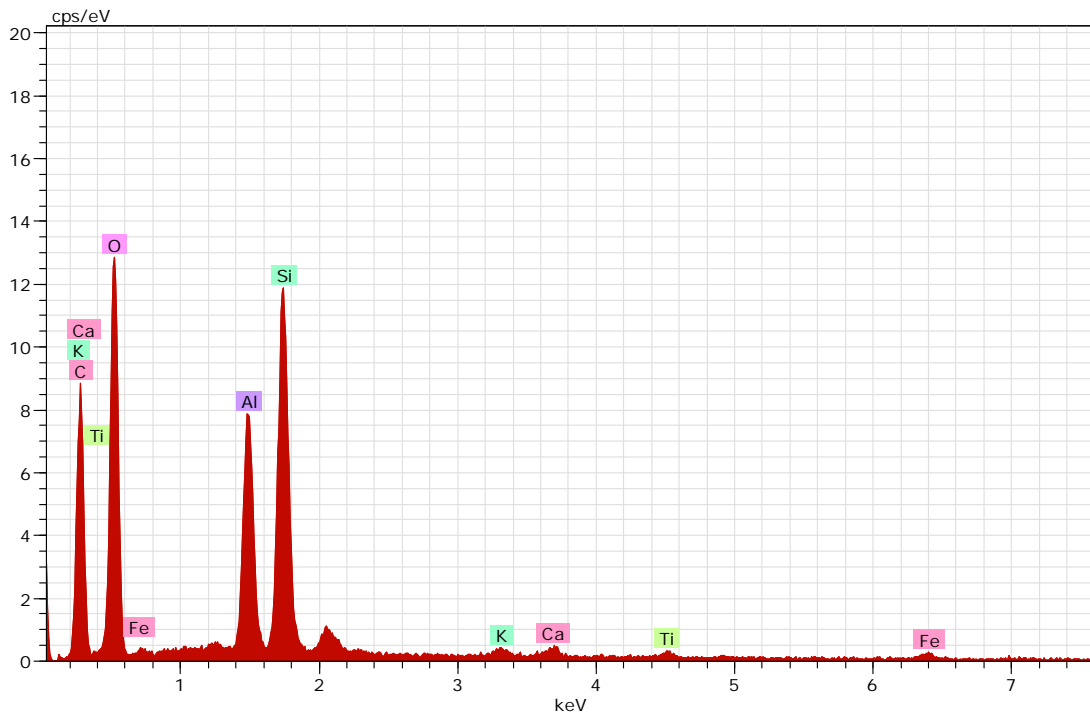


Figure 3.2: Elemental composition from Scanning Electron Microscopy -EDS (SEM) for Fly ash

Fig. 3.1 shows the view of fly ash used in the present report. It can be seen that the fly ash has round particles with smooth external surface. The elemental composition of the fly ash was also

determined with help of Energy dispersion spectroscopy (EDS) technique. Specific gravity of the fly ash was evaluated using Le Chatelier Flask. Table 3.2 presents the calculation methodology adopted for the evaluation of specific gravity. The average specific gravity of fly ash was found to be 2.18.

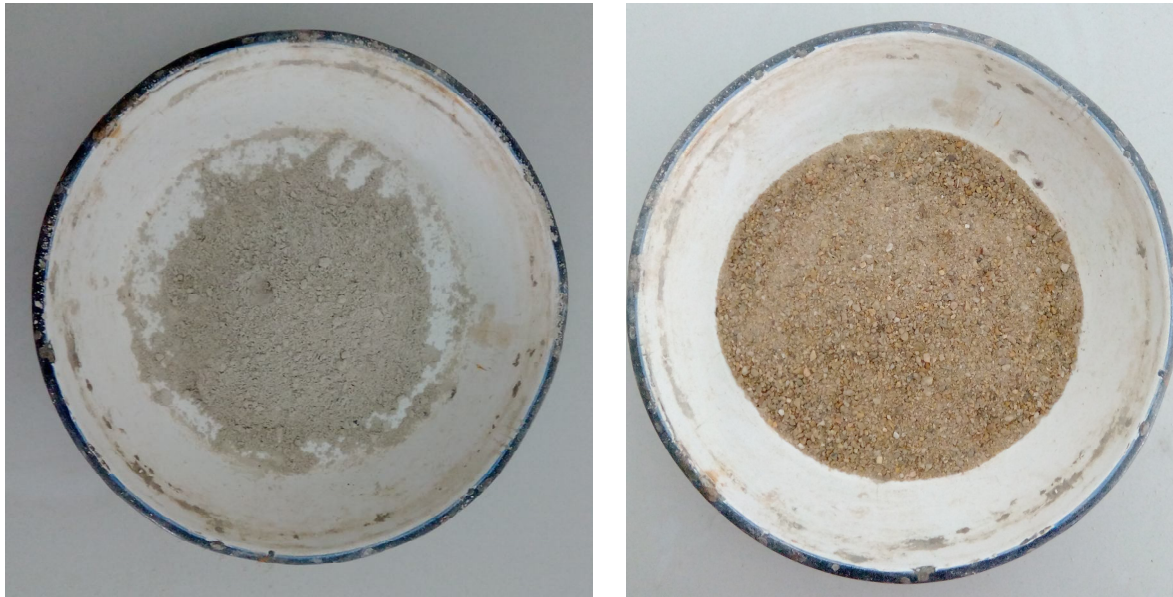
Table 3.2: Calculation of specific gravity of fly ash used in the present study

Particulars	Unit	Sample 1	Sample 2
Weight of empty flask (W_1)	gm	30	30
Weight of flask + fly ash (W_2)	gm	80	80
Weight of flask + fly ash + kerosene (W_3)	gm	107.5	107
Weight of flask + kerosene (W_4)	gm	72.5	72.5
<i>Calculation</i>			
Specific gravity of fly ash ($(W_2 - W_1) / ((W_2 - W_1) - (W_3 - W_4)) * 0.78$)		2.2	2.165
Average Specific Gravity of fly ash			2.18

During the curing, fly ash reacts with other components chemically. The chemical properties of the fly ash plays vital role to attain the strength properties and resistance for durability. Hence, it is imperative to investigate the chemical composition of the fly ash. Table 3.3 presents the chemical composition of the fly ash. The chemical composition of the fly ash was found in tandem with the elemental analysis obtained from the EDS analysis.

Table 3.3: Chemical properties of Fly ash used in the present study (Dirk India Limited)

Test No.	Properties	Unit	Standard values (IS-3812)	Experimental Values (Pozzocrete 63)
1	Loss on Ignition (Max.)	%	5	0.94
2	$SiO_2 + Al_2O_3 + Fe_2O_3$	%	70 min. by mass	92.26
3	SiO_2	%	35 min. by mass	58.88
4	MgO	%	5 max. by mass	1.64
5	SO_3	%	3.00 max. by mass	0.74
6	Na_2O	%	1.5 max. by mass	0.50
7	Total Chlorides	%	0.05 max by mass	0.025



a) Fly ash

b) Fine aggregate

Figure 3.3: Fly ash and fine aggregate used in the present study

3.3 Fine aggregate

Figure 3.3 demonstrates the view of the fine aggregate used in the present study. The summary of properties of the fine aggregate is presented in Table 3.4. The fine aggregate, having rounded and sub rounded particles. Figure 3.4 demonstrates the particle size distribution curve of the fine aggregate. It can be seen that the particle size distribution curve of the fine aggregate lies between the curve representing the maximum and minimum boundary of the Zone-2. Further, the fineness modulus was found to be equal to 2.66. Based on the fineness modulus, it can be classified as zone-2.

Further, the specific gravity was evaluated using Pycnometer as explained in Table 3.4. The average specific gravity was obtained by performing the test on identical samples. The average specific gravity of the fine aggregate was found to be equal to 2.598.

The water absorption of the fine aggregate was computed as explained in Table 3.4. For this purpose, the fine aggregates were saturated for 24 hours and dried in oven. In this process all the relevant weights were recorded and water absorption was computed, which is found to be equal to 2.41%.

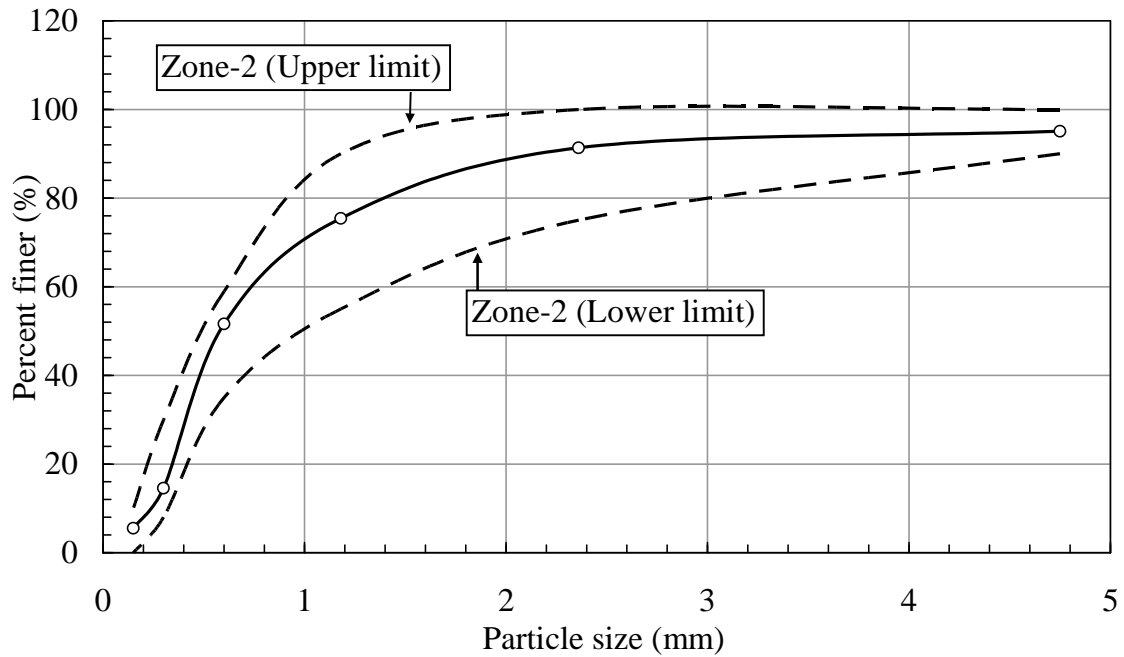


Figure 3.4: Particle size distribution curve for fine aggregate

Table 3.4: Calculation of specific gravity and water absorption capacity of fine aggregate

S. N.	Particulars	Unit	Sample 1	Sample 2
1	Weight of Pycnometer (W_1)	gm	647	647
2	Weight of Pycnometer + Aggregate (W_2)	gm	1147	1147
3	Weight of Pycnometer + Aggregate + Water (W_3)	gm	1847	1844
4	Weight of Pycnometer + Water (W_4)	gm	1538	1538
5	Weight of Saturated Surface Dry Aggregate in Air (W_5)	gm	511	507
6	Weight of Oven Dry Aggregate (W_6)	gm	499	495
Calculation				
7	Specific Gravity = $(W_2 - W_1) / (W_2 - W_1) - (W_3 - W_4)$		2.618	2.577
8	Average Specific Gravity		2.598	
9	Water Absorption = $((W_5 - W_6) / W_6) \times 100$	%	2.4	2.424
10	Water Absorption	%	2.412	

3.4 Sodium Hydroxide (NaOH)

Liquid sodium hydroxide was prepared from the solid sodium hydroxide pallets (98% pure) by using appropriate amount of water to achieve predefined concentration of the same.

3.5 Sodium Silicate (Na₂SiO₃)

Locally available Sodium Silicate was used in the present study.

CHAPTER-4

EXPERIMENTAL INVESTIGATION ON THE GEOPOLYMER MORTAR

4.1 General

The investigation of various parameters provides the platform to understand the behaviour of the geopolymer mortar. It will help enhance the opportunity to usage of geopolymer mortar in various construction purposes. Further, the influence of environmental actions such as attack of carbon dioxide (CO₂) on the durability of the mortar helps anticipating the service life of the mortar. In the present study, two series of experimental investigations were conducted. They are Series A (influence of mix proportions on the compressive strength of the mortars) and Series B (influence of mix proportions on the durability of the mortars against carbonation). This chapter discusses the testing procedure, testing programs and their results and discussions.

4.2 Investigation on Compressive Strength

The series A contains the test program for the examining the effect of mix proportions on the compressive strength of the geopolymer mortar. This section presents the testing methodology, testing program, results and discussions.

4.2.1 Test Method

Preparation of mortar specimen

For preparation of geopolymer mortar specimens, fly ash and alkaline activating solution (NaOH and Na₂SiO₃) in predefined proportions were blended together using manual mixing technique. Further, the fine aggregate was mixed into the activated fly ash mix paste another five minutes. The prepared mortar was mix upto when the good consistency was achieved. Then prepared mortar was poured in to the steel moulds having length of 50mm, breadth of 50mm and height of 50mm and vibrated using vibration table up to 2 minutes to get rid of entrapped air (Fig 41.). The specimens were preserved at room temperature for the duration of 300 minutes. The specimens were cured at constant temperatures of 60°C or 90°C for 24 hours. After completion of curing, the mortar samples were removed by unpacking the moulds, and kept for cooling at room temperature.



a) Moulds



b) Specimens

Figure 4.1: perspective view of the moulds and geopolymer mortar specimens



Figure 4.2: The compression testing machine for the testing of geopolymer mortar

Testing procedure

The mortar samples obtained after curing are selected for testing for compression tests. Specimens are tested according to IS-2250. Three sample each having identical mix design were tested for compression test. The average compressive strength was computed from these samples. Further, the crushed samples were obtained from these samples for further microscopic analyses. The crushed powder was utilized to analyze their mineralogical composition and structural arrangement.

Table 4.1: Summary of the test program adopted for Series A in the present study

S.N	Test legends	NaOH concentration	Aggregate to binder ratio	Curing temperature
1	S-1	14M	1:1	60°
2	S-2	14M	2:1	60°
3	S-3	14M	3:1	60°
4	S-4	11M	1:1	60°
5	S-5	11M	2:1	60°
6	S-6	11M	3:1	60°
7	S-7	8M	1:1	60°
8	S-8	8M	2:1	60°
9	S-9	8M	3:1	60°
10	S-10	14M	1:1	90°
11	S-11	14M	2:1	90°
12	S-12	14M	3:1	90°
13	S-13	11M	1:1	90°
14	S-14	11M	2:1	90°
15	S-15	11M	3:1	90°
16	S-16	8M	1:1	90°
17	S-17	8M	2:1	90°
18	S-18	8M	3:1	90°

4.2.2 Test Program

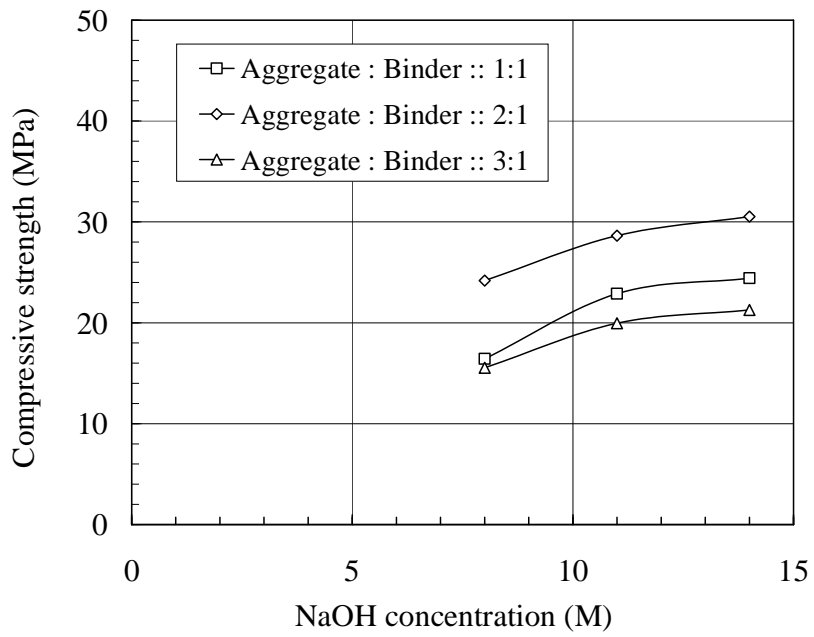
Based on the literature, the proportions of the several parameters for the preparation of mix composition were adopted such as alkaline activator to fly ash ratio, Sodium Silicate to Sodium hydroxide ratio, curing duration and delay time. Here the delay time refers to the time duration between, the pouring of the mix into the moulds and the initiation of the curation by heating. The value of the alkaline activator to fly ash ratio, Sodium Silicate to Sodium hydroxide ratio and delay time were considered 0.4, 1 and 5 hours. In the present study, parameters such as NaOH concentration, aggregate to binder ratio and curing temperature were varied by keeping above mentioned parameters constant. The table 4.1 summarizes the test program for the Series A. It is important to note that the test program given in the table 4.1 was conducted twice. They are i) Series A1 for samples cured upto 7 days, ii) series A2 for samples cured up to 28 days.

4.3 Results and Discussion

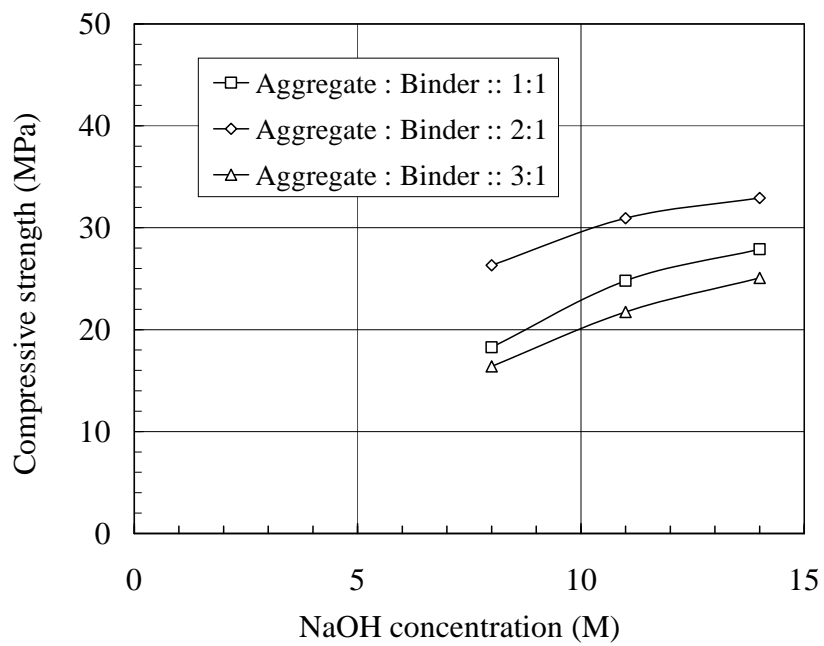
The results of the series A are reported in terms of compressive strength. The compressive strength of the specimen is computed by taking average compressive strength obtained from three samples having identical compositions. As discussed in Table 4.1, the present section presents the influence of mix composition parameters on the compressive strength of the mortar.

4.3.1 Influence of NaOH concentration on Compressive Strength

Fig 4.3 shows the variation of compressive strength achieved at 7 days with the concentration of NaOH. As can be noticed, the compressive strength increases with the increase in concentration of Alkali activator. The increase in compressive strength can be attributed to higher amount to leaching of Si^{4+} and Al^{3+} ions . The leaching of Si^{4+} and Al^{3+} ions led to the formation of aluminosilicate gel, which in turn provides the compressive strength to the mortar sample. The maximum compressive strengths for the mortar cured at 60°C and 90°C were found to be equal to 30.53Mpa and 32.92MPa respectively. The maximum compressive strengths were achieved at the 14M concentration of alkali activator.

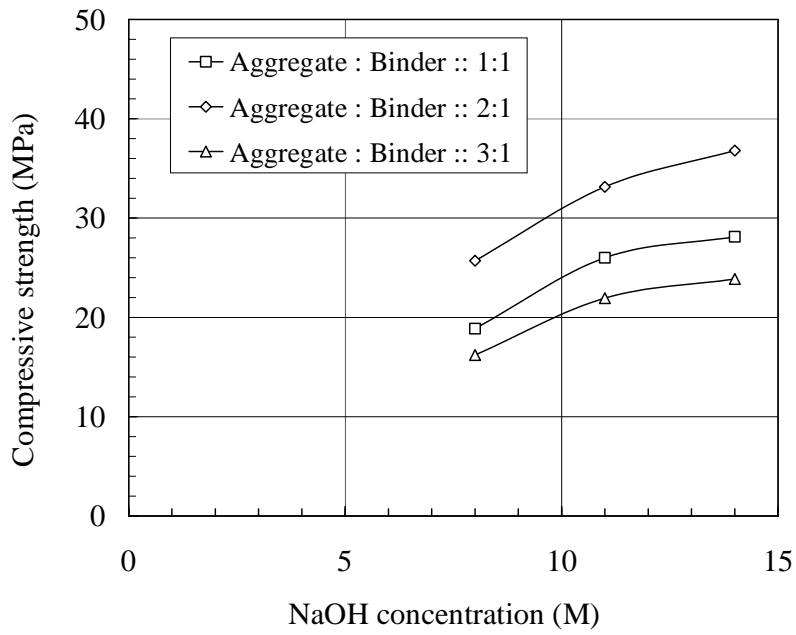


a) Curing temperature(T_c) = 60°C

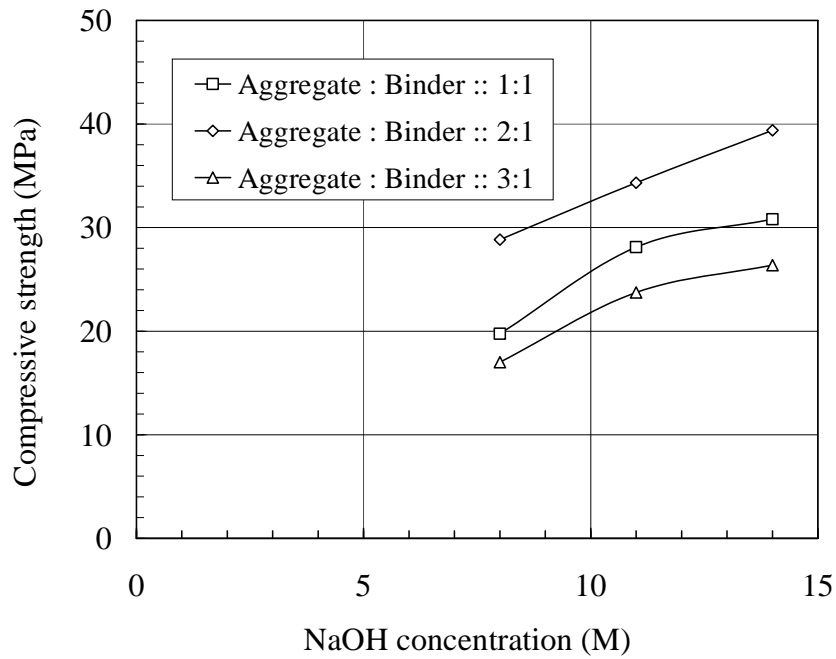


b) Curing temperature(T_c) = 90°C

Figure 4.3: Variations in 7 days compressive strength of mortar with concentration of NaOH



a) Curing temperature(T_c) = 60°C



b) Curing temperature(T_c) = 90°C

Figure 4.4: Variations in 28 days compressive strength of mortar with concentration of NaOH

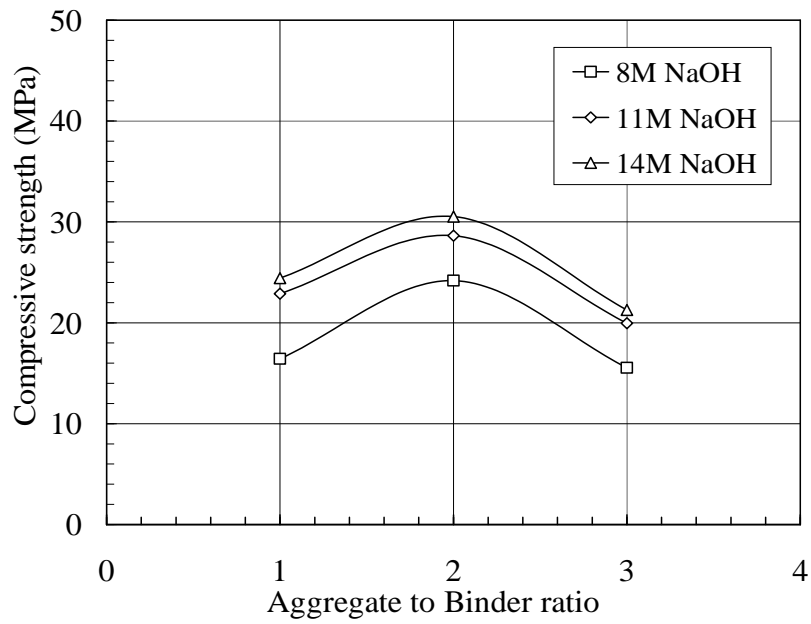
Figure 4.4 shows the comparison of variation in compressive strength (at 28 days) of the mortar cured at curing temperature of 60°C and 90°C with concentration of sodium hydroxide. It was observed that compressive strength enhanced significantly with addition of NaOH having increased concentration (8M, 11M and 14M).

The increased compressive strength was due to higher dissolution of Si^{4+} and Al^{3+} ions, which resulted in presence of higher amount of alumino-silicate gel within the matrix of mortar. The alumino-silicate gel attains compressive strength when subjected to higher temperature. The increase in temperature upto certain extent may help in achieving higher strength, which is evident from results that compressive strength of mortar cured at 90°C was higher than that cured at 60°C (Fig 4.4).

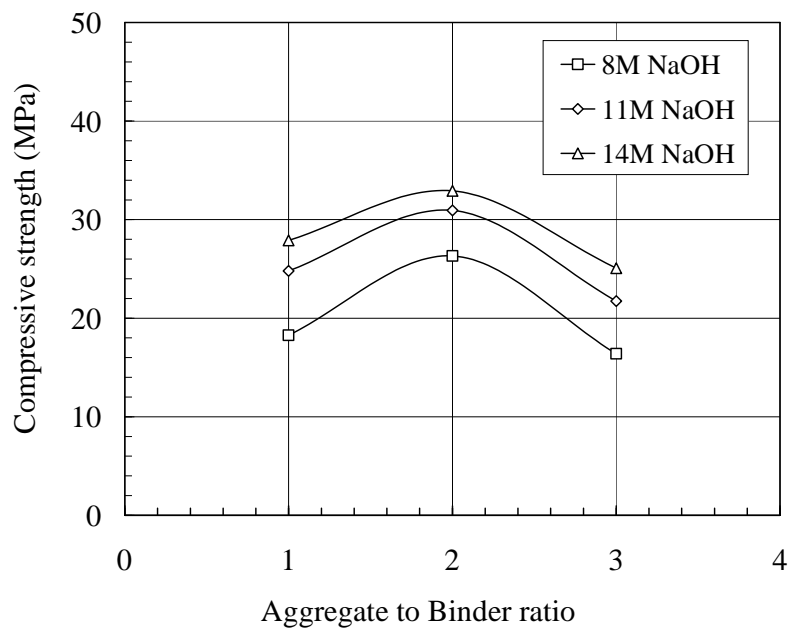
4.3.2 Influence of aggregate to binder ratio on Compressive Strength

Figure 4.5 depicts the variation of 7-days compressive strength of geopolymer mortar with the change in aggregate to binder ratio. For this purpose, various concentrations of NaOH were also used. It was found that compressive strength of the mortar increases with decrease in aggregate content from 3:1 to 2:1. Further, decrease in aggregate content from 2:1 to 1:1 leads to decrease in compressive strength.

Compressive strength of alkali activated fly ash geopolymer mortar is governed by strength of the binder (alkali activated fly ash) and proper bonding between fine aggregate and binder material. Increase in compressive strength at low values of aggregate to binder ratio can be attributed to proper bonding between fine aggregate and geopolymer mortar. Further, decrease in compressive strength due to decrease in aggregate content can be attributed to increased porosity of the geopolymer mortar due to presence of smaller fraction of fine aggregates.



a) Curing temperature (T_c) = 60°C



b) Curing temperature (T_c) = 90°C

Figure 4.5: Variations in 7 days compressive strength of mortar with Aggregate to Binder ratio

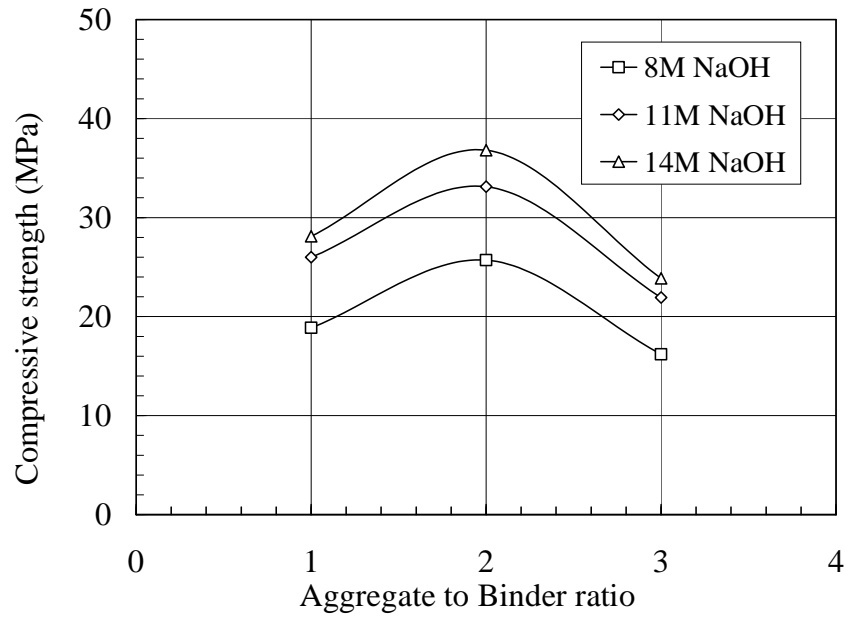
Further, influence of change in concentration of sodium hydroxide on the compressive strength for specimens having similar variation of aggregate content was investigated. Identical pattern of the variation of compressive strength with the change in aggregate content was measured for all the samples having sodium hydroxide of different concentration.

However, it was observed that specimen prepared with high concentration of sodium hydroxide possessed higher compressive strength. It can be due to increased alkali activation of fly ash and proper leaching of Al^{3+} and Si^{4+} ions from fly ash to geopolymer matrix.

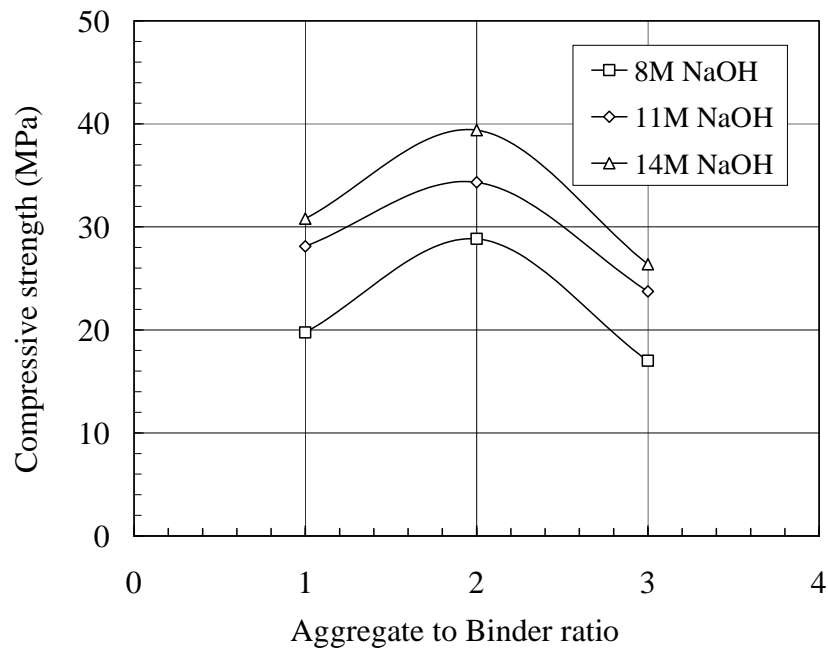
Fig 4.5a demonstrates the variation of 7 days compressive strength of geopolymer mortar with aggregate content and concentration of NaOH for curing temperature of 60°C. The nature of curve were found to be identical for curing temperature 60°C and 90°C. The maximum compressive strength of 26.31Mpa, 30.93Mpa and 32.92Mpa at optimum binder to sand ratio of 2:1 were obtained for different concentration of NaOH of 8M, 11M and 14M respectively.

Figure 4.6 demonstrates the variation 28 days compressive strength with concentration of NaOH and aggregate to binder ratio for the specimen cured at temperatures 60°C and 90°C. Maximum compressive strength was attained by mixing aggregate-binder ration in 2:1 for concentration of NaOH equal to 8M, 11M and 14M. Higher compressive strength were observed for higher concentration of NaOH which signifies the importance of contribution of leaching phenomena for achieving compressive strength.

Further, the influence of curing temperature on the compressive strength was also examined. It was noticed that higher curing temperature helped achieving higher compressive strength. It highlights the hardening of the geopolymer matrix due to heating of the geopolymer mortar.



a) Curing temperature(T_c) = 60°C

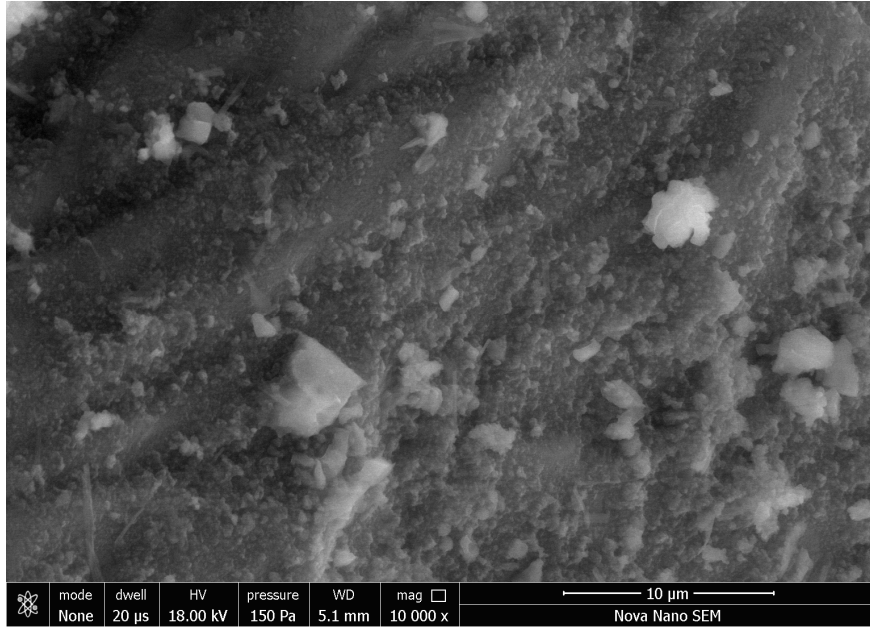


b) Curing temperature(T_c) = 90°C

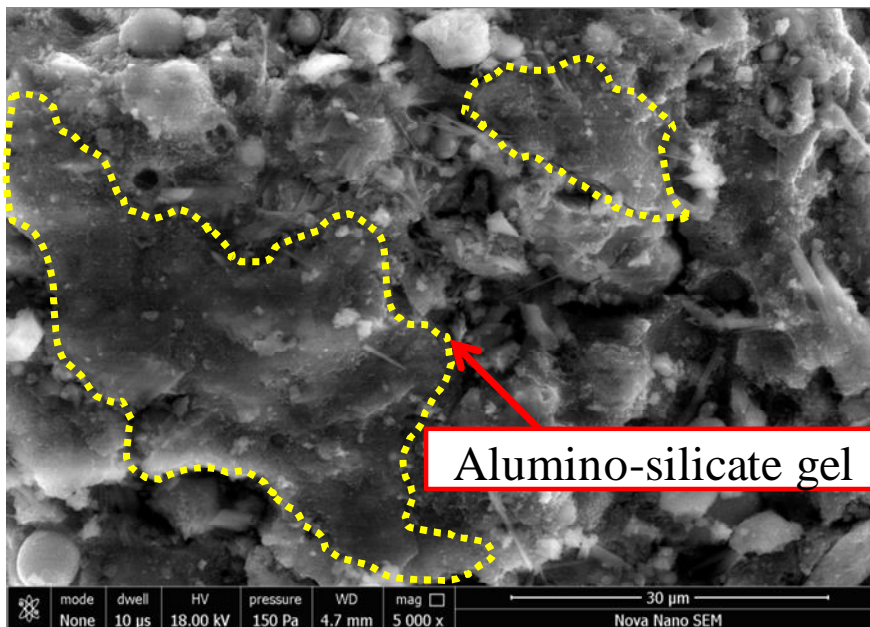
Figure 4.6: Variations in 28 days compressive strength of mortar with Aggregate to Binder ratio

4.3.3 Influence on Micro-structure of Geopolymer Mortar

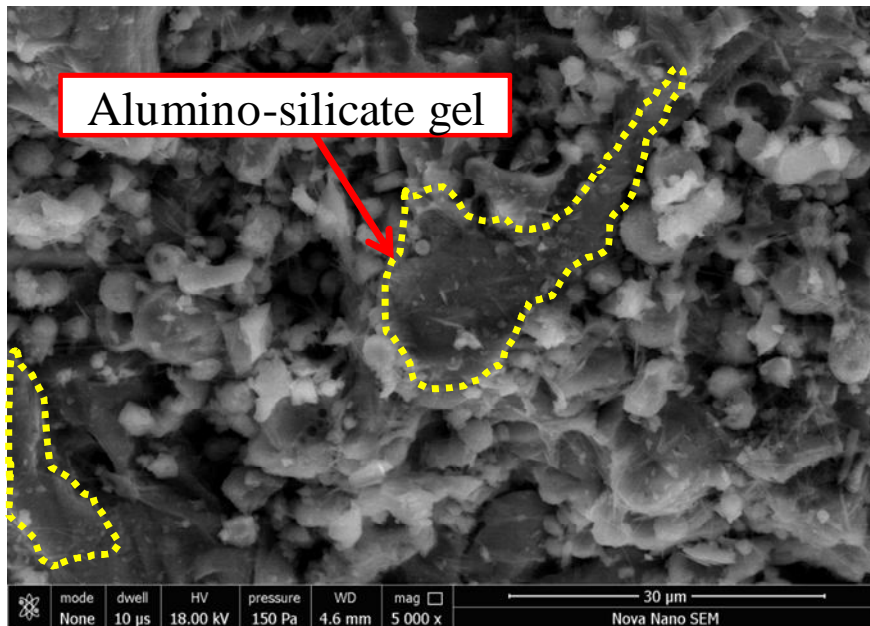
Scanning Electron Microscopy



a) Aggregate to binder ratio = 1:1



b) Aggregate to binder ratio = 2:1



c) Aggregate to binder ratio = 3:1

Figure 4.7: Micrographs of geopolymer mortar obtained from SEM (14 M NaOH)

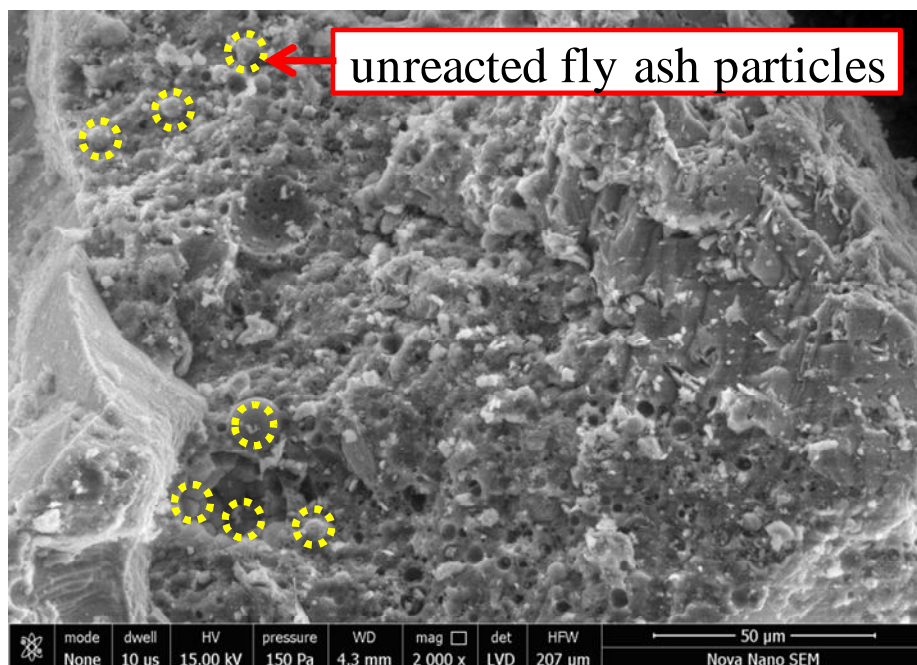
In the present study, Scanning electron microscope available at Material Research Center, MNIT Jaipur was used to investigate the variations in surface features in the geopolymer mortar and unreacted fly ash. Fig 4.7 illustrates the micrographs of the mortar made up of 14M NaOH using varying aggregate to binder ratio. Plenty of Alumino-silicate gel was observed in mortar made up of aggregate to binder ratio equal to 1:1. In case of aggregate to binder ratio 2:1 and 3:1, the amount of the gel present in the matrix of the mortar decreases. The micrographs are in consonance with the compressive strength results.

Fig. 4.8 demonstrates the micrographs obtained from SEM for the mortar made of using sodium hydroxide of varying concentration. NaOH acts as dissolution and leaching agent during the preparation of mortar. The higher concentration of the NaOH accelerates the dissolution of fly ash particles in to the matrix which in turn will become the alumino-silicate gel. In Fig 4.8, in matrix of the combination of the gel, fly ash and fine aggregate, round particles of the fly ash can be noticed. It was observed that in case of mortar made up of 8 M NaOH, significant number of unreacted fly ash particles were present. The presence of unreacted fly ash particles indicates towards the insufficiency of the NaOH to dissolve all the fly ash particle and under-utilisation

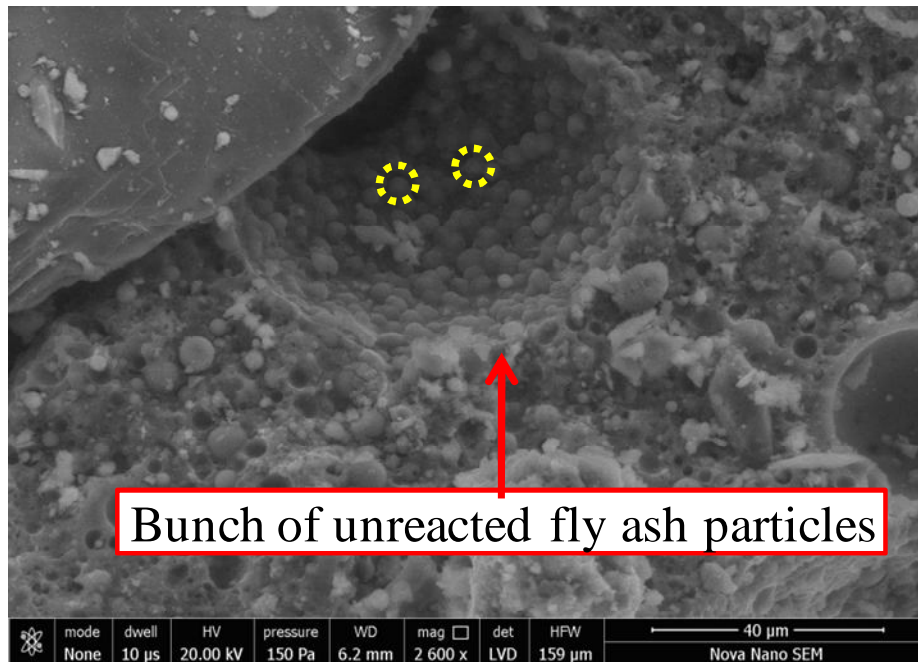
the potential of mortar to achieve strength. However, in case of the mortar with 11M and 14 NaOH, a noticeable improvement in dissolution of fly ash particle was noted.



a) 14 M NaOH



b) 11 M NaOH

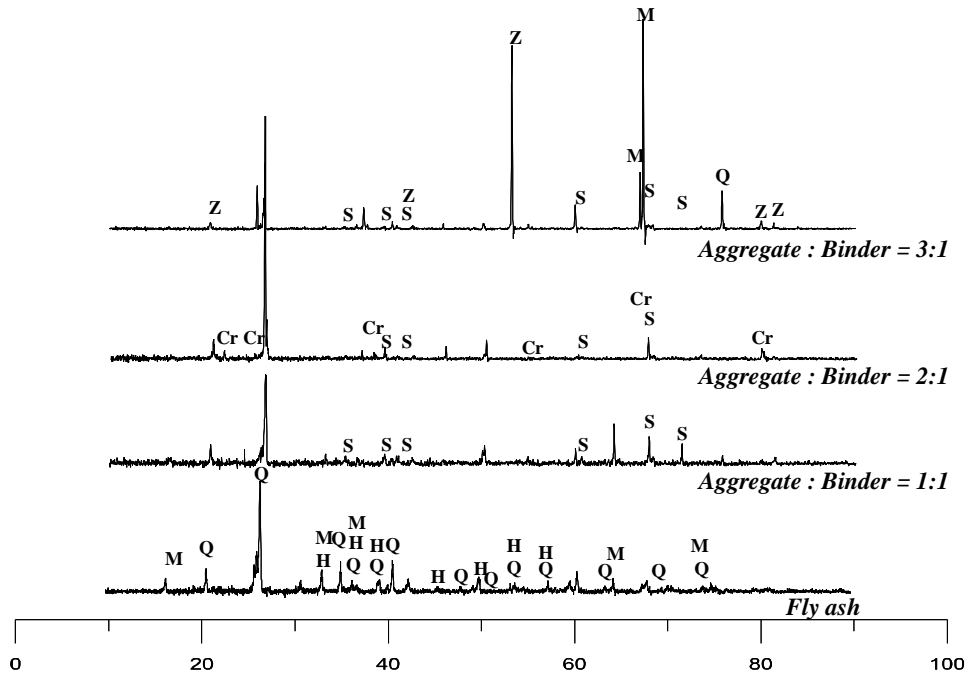


c) 8 M NaOH

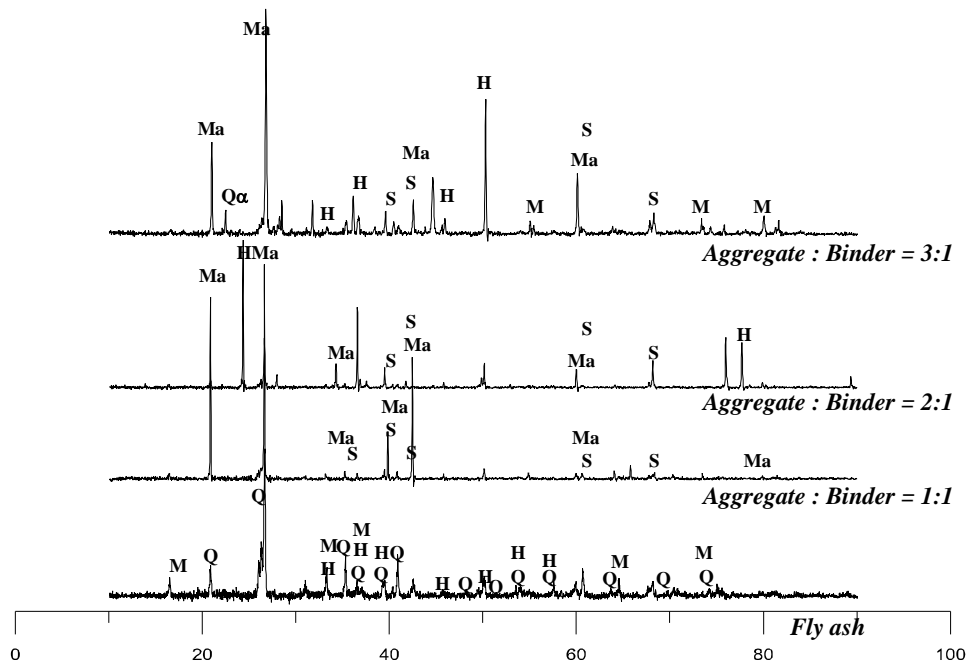
Figure 4.8: Micrographs of geopolymer mortar at various concentration of NaOH

X-ray diffractometry

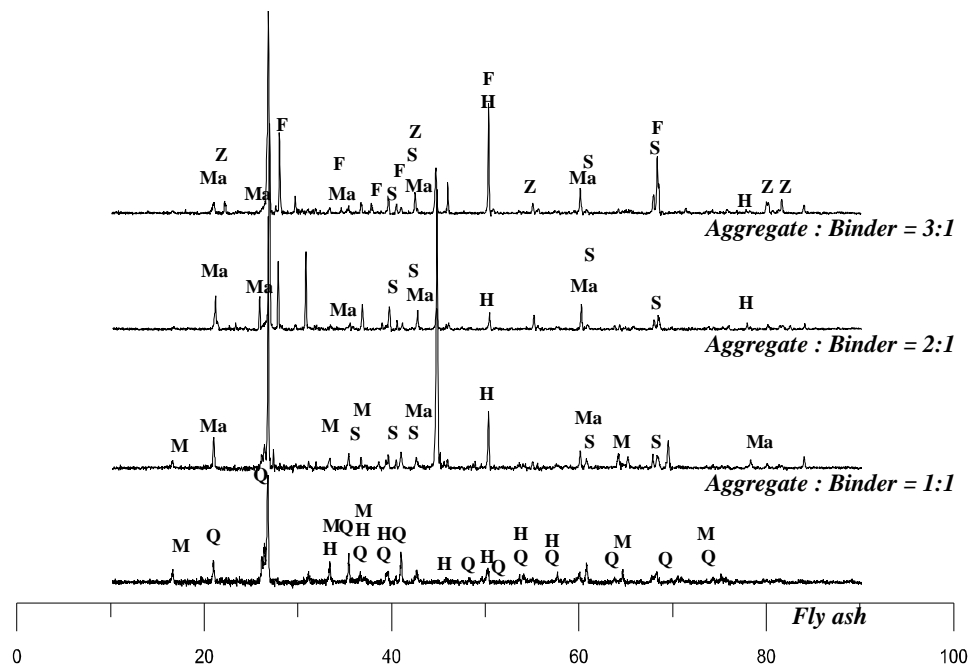
X-ray diffraction analyses were performed using high resolution powder X-ray diffractometer available at MRC (Material Research Center, MNIT Jaipur) having generator setting of 40mA and 40kV. The tests were conducted as per standard guidelines at room temperature of 25°C. angular range of 20°, step size of 0.03° and wavelengths $\lambda = 1.5 \text{ \AA}$ were adopted for the data acquisition during the experiment. Further, the data obtained from the XRD were analysed using software PANalytical X'pert HighScore. The software processes the data and removes the background noise using in-built mathematical functions. The processed data is used for further identification of the peaks. The peaks are matched with standards minerals and available peaks are marked. The software automatically uses the standard data file provided by ICSD (International Chemical Data Service). In this section, crystallography of the geopolymer mortar was studied and their results are reported.



a) Concentration of NaOH = 14 M



b) Concentration of NaOH = 11 M



c) Concentration of NaOH = 8 M

Figure 4.9: X-ray diffraction patterns of alkali-activated geopolymer mortar

Fig 4.9a-c report the variation in diffractograms of the geopolymer mortar with change in aggregate to binder ratio. Fig 4.9a reports the diffractogram of the geopolymer mortar made up using 14 M NaOH. At bottom, diffractogram of the fly ash alone, which contains the various minerals such as Mullite, Quartz, Hematite. The presence of these mineral are in consonance with the chemical composition of the fly ash as reported in earlier chapter. Further, due to geopolymerization process, crystallography of the mortar is found to be changed. However, crystals present in the geopolymer mortar changes with the change in mix composition. The mortar with the aggregate binder ratio of 1:1 possessed the sodalite mineral. The mortar having aggregate binder ratio of 2:1 and 3:1 possessed the group of minerals respectively, they are i) Sodalite and Cristobalite and ii) sodalite and Zeolite.

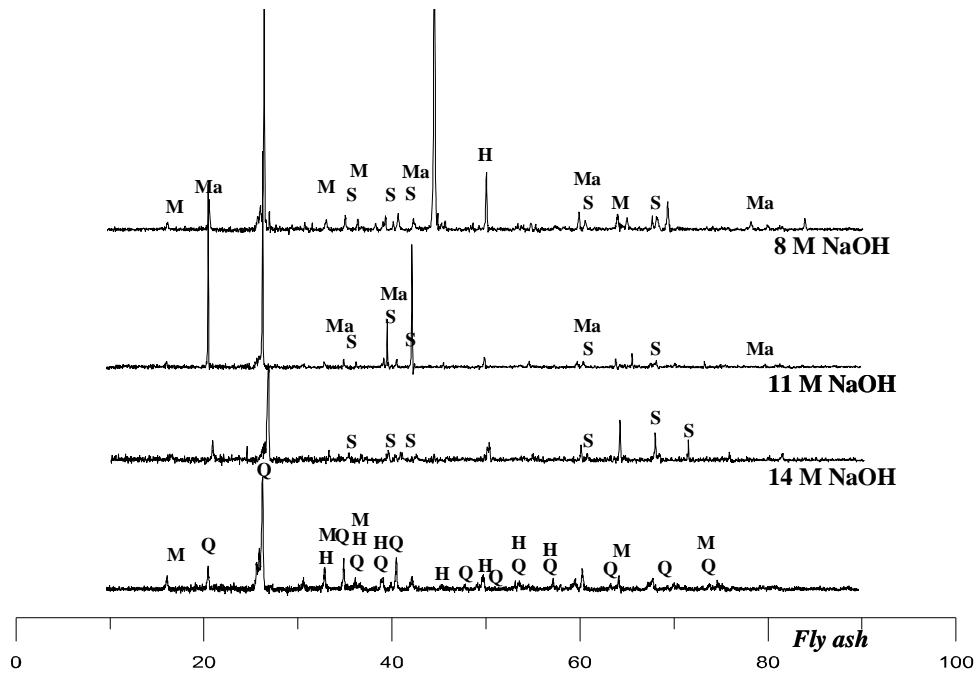
Fig 4.9b shows the diffractogram of the mortar made up using 11 M NaOH. The change in crystallography is clearly visible due to geopolymerisation process. The diffractogram shows that three set of crystals were found in the mortars having aggregate to binder ratios (1:1, 2:1 and 3:1) which are i) Sodalite and magnetite, ii) Sodalite, megantite and hematite and iii) Sodalite,

magnetite and hematite respectively. This demonstrates the influence of mix composition on the crystal structure of the mortar matrix.

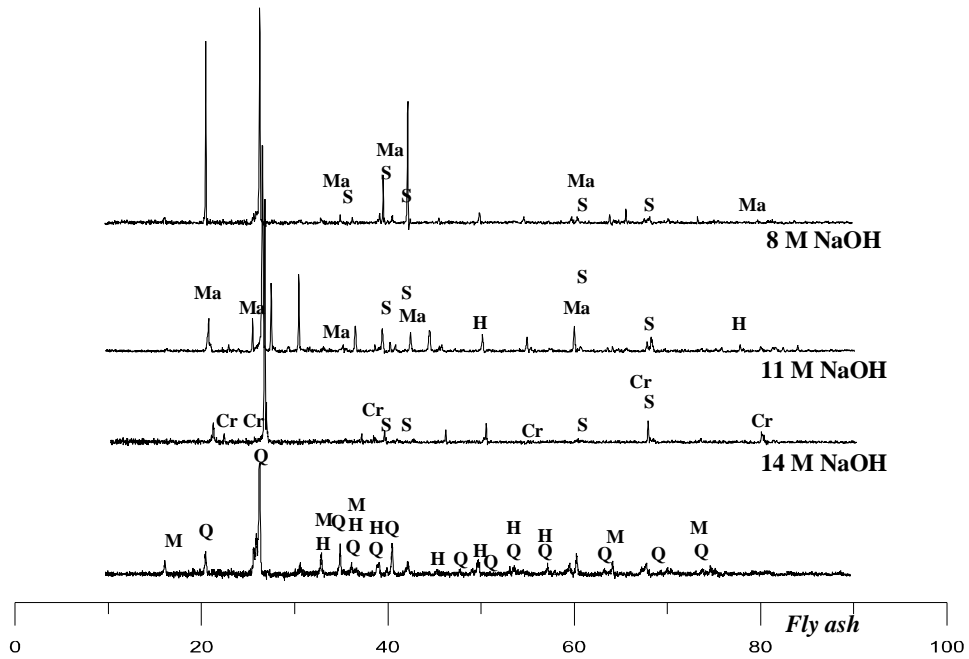
Fig 4.9c depicts the diffractogram of the geopolymer mortar having 8M NaOH. The crystal structure is immensely influenced by the geopolymerisation process, as evident from diffractograms. It can be noticed that mortar made up of different aggregate binder ratio (1:1, 2:1 and 3:1) demonstrated presence of different set of crystals; they are i) Sodalite, Mullite and magnetite, ii) Sodalite, Mullite and hematite and iii) Magnetite, Sodalite and Fledspar.

Fig 4.10a-c present the variation of XRD diffractograms of the geopolymer in order to study the influence of the concentration of NaOH. It can be seen that for mortar made up of higher concentration of the Sodium hydroxide, sodalite was prominent in the matrix of the mortar. The presence of the sodalite was found irrespective of the variation in the aggregate binder ratio. In lower concentration of NaOH, several minerals were found. For example, in mortar having aggregate binder ratio of 1:1, for 8M and 11M NaOH two set of minerals were found they are i) Hematite, Magnetite and Sodalite and ii) Magnetite and Sodalite.

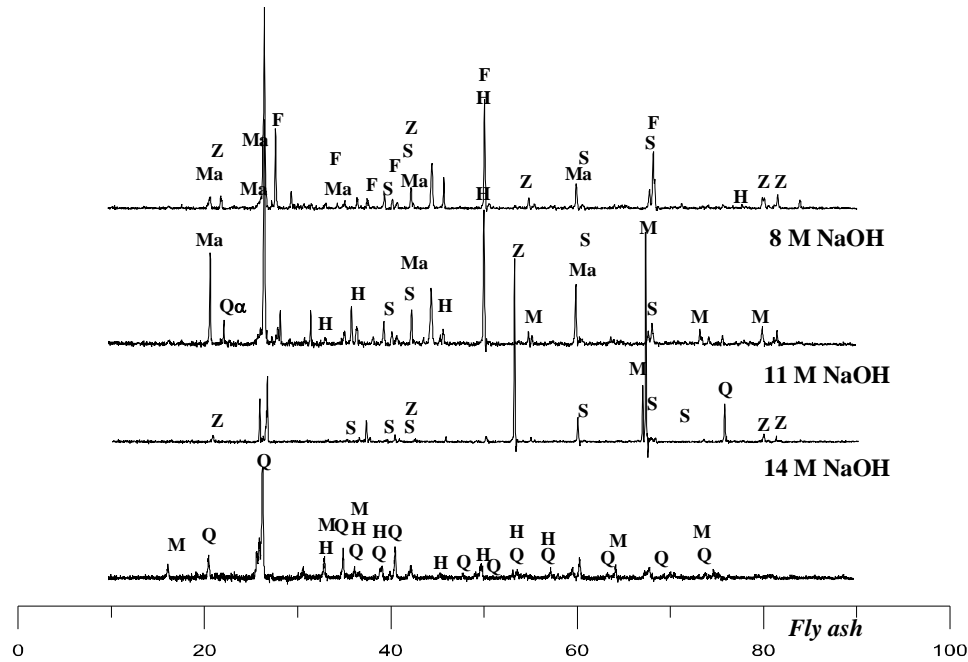
Similarly, the mortar with aggregate-binder ratio equal to 2:1, two set of minerals (i.e. i) Magnetite and Sodalite and ii) Magnetite and Sodalite) were found for 8M and 11M NaOH, respectively (Fig 4.10b). Further, the mortar made up with aggregate-binder ratio equal to 3:1, presence of two group of minerals (i.e. i) Zeolite, Magnetite, Feldspar and Sodalite and ii) Magnetite, Hematite and Sodalite) were observed for the for 8M and 11M NaOH, respectively



a) Aggregate : Binder = 1 : 1



b) Aggregate : Binder = 2 : 1



c) Aggregate to binder = 3:1

Figure 4.10: X-ray diffraction patterns of the alkali-activated geopolymer mortar

4.4 Investigation on apparent porosity and water absorption of geopolymer mortar

The porosity of the mortar has been vital and necessary property to predict the durability and strength of the mortar. In this study, influence of the parameters such aggregate binder ratio and curing temperature on the apparent porosity of the geopolymer mortar was examined.

4.4.1 Test method

The apparent porosity can be predicted indirectly by calculating bulk density. However, being a porous matrix, sometime indirect methodology may produce erroneous results. In the view of above, Montes et al, 2005 proposed a novel methodology to measure the porosity of the mortar and concrete by using Archimedes principle. The apparent porosity (n) can be expressed in terms of weights.

$$n = \frac{(M_w - M_d)}{(M_w - M_s)} \times 100\% \quad (4.1)$$

Here,

M_w = Weight of specimen after immersing into the water for 48 hours

M_d = Weight of specimen after keeping for drying in oven at 85°C for 24 hours

M_s = Weight of specimen when it is suspended in water

In the present study, specimens of geopolymer mortar were cast by mixing the various components in predefined proportions. The dimensions of the specimen were adopted as 50 mm x 50 mm x 50 mm. After air curing of the mortar specimen after the duration of 28 days, the sample were kept in oven for drying. The dry weight was achieved at the gap of 24 hours, and the difference between subsequent duration of drying. This process was followed until the difference between dry weight measured in two subsequent drying duration reached in the range of 0.05%. This drying weight was referred as M_d . Further, the specimen was soaked in to the water for 48 hours and its saturated surface dry weight was measured. The process was followed in each 24 hours until the tolerance of error reaches within the limit of 0.05%. The final saturated surface dry weight was referred as M_w . Finally weight of the specimen was achieved in suspension condition in the water, which is referred as M_s . Finally based on the equation , the apparent porosity was calculated.

Similarly, the water absorption can also be calculated as explained below

$$w_{ab} = \frac{(M_w - M_d)}{(M_d)} \times 100\% \quad (4.2)$$

4.4.2 Test program

In this, the effect of change in aggregate to binder ratio and curing temperature on the apparent porosity and water absorption capacity of the mortar was investigated. For this purpose, test matrix is adopted as shown in table 4.2. Average apparent porosity was calculated by measuring the apparent porosity of three specimens having identical composition and curing condition. Further section reports the results and discussion of all the tests conducted for the evaluation of the apparent porosity and water absorption capacity.

4.4.3 Results and discussion

Fig 4.11 demonstrates the variation of porosity with the aggregate to binder ratio and curing temperature. It can be seen that the porosity decreases with increase in aggregate to binder ratio up to a certain point; afterwards, it increases with further increase in aggregate to binder ratio. The initial decrease in porosity can be attributed to proper compaction of the fine aggregates and binder in to the mortar matrix. The increases in curing temperature helped mortar to achieve densified matrix hence lesser porosity, which is evident from the Fig 4.11.

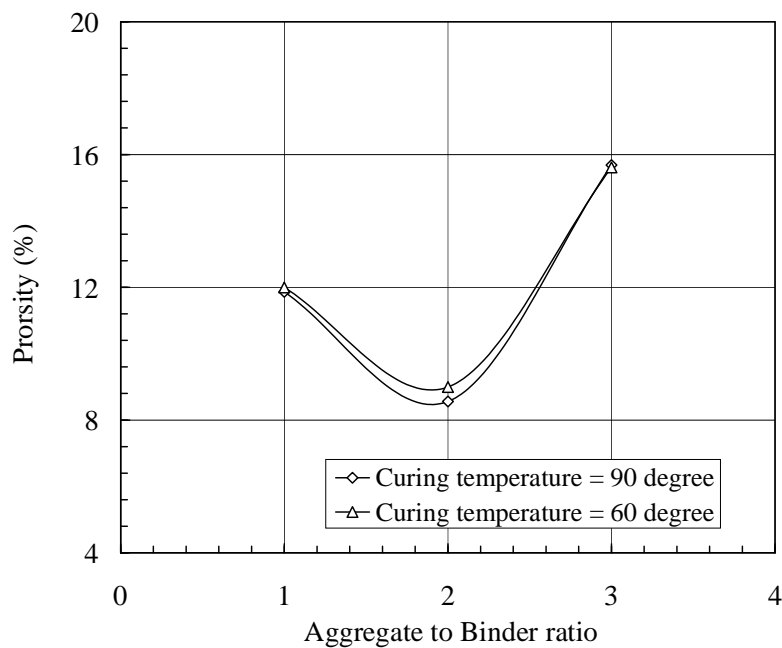


Figure 4.11 : Variation of porosity with aggregate to binder ratio

Fig. 4.12 demonstrates the relation between average compressive strength, average porosity and curing temperature. It can be noted that compressive strength of the alkali-activate geopolymer mortar is inversely proportional to average porosity. With increase in temperature, at approximately constant porosity, average compressive strength increases. The above relation was also fitted in to polynomial in order to obtain empirical relation between parameters, which can be used to predict the average compressive strength of the geopolymer mortar.

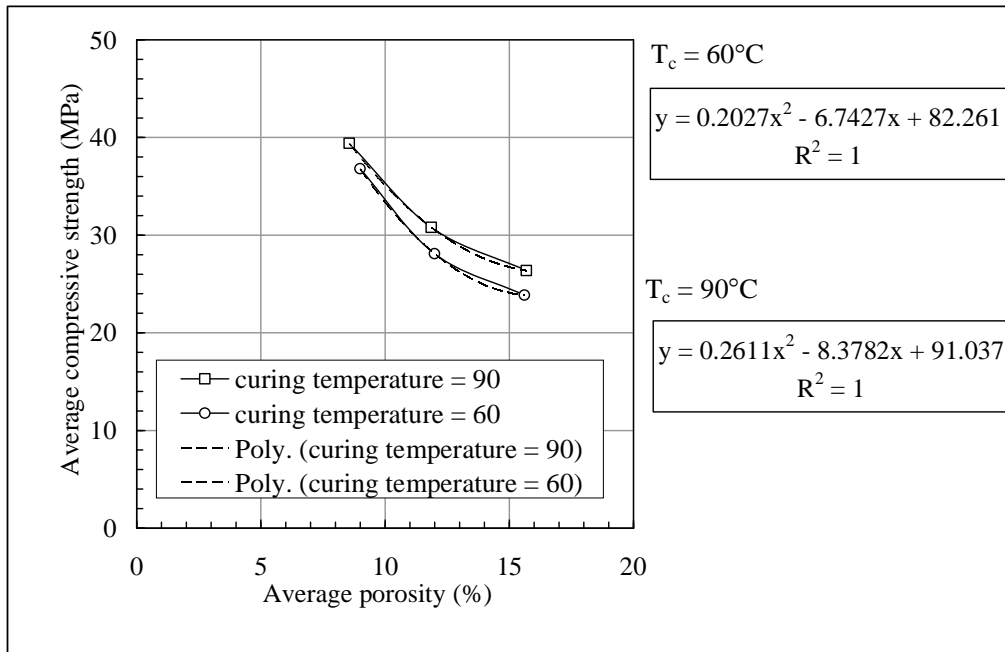


Figure 4.12 : Relation between compressive strength and porosity

4.5 Investigation on Carbonation of Geopolymer Mortar

According to literature, the durability of mortar is significantly affected by the attack of CO_2 . In order to understand the process of the carbonation and effect of mix composition of the mortar on the carbonation was studied by conducting series of experiment. This section presents the test method adopted in the present study, test program, test results and discussions.

4.5.1 Test Method

The carbonation reaction was performed on the mortar specimens were conducting according to standards CPC18.

Preparation of mortar specimen

Pre-defined proportions of the various components were blended manually to achieve the mix composition. The paste was poured into the moulds to prepare the beam specimen. The beam specimen has length of 160 mm, breadth of 40mm and height of 40mm. After keeping at room temperature for 300 minutes, the beam specimens were kept into the oven for 24 hours in order to achieve the curing due to heating.



a) Casting of specimens



b) Samples after painting



c) Samples within the carbonation chamber

Figure 4.13: Carbonation process used in the present study

Testing procedure

Accelerated carbonation tests were carried out in a chamber having specific experimental condition such as atmospheric pressure, temperature equal to $20 \pm 2^\circ\text{C}$, relative humidity equal to $65 \pm 5\%$ containing a constant concentration of CO_2 . The concentration of CO_2 was maintained 5% in order to get reasonable duration of the test in the laboratory. After a preliminary literature survey from the previously conducted experimental studies. The relative humidity was maintained at 65% by using an ammonium nitrate saturated solution in the carbonation chamber. Specimens were placed for the carbonation just after exposing them to the air for at least 7 days.

After air curing, the specimens were painted in the four faces along the length of the specimen with ASIAN paints in two layers as explained in CPC18. The carbonation was quantified in terms of variation in carbonation with the depth. The carbonation depth can be used to compute the carbonation co-efficient of the geopolymer mortar. For the measurement of the carbonation depth, the beam specimen of the mortar was split in to two parts along the length after the completion of carbonation process in the carbonation chamber. The phenolphthalein solution, prepared by mixing 1% phenolphthalein in 70% Ethyl alcohol, was sprayed on the freshly broken surface. The solution is used to identify the change in pH of the freshly broken surface. Due to the inherent quality of the solution, non-carbonated surface becomes pink, while carbonated surface remains colorless. The Difference in the color profile were measured by scale and reported. The measurement of the carbonation depth was conducted strictly following the CPC18 guidelines. These guidelines recommends the methodology when, profile of the carbonation was undulated or curvy in nature.

4.5.2 Test Program

The test series for the carbonation experiments was termed as Series-B. Mix composition having NaOH concentration equal to 14 was selected from the Series A. Further, the parameters such as Aggregate to binder ratio and curing temperature were varied by keeping values of other parameters constant. Table 4.2 presents the test program of the series B and their corresponding values of the parameters.

Table 4.2: Summary of the test program adopted for Series B in the present study

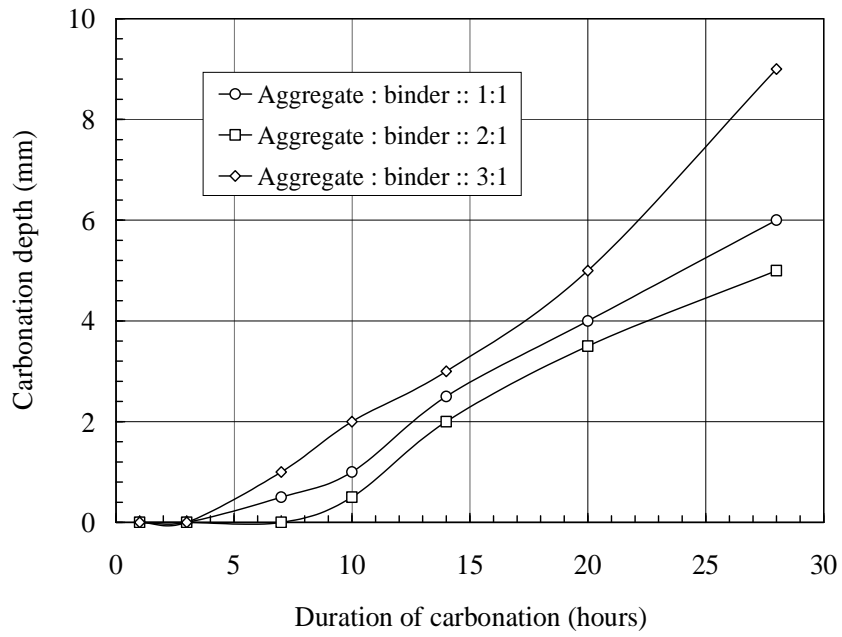
S.N	Test Legends	NaOH concentration	Aggregate to binder ratio	Curing Temperature
1	C-1	14M	1:1	60°
2	C-2	14M	2:1	60°
3	C-3	14M	3:1	60°
4	C-4	14M	1:1	90°
5	C-5	14M	2:1	90°
6	C-6	14M	3:1	90°

4.6 Results and Discussion

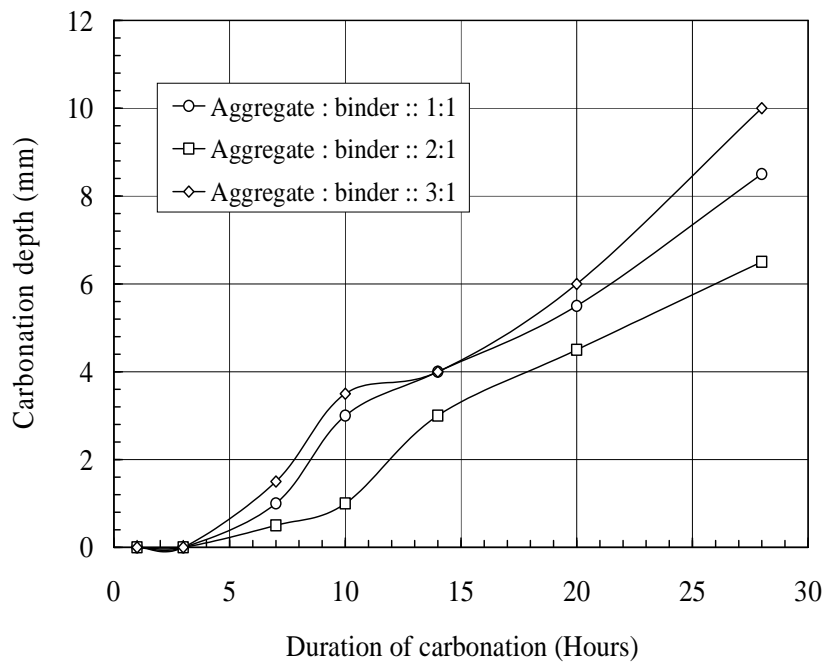
In this study, the influence of aggregate to binder ratio and curing temperature on the carbonation process of the geopolymer mortar was studied. For this purpose, several samples having mix composition stated in Table 4.2 were placed in carbonation chamber. The samples were tested at specific times and carbonation depth was measured according to CPC-18. In the next section, results of the carbonation tests are reported.

4.6.1 Influence on Carbonation Depth

Fig 4.14 depict the variation in carbonation depth with time and aggregate content in the geopolymer mortar. As can be noticed from the Fig 4.14a, carbonation depth increases with carbonation time. Carbonation depth for the samples having aggregate binder ratio of 2:1 was found to be minimum. It was also observed from the results of compressive strength test and porosity test that the geopolymer made up of aggregate to binder ratio 2:1 demonstrated maximum compressive strength and minimum porosity. Hence, the reduced amount of carbonation can be attributed to decreased porosity and compact state of geopolymer matrix. Similarly, the sample having aggregate-binder ratio 3:1 demonstrated maximum values of the carbonation depth throughout the test. The higher amount of carbonation can be due to increased porosity of the sample as evident from the porosity test results reported in previous sections.



a) Curing temperature = 60°C



b) Curing temperature = 90°C

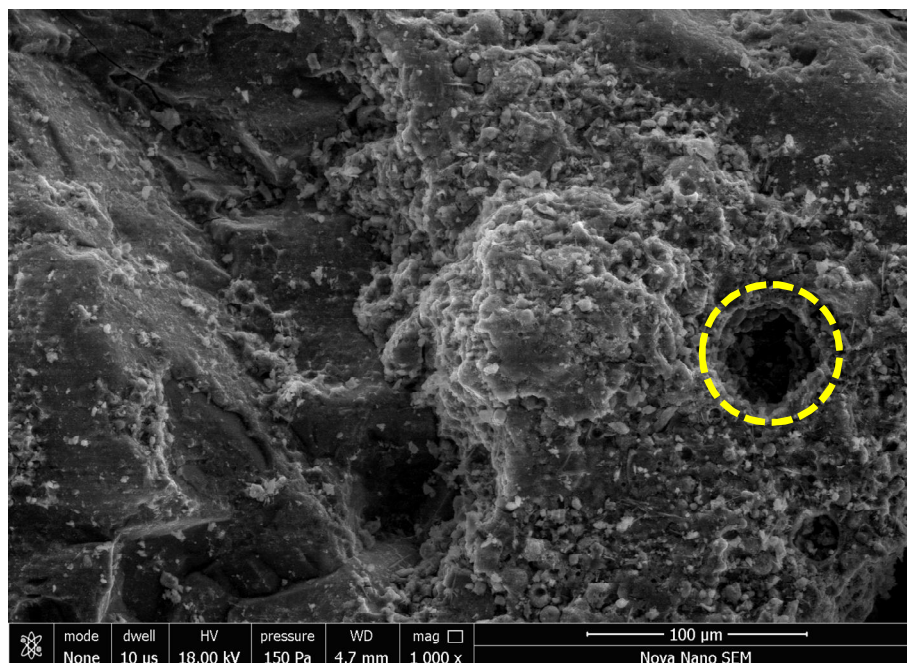
Figure 4.14: Variation of carbonation depth with carbonation time in hours

Figure 4.14b demonstrates the variations in carbonation depth with carbonation time for the samples cured at 90°C. It can be seen that marginal decrease in carbonation depth was observed due to increase in curing temperature. This can be attributed to solidification of the gel due to elevated temperature which results into decrease in porosity of the matrix. The decrease in porosity hinders the diffusion of CO₂ in to the matrix of the geopolymer mortar.

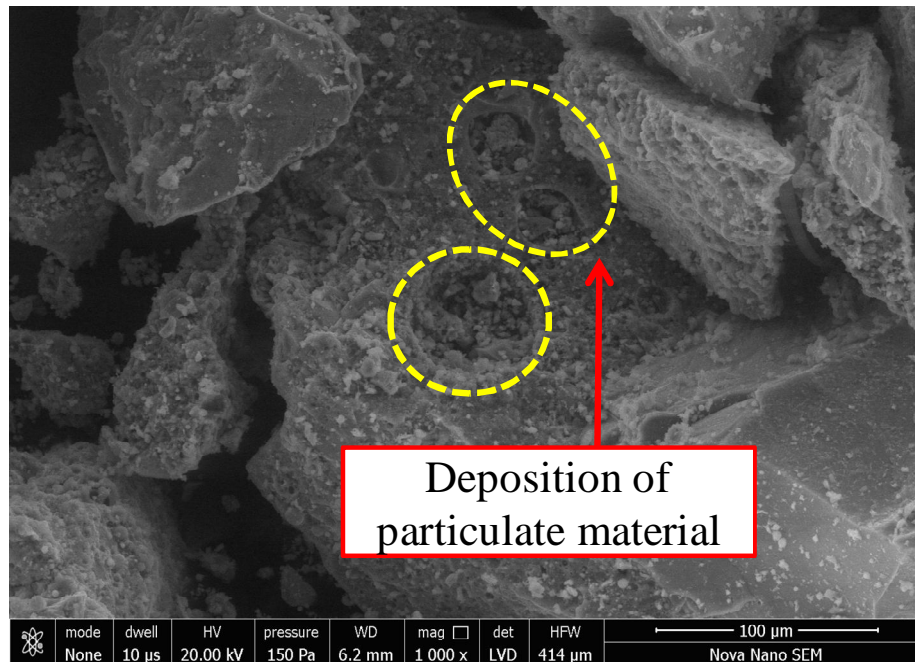
Hence, based on the results of the carbonation experiments it can be concluded that durability of the geopolymer mortar against the carbonation can be improved by selecting proper mix composition of the mortar. It was found that fine aggregate to binder ratio and curing temperature are vital mix composition parameters which dictate durability of geopolymer mortar when subjected to carbonation.

4.6.2 Influence on Micro-structure of Geopolymer Mortar due to Carbonation

Fig 4.15 presents the images taken from the SEM of the mortar before and after the carbonation process. It compares the change in microstructure of the surface of the mortar during the carbonation. It can be seen that the small depressions in the external surface of the mortar are present before the carbonation, which can be due to uneven geo-polymerization of the mortar.



a) Before carbonation



b) After carbonation

Figure 4.15: Micrographs of geopolymer mortar before and after carbonation

In Fig 4.15b, it is interesting to note that the depression in the surface underwent change due to carbonation. The carbonation led to deterioration of the surface at micro-level. As evident from the image, the deteriorated material was found in particulate form.

Fig 4.16 shows the micrographs of the geopolymer mortar comprised of aggregate to binder ratio varying from 1:1 , 2:1 and 3:1. It can be seen that needle type crystals were formed due to carbonation of the mortar. The formation of the crystal is maximum in case of mortar having aggregate to binder ratio of 3:1. The presence of different type of crystals is further investigated with the help of XRD analysis.

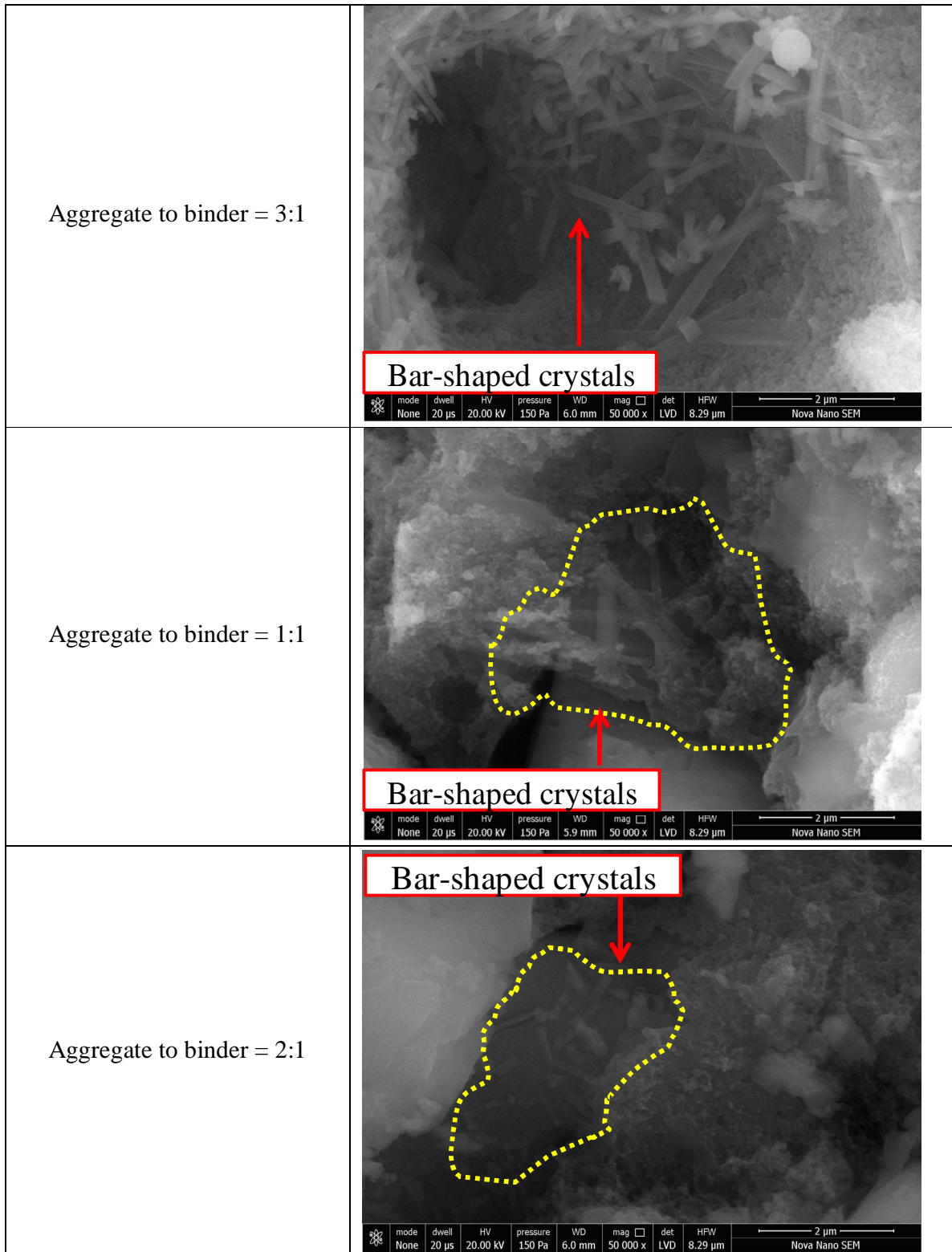
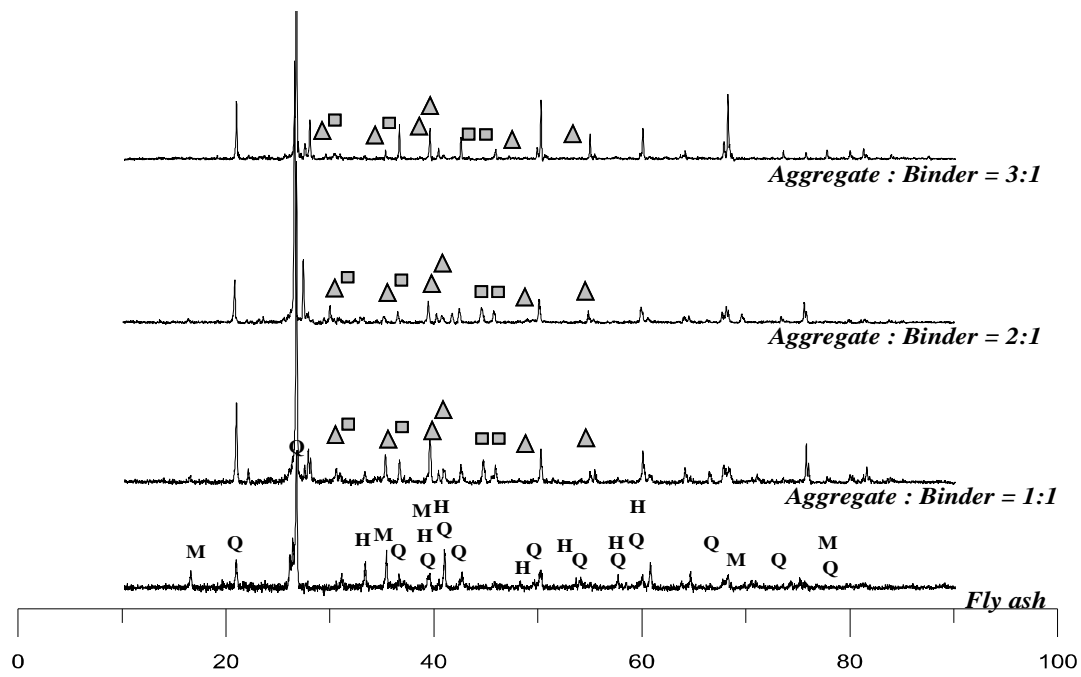
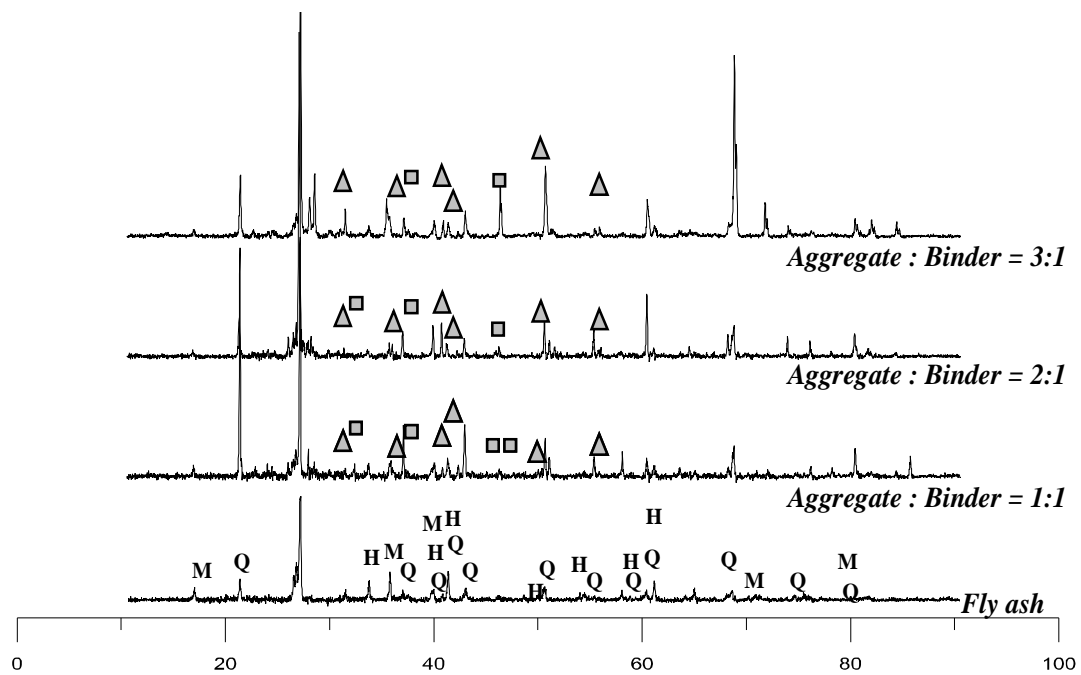


Figure 4.16: Micrograph of carbonated samples prepared at various aggregate to binder ratio



a) Curing temperature = 60°C



b) Curing temperature = 90°C

Figure 4.17: XRD diffraction pattern of carbonated geopolymer mortar

In this section, XRD diffractogram of carbonated geopolymer mortar were obtained for varying aggregate-binder ratios (1:1, 2:1 and 3:1) and curing temperature (60°C and 90°C). The carbonation process significantly changes the pore chemistry. In this study, with the help of diffractograms, change in pore solution chemistry could be identified. For this purpose, a mineral named as Nahcolite (\blacktriangle), was investigated. As shown in Figure 4.17 , at the onset of carbonation, change in pore solution chemistry took place and formation of Nahcolite (\blacktriangle) was observed. In parallel, Sodium carbonate hydrate (\blacksquare) was also formed. During carbonation, on prevalent thermodynamic and environmental condition, Nahcolite (\blacktriangle), was most favorable product to be formed. Similar types of observations were noticed in all types of mortars having varying mix composition of aggregate-mortar ratio and process parameters such as curing temperatures.

CHAPTER-5

CONCLUSIONS

5.1 General

In this study, experimental investigation followed by detailed literature was carried out. The effect of mix composition parameters and process parameters on the performance of the alkali activated geopolymer mortar was examined. Further carbonation of the mortar and its effect on its morphology was studied. This chapter summarizes the conclusion of the whole experimental investigation.

5.2 Based on Compressive strength tests

- NaOH helps in dissolution of fly ash into the mortar matrix. Higher concentration of NaOH was found to influence the formation of gel which resulted into increase in compressive strength.
- The influence of the aggregate to binder ratio was observed in specific manner. An optimum ratio is needed to ensure maximum compressive strength. In the present study, the aggregate to binder ratio of 2:1 was found to be optimum, which can be attributed to proper compactness of the mortar as evident from porosity values.
- Porosity and water absorption capacity of the geopolymer mortar is also found to be dependent on curing temperature and aggregate-binder ratio. Further, the porosity and compressive strength of the geopolymer mortar was also found to be linked with each other. The relationship between them is demonstrated with help of empirical equation.

5.3 Based on Carbonation of the geopolymer mortar

- Degree of carbonation was measured in terms of carbonation depth, which is found to increase with decrease in curing temperature. It highlights the importance of curing temperature in order to arrest the possible degradation of mortar when subjected to carbonation, which in turn influences the long term performance of the mortar.

- Similarly, aggregate-binder ratio of 2:1 was found to be best performer against the carbonation. It indicates the importance of proper selection of mix composition to ascertain the performance of geopolymer mortar

5.4 Based on XRD diffractograms and SEM analysis

- Diffractograms of the various mortar provide the information about the crystal formed during geopolymerisation of the geopolymer mortar. In the fly ash, Quartz, mullite and hematite are present. The several crystals formed due to geopolymerization are sodalite, magnetite, zeolite and feldspar.
- Similarly, during carbonation, visible change in morphology was observed. The newly formed minerals is Nahcolite.
- SEM provided the crucial information about change in surface features of the geopolymer mortar. Further, with help of the micrographs, presence of unreacted fly ash particle in some of the samples were traced. This indicates towards the insufficiency of concentration of NaOH to dissolve the whole fly ash.

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